Project Report No. 6
South Georgia Minerals Program

State Department of Conservation
Department of Mines, Mining and Geology

A. S. Furcron, Director

EFFINGHAM COUNTY
SANDS AND HEAVY MINERALS
PHOSPHATE - SAND

By

Mineral Engineering Branch, Engineering Experiment Station
Georgia Institute of Technology and
Georgia Department of Mines, Mining and Geology

May 1967

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Project A-880-001 of the Georgia Institute of Technology,
Atlanta, Georgia

ERRATA

Electric and gamma-ray logs for Hole Nos. Ef-3 and Ef-4 in Effingham County and Hole No. Ec-12 in Echols County were not run; therefore, pages 13, 14, and 75 are missing from this report.

FOREWORD

Work, to date and as reported in previous project reports, has been focused primarily on phosphates. A fairly recent publication noted the apparent existence of five "old beaches" in the southern part of Effingham County running in a southwestward-northeastward direction.* "Old beaches" are logical sites for heavy minerals deposits. Accordingly, a drilling program was carried out in Effingham County with several objectives: existence of economic heavy minerals deposits, quality of sands, to determine the economic feasibility of phosphorite deposits known to exist in the county, and to obtain geologic control information.

Relatively small variations in "accepted" methods of analyses for phosphates had been found to produce undesirable, but not highly significant, variations in results from one analyst to another. Wet chemical, including spectrophotometric, atomic absorption, and x-ray fluorescence techniques offer a choice of methods. Accordingly, details of procedures adopted for this minerals program to date are presented herein as Appendix III.

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*"Geomorphology of the Lower Coastal Plain from the Savannah River Area, Georgia, to the Roanoke River Area, North Carolina," Evelyn Sinha, Ph.D. Dissertation, University of North Carolina, 1959.

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SOUTH GEORGIA MINERALS PROGRAM

INTRODUCTION

Five project reports have been issued to date on this program which was initiated on July 1, 1965. All information is published and released to the general public as soon as feasible. While desirable, it does mean that a single project report does not treat a single subject, such as "heavy minerals," nor a single county or region, nor contain all information; new information as acquired being presented in succeeding reports. Project Report No. 6 illustrates this point.

It is expected that separate comprehensive reports covering a single subject or a specific area will be prepared when all data to be obtained under this program have been obtained.

Emphasis in Project Report Nos. 2-5 inclusive has been on phosphates with some information on heavy minerals in Echols County presented in Report No. 2. With the rather recent release of a publication indicating the presence of "old beaches" in Effingham County and knowing of its sand mining operations, a drilling program in Effingham County had its emphasis "shifted" to heavy minerals and sands, but not overlooking phosphorites. Then, in order to summarize the sands picture, data on deposits in other counties drilled, but not previously reported, are included herein. Also included, as not available earlier, are reproductions in reduced form of electrical resistivity and gamma-ray logs of holes drilled and reported in Project Reports 2 and 3.

Research has shown that minor variations in laboratory phosphate analyses resulted in undesirable variations in results. This, plus the fact that a "choice" between different techniques was available, made it desirable to present in detail the laboratory procedures determined to be most suitable for the South Georgia Minerals Program. These procedures and discussions are included as Appendix III.

Figure 1 is an outline map of the counties in the Coastal Plain showing the location of all holes drilled and reported to date.

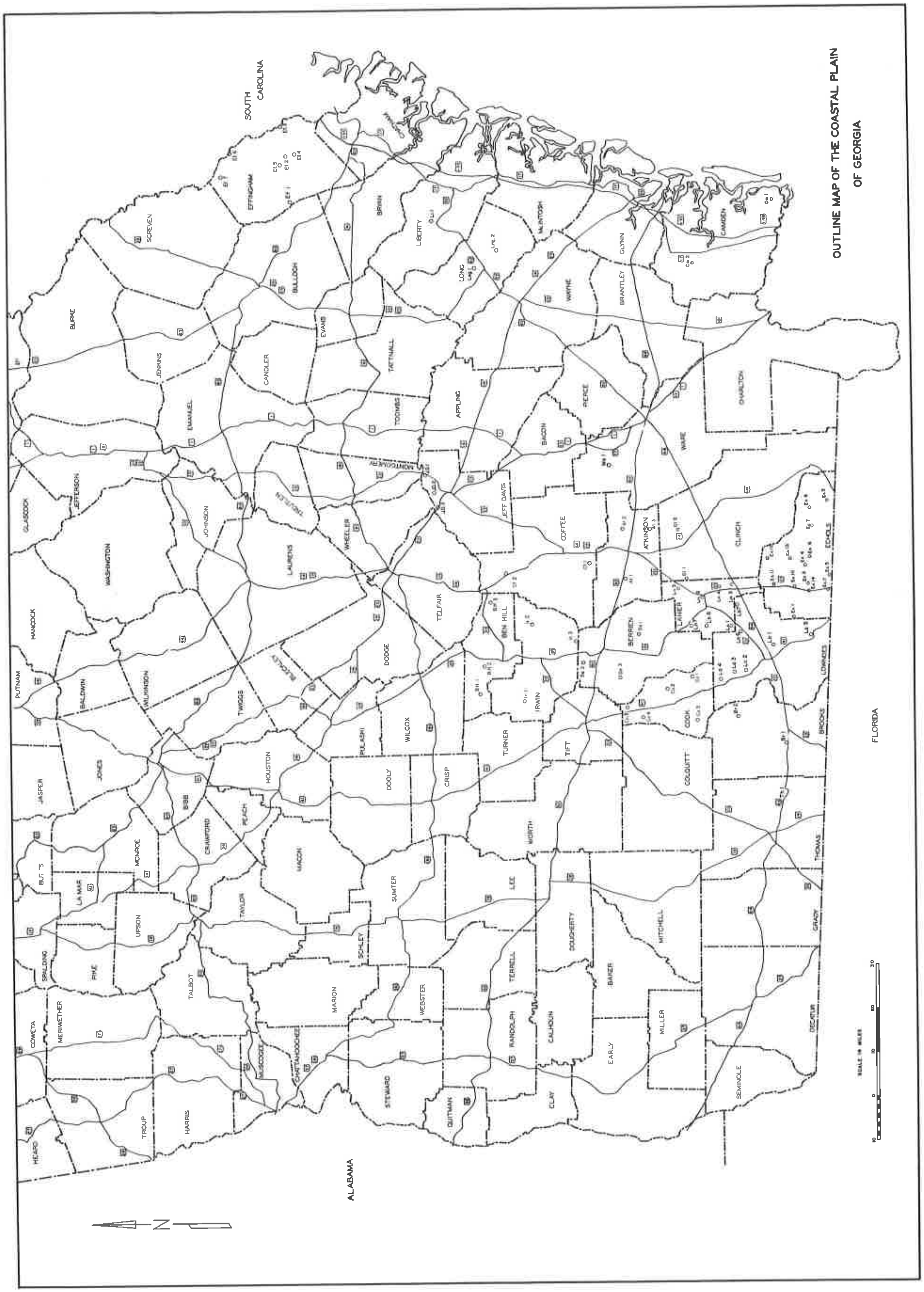

Figure 1. Index Map of Program Area

OBJECTIVE AND SCOPE

The objective of the South Georgia Minerals Program is to determine the existence, preliminary quality-quantity data, and approximate location of mineral deposits having economic feasibility potential for establishing new, or expanded, mineral industries in Georgia.

The scope of this report, Project Report No. 6, includes:

1. Data obtained from core drilling in Effingham County.
2. An evaluation of "sands" and "heavy minerals" in Effingham County and in other counties reported to date.
3. Reproduction of electrical resistivity and gamma-ray logs not formerly reported.
4. Details of analytical procedures developed and adopted for this program.

PROCEDURE

Project procedures and core processing were continued as detailed in Project Report No. 5 except that petrologic descriptions of core materials were omitted.

Composition analyses were performed as detailed in Appendix III. The methods used may be summarized by:

Component	Method
Acid Insoluble Residue (Sand and Insoluble Silicates)	Wet Chemical - Titration
Bone Phosphate of Lime (BPL)	Wet Chemical - Titration
Silica	Wet Chemical - Spectrophotometer
Titania	Wet Chemical - Spectrophotometer
Calcium Oxide (Lime)	Atomic Absorption Spectrophotometer
Alumina	Atomic Absorption Spectrophotometer
Iron Oxide	Atomic Absorption Spectrophotometer
Magnesia	Atomic Absorption Spectrophotometer

RESULTS SUMMARY

Electric and gamma-ray logs on logged holes reported to date have now been presented.

Phosphates of inadequate current economic criteria occur in Effingham County from as close to surface; generally from 20-25 feet and continuing throughout the core, note Ef-3 in particular. Fair to poor concentrations of BPL (5-9%) in the matrix was encountered at depths of 78 feet and more. Only one 5 foot layer, in Ef-3, was found to have a BPL content of over 9%. Based on sea level elevations, the higher quality deposits seemed to have a downward trend to the eastward and an upward trend to the south. This indicates a desirability of explorations for phosphates southward into Chatham County.

There are definite commercial potentials in the sands in each of Atkinson, Ben Hill, Berrien, Effingham, Irwin, and Lowndes Counties. Based on quality and uniformity of significantly thick layers, the best potential for glass sands were in At-1, At-3, BH-2, Be-3, Ef-3, Ef-5, Ir-3, and Lo-4. Simple attrition scrubbing and screening should upgrade the quality. Proximity to a market, however, probably will be a deciding factor. The sands are adequate for other, more local, uses such as highway construction and foundry.

Heavy minerals apparently do not occur, in the areas drilled, in sufficient concentrations to be of economic interest. This was anticipated.

A complete detailed description of the analytical procedures being used is presented in Appendix III. Difficulties experienced with analyses of high quality sands are discussed.

EFFINGHAM COUNTY

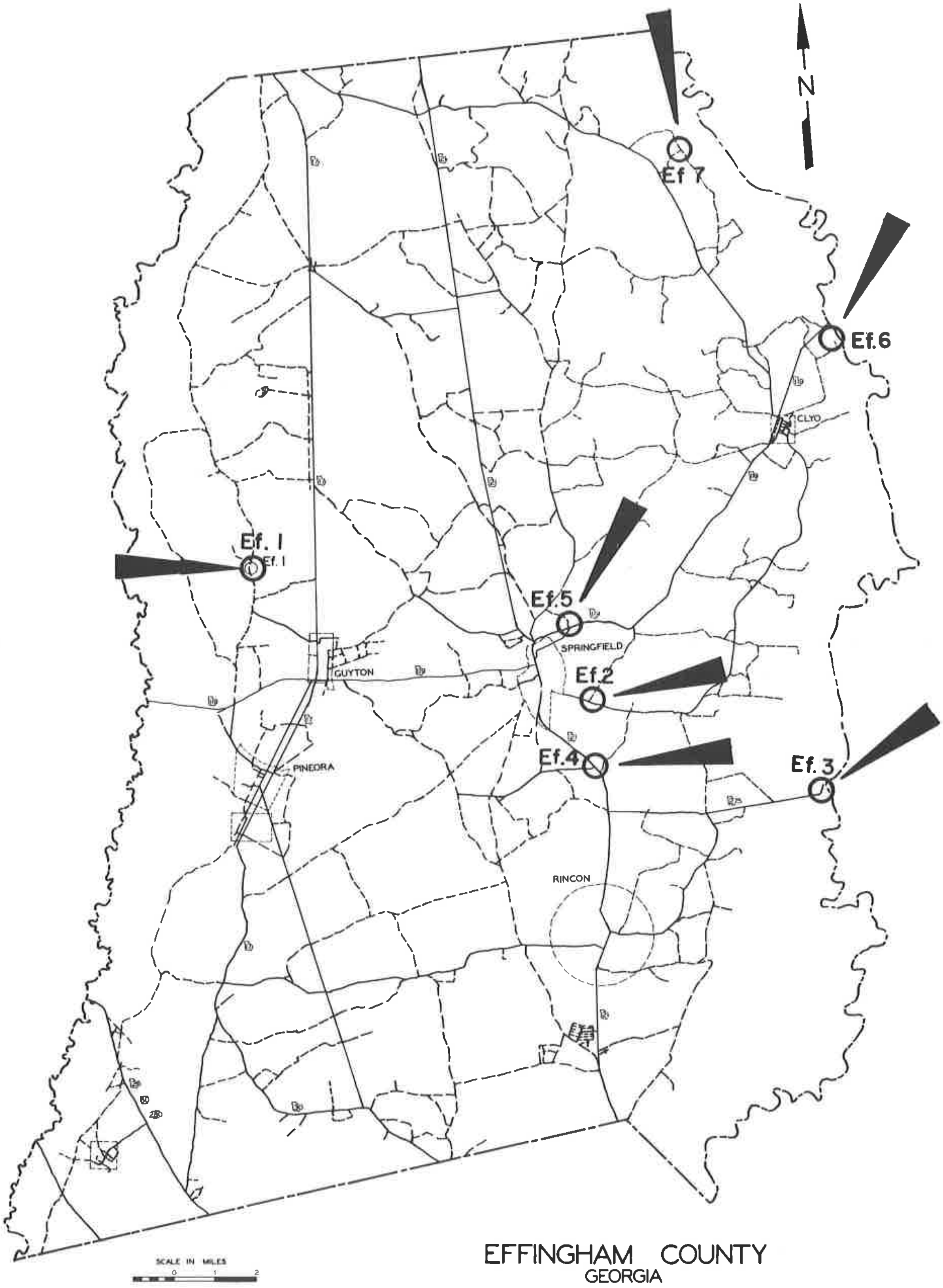

Figure EF-1. Location of Holes - Effingham County

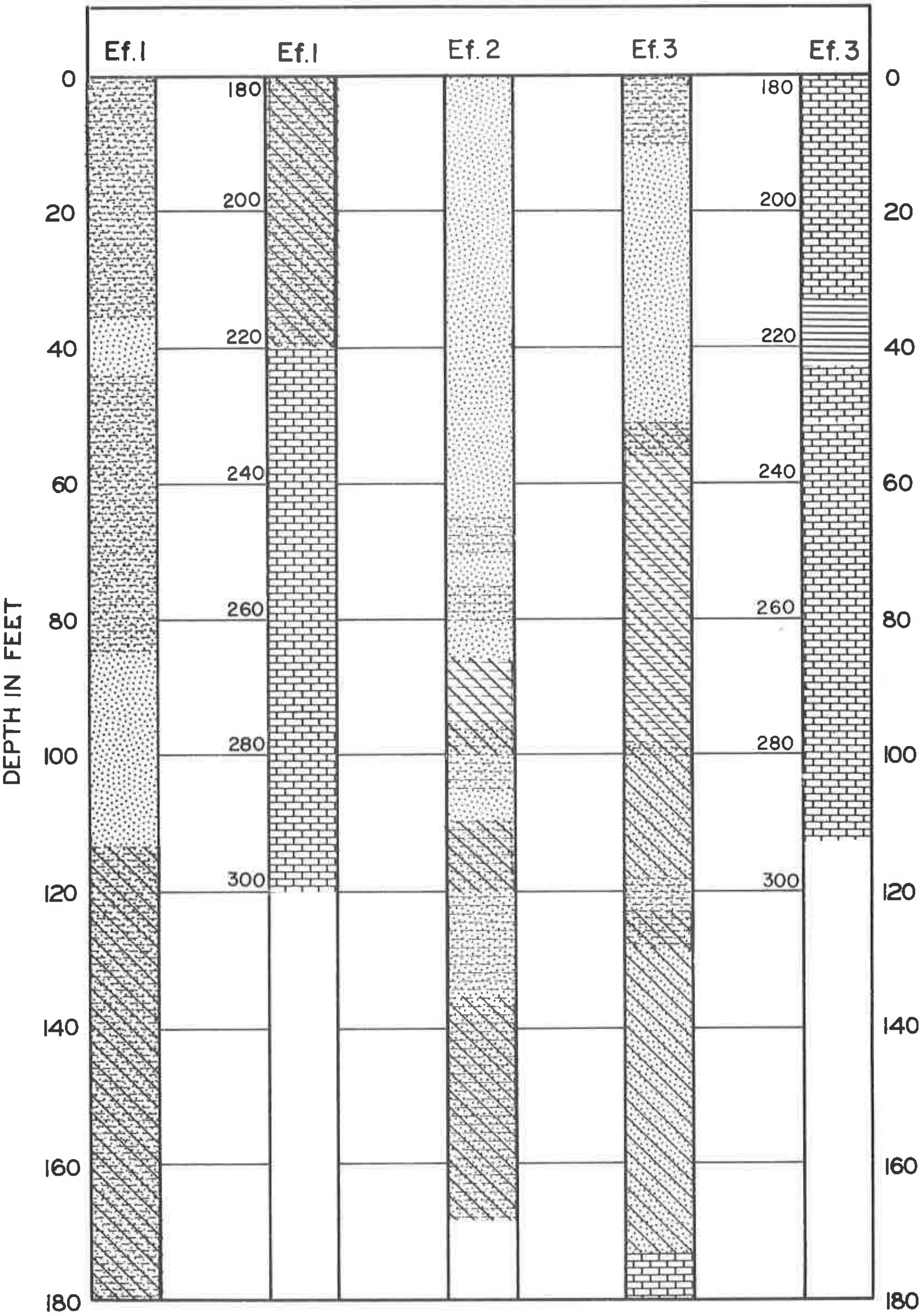

Figure EF-2. Lithologic Logs - Effingham County
Hole Nos. Ef-1, Ef-2, Ef-3

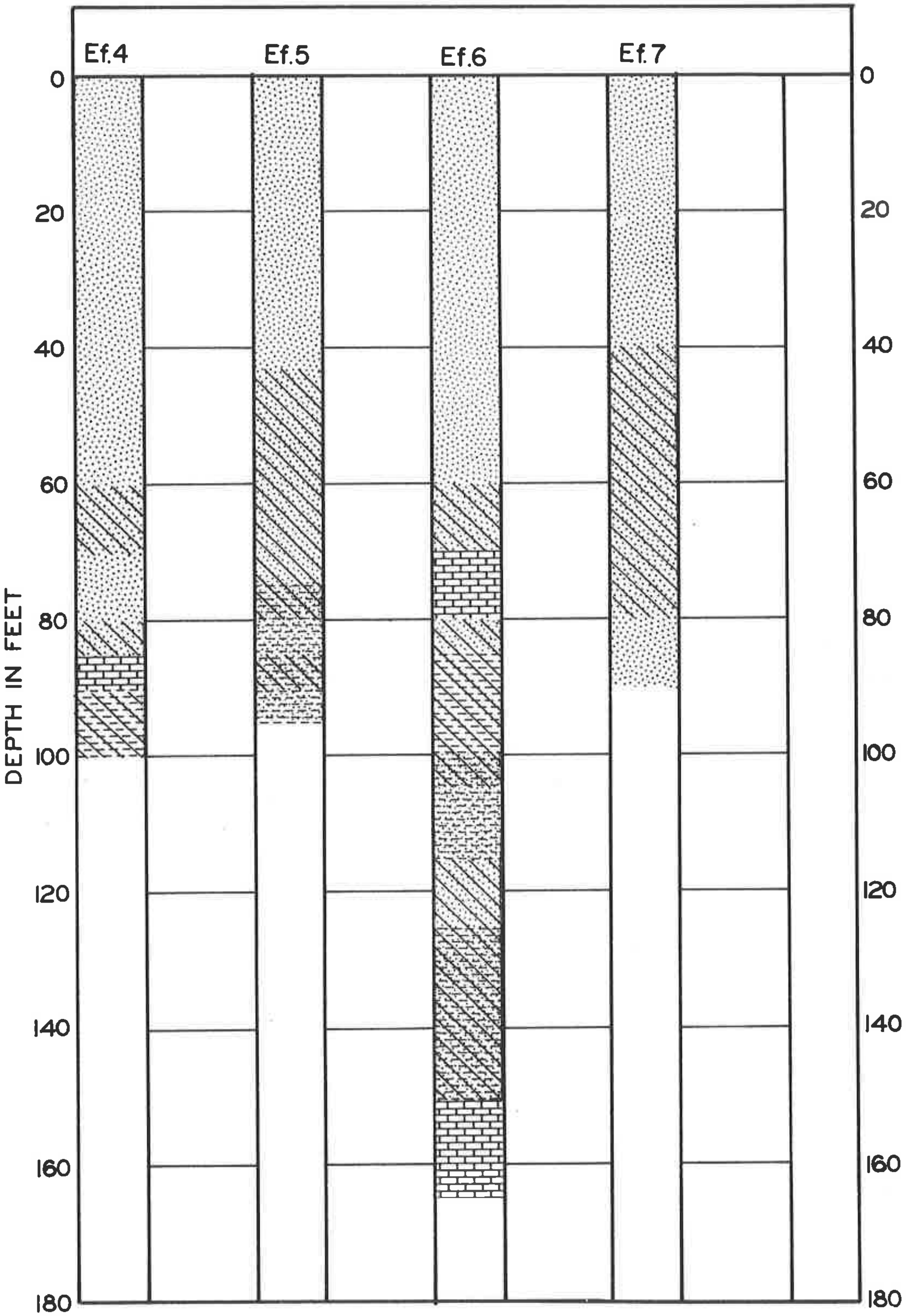

Figure EF-2. Lithologic Logs - Effingham County
Hole Nos. Ef-4, Ef-5, Ef-6, Ef-7

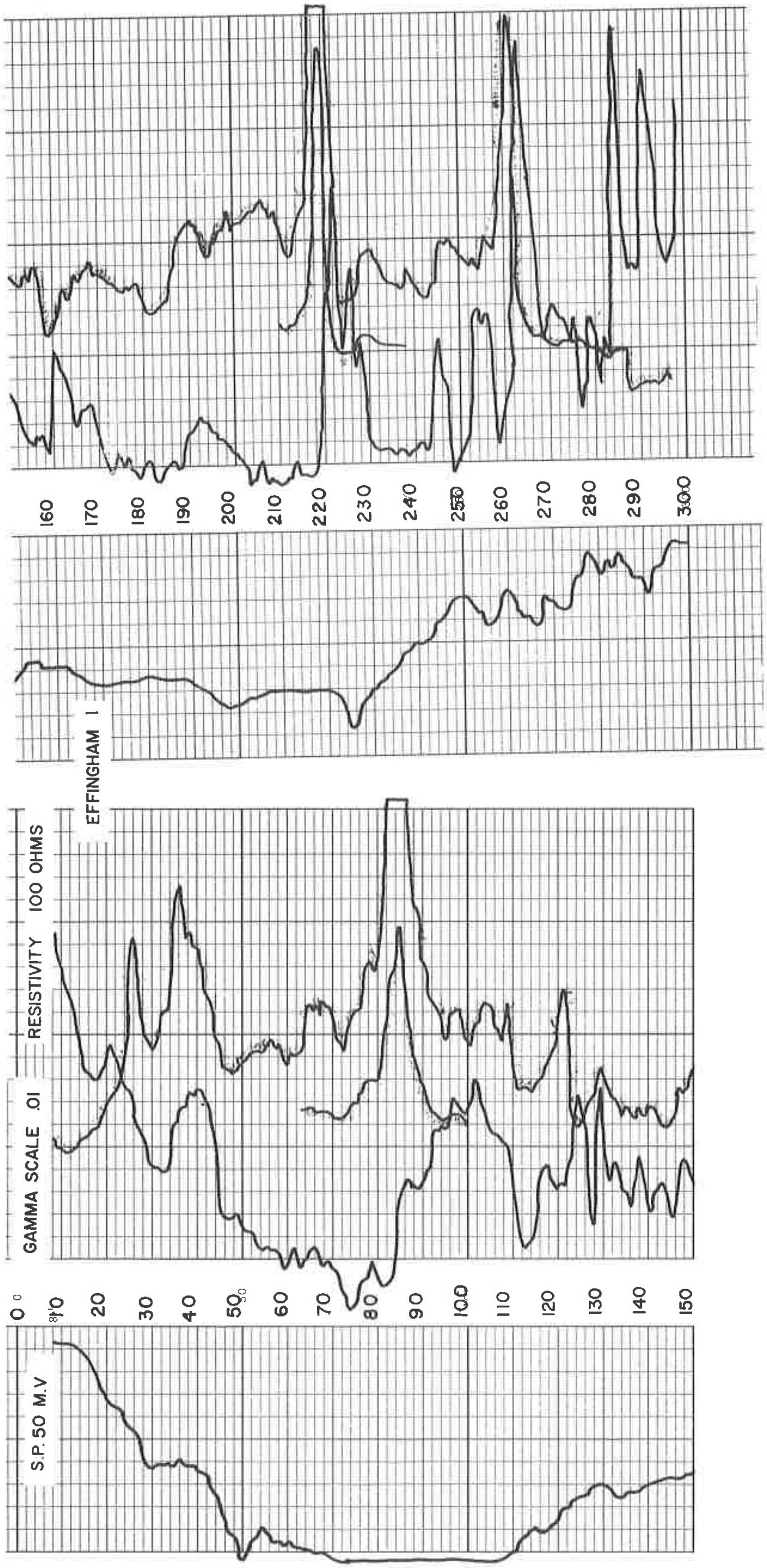

Figure EF-3. Electric and Gamma-Ray Logs - Effingham County
Hole No. Ef-1

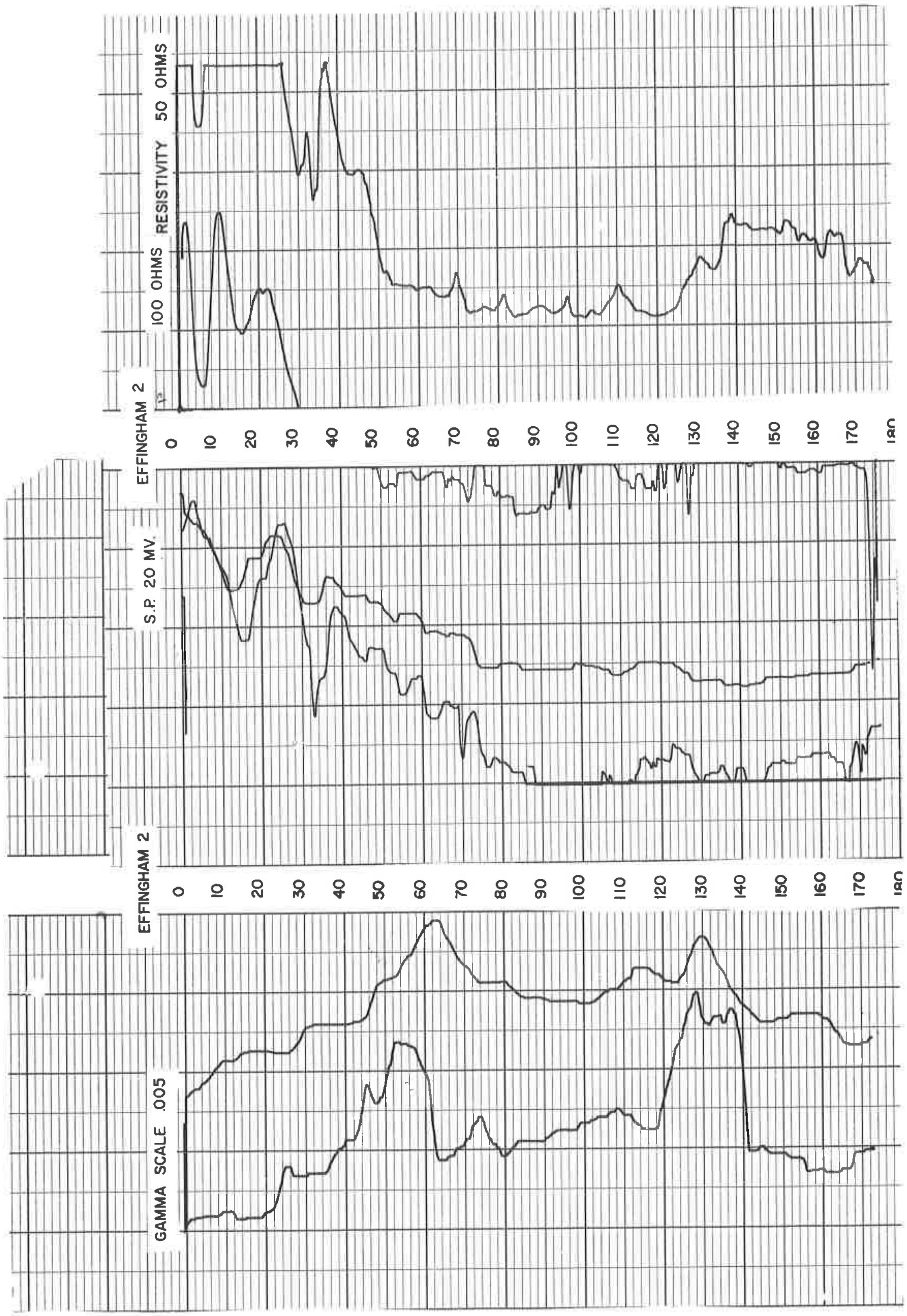

Figure EF-3. Electric and Gamma-Ray Logs - Effingham County
Hole No. Ef-2

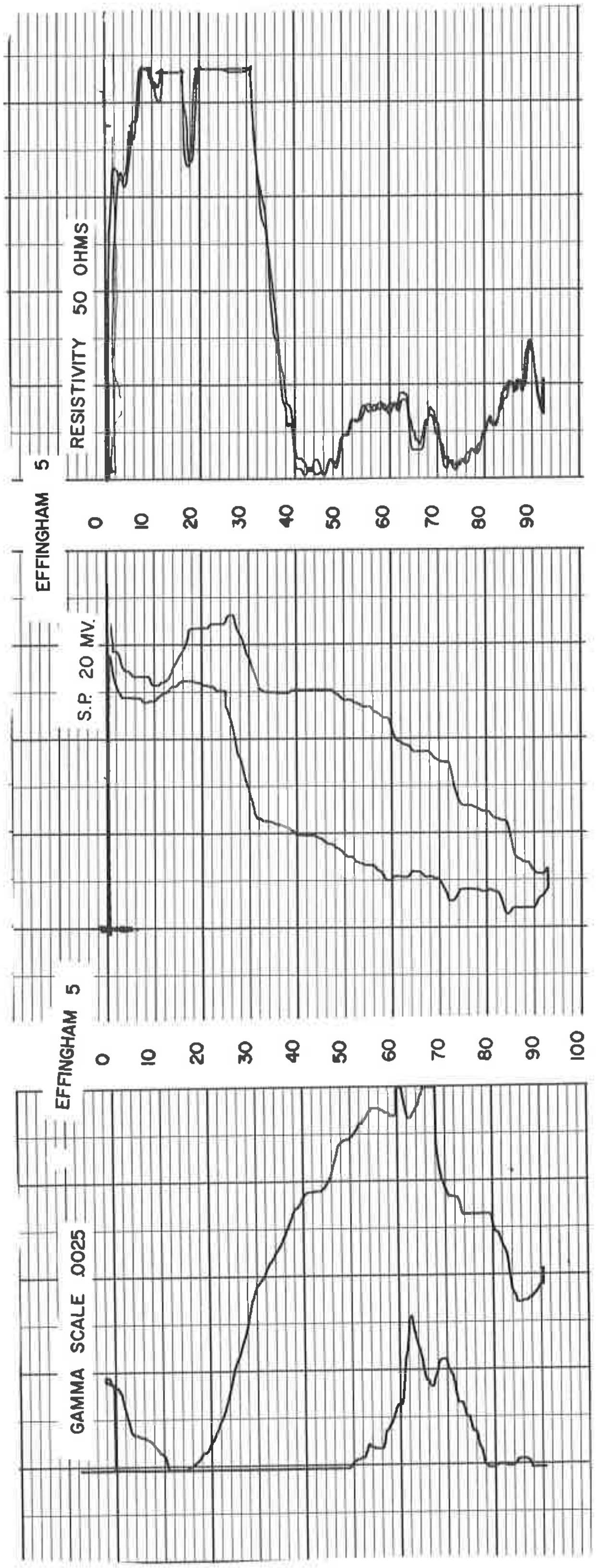

Figure EF-3. Electric and Gamma-Ray Logs - Effingham County
Hole No. Ef-5

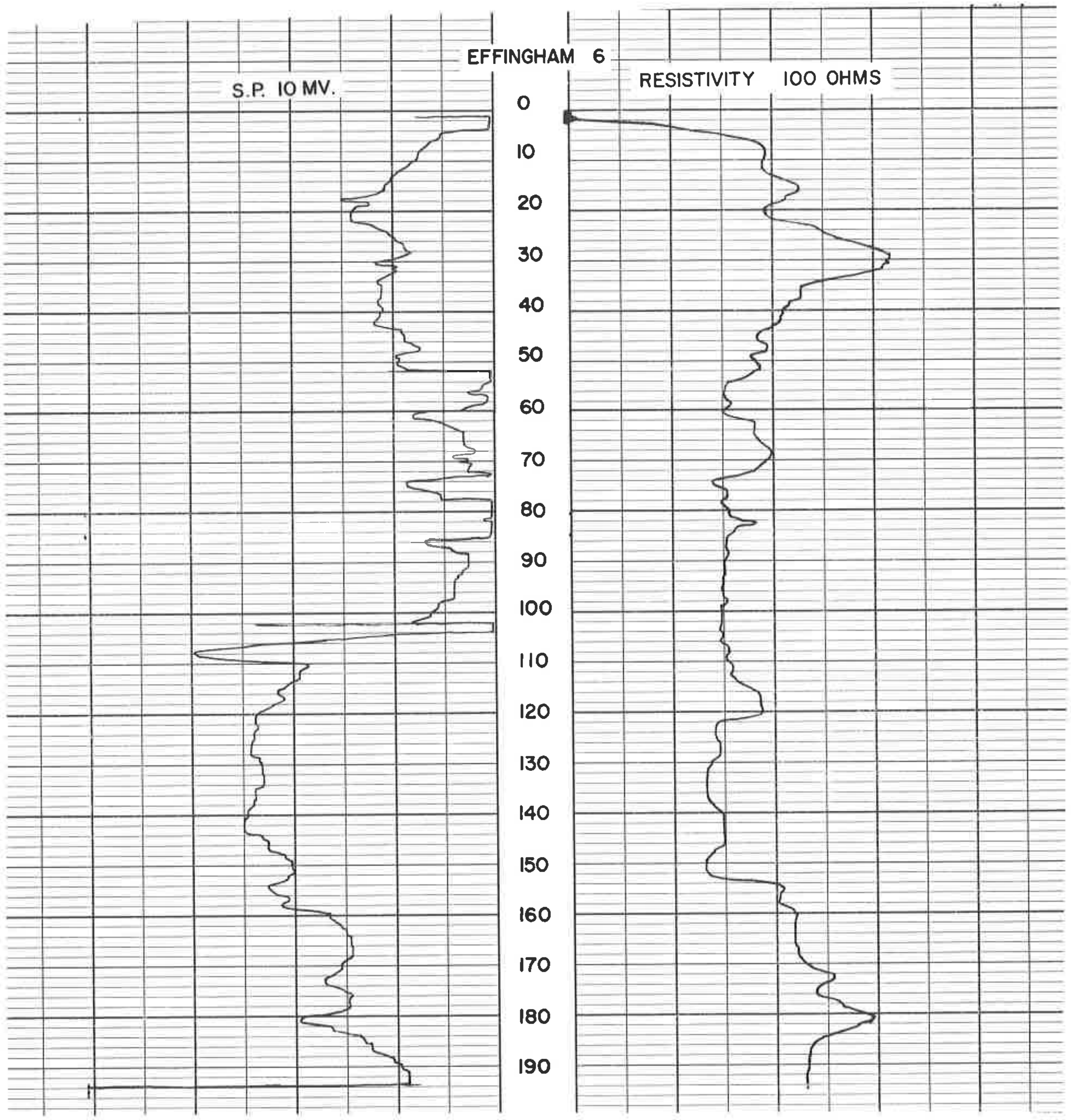

Figure EF-3. Electric and Gamma-Ray Logs - Effingham County
Hole No. Ef-6

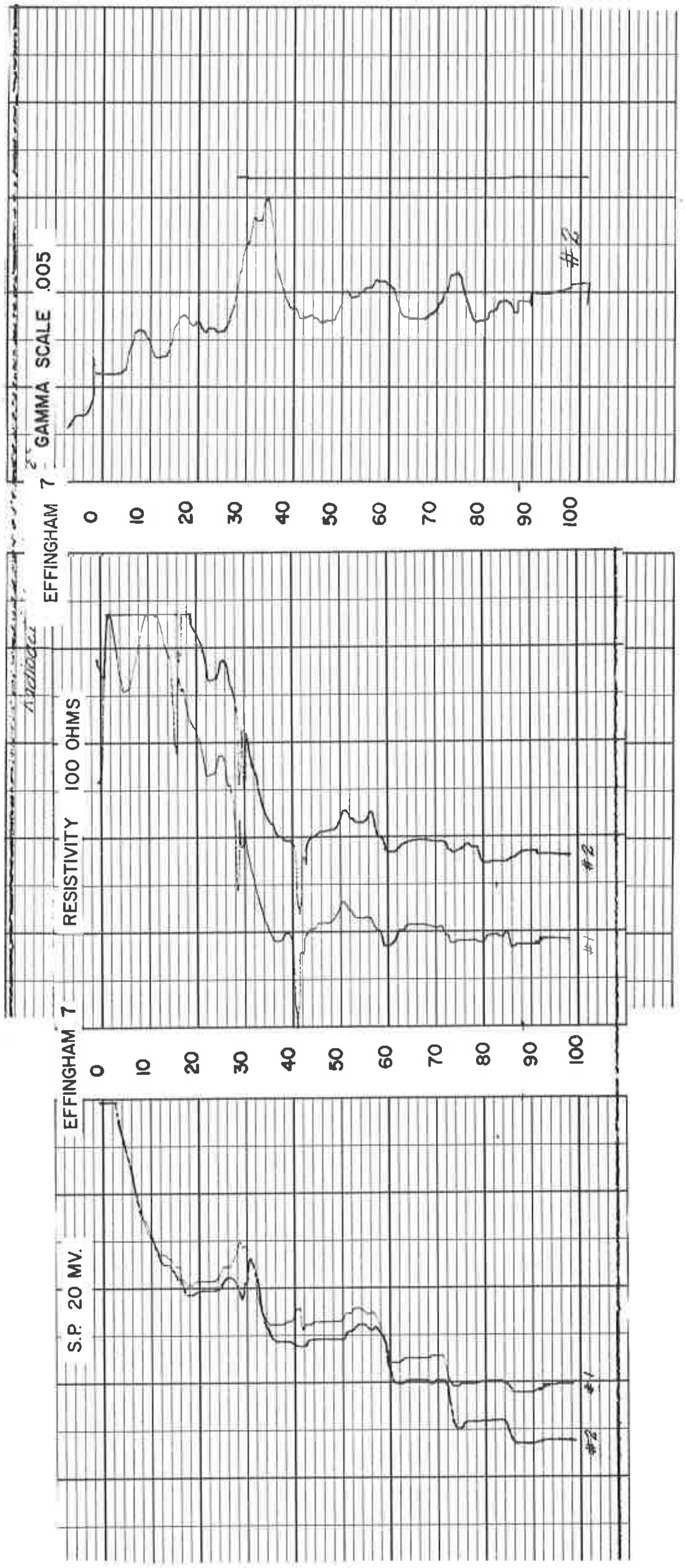

Figure EF-3. Electric and Gamma-Ray Logs - Effingham County
Hole No. Ef-7

TABLE EF-1

BPL DETERMINATION ON CORES

Effingham County

Hole No.	Surface Elevation (Sea Level) Feet	Depth Feet	Core Recovery Feet	Core Recovery %	BPL %
Ef-1	70	8-15	W.S.	-	0.0
		15-24	W.S.	-	0.0
		24-28	W.S.	-	0.0
		28-32	5	100+	0.0
		32-38	-	-	-
		38-42	4	100	0.8
		42-45	W.S.	-	0.0
		45-50	5	100	1.3
		50-55	5	100	0.4
		55-60	4.5	90	1.6
		60-65	4	80	1.8
		65-78	7	55	1.9
		78-93	9	60	4.9
		93-100	W.S.	-	6.4
Ef-2	63	0-10	1	10	0.0
		10-20	6	60	0.0
		20-25	2	40	0.0
		25-30	3	60	0.6
		30-40	6	60	0.3
		40-45	5	100	0.4
		45-50	10	100	0.4
		50-55	5	100	0.4
		55-60	5	100	1.3
		60-65	5	100	4.4
		65-70	5	100	0.7
		70-75	5	100	1.3
		75-80	5	100	1.2
		80-85	5	100	1.1
		85-90	5	100	1.7
		90-95	5	100	3.0
		95-100	5	100	1.4
		100-105	5	100	1.5
		105-110	5	100	2.6
		110-115	5	100	2.0
		115-120	5	100	1.4
		120-125	5	100	6.7
		125-135	6	60	3.6
		135-145	8	80	2.5

(Continued)

TABLE EF-1 (Continued)

BPL DETERMINATION ON CORES

Effingham County

Hole No.	Surface Elevation (Sea Level) Feet	Depth Feet	Core Recovery		BPL %
			Feet	%	
Ef-2 cont.	63	145-150	5	100	4.0
		150-155	5	100	-
		155-160	5	100	3.8
		160-165	5	100	3.0
		165-168	3	100	2.2
Ef-3	60	0-5	-	-	0.0
		5-10	-	-	0.0
		10-18	2.5	30	0.0
		18-25	7	100	1.4
		25-30	5	100	0.0
		30-35	5	100	1.1
		35-40	4	80	3.7
		40-46	6	100	4.1
		46-51	5	100	3.5
		51-56	5	100	3.6
		56-61	5	100	1.7
		61-66	5	100	1.5
		66-71	5	100	2.0
		71-76	5	100	1.4
		76-78	2	100	2.9
		78-83	5	100	2.7
		83-88	5	100	3.5
		88-93	5	100	4.7
		93-98	5	100	2.2
		98-103	5	100	9.3
		103-108	5	100	1.8
		108-113	5	100	1.9
		113-118	5	100	2.0
		118-123	5	100	2.6
		123-128	5	100	3.2
		128-133	5	100	3.5
		133-138	5	100	4.8
		138-143	5	100	3.0
		143-148	5	100	3.0
		148-153	5	100	3.8
		153-158	5	100	5.5
		158-163	5	100	4.9

(Continued)

TABLE EF-1 (Continued)

BPL DETERMINATION ON CORES

Effingham County

Hole No.	Surface Elevation (Sea Level) Feet	Depth Feet	Core Recovery Feet	Core Recovery %	BPL %
Ef-3 cont.	60	163-173	5	50	7.0
		173-183	5	50	4.7
		183-193	7	70	0.0
		193-203	7	70	0.0
		203-205	2	67	2.3
		205-213	8	100	0.0
		213-223	2	20	0.0
		223-233	3	30	7.4
		233-243	2.5	25	0.0
		243-253	10	100	0.0
		253-263	8	80	0.0
		263-273	6	60	0.0
		273-283	5	50	0.0
		283-293	4	40	0.0
Ef-4	52	0-5	5	100	0.0
		5-10	5	100	0.0
		10-15	3	60	0.0
		15-20	3	60	0.0
		20-25	2	40	0.0
		25-30	2	40	0.0
		30-35	5	100	0.0
		35-40	5	100	0.4
		40-45	5	100	1.7
		45-50	5	100	0.8
		50-55	5	100	0.9
		55-60	5	100	1.5
		60-65	5	100	2.4
		65-70	5	100	1.7
		70-75	5	100	1.4
		75-80	5	100	1.4
		80-85	5	100	1.2
		85-90	5	100	1.8
		90-96	6	100	2.5
		96-100	4	100	1.3

(Continued)

TABLE EF-1 (Continued)

BPL DETERMINATION ON CORES

Effingham County

Hole No.	Surface Elevation (Sea Level) Feet	Depth Feet	Core Recovery Feet	Core Recovery %	BPL %
Ef-5	70	0-5	W.S.	-	0.0
		5-10	W.S.	-	0.0
		10-20	8	80	0.0
		20-23	3	100	0.0
		23-28	5	100	0.7
		28-33	5	100	1.4
		33-38	5	100	1.2
		38-43	5	100	1.2
		43-48	5	100	2.5
		48-53	5	100	2.4
		53-55	2	100	3.4
		55-65	8	80	0.2
		65-70	5	100	0.7
		70-75	5	100	0.9
		75-80	5	100	2.2
		80-85	5	100	1.6
		85-90	5	100	2.0
		90-98	8	100	0.3
Ef-6	50	0-5	W.S.	-	0.0
		5-10	W.S.	-	0.0
		10-15	4	80	0.0
		15-20	2	40	0.0
		20-25	4	80	0.7
		25-30	2	40	0.6
		30-35	5	100	2.3
		35-40	3	60	4.2
		40-45	3	60	4.6
		45-50	3	60	4.8
		50-55	5	100	3.6
		55-60	5	100	2.9
		60-65	5	100	2.6
		65-70	5	100	3.7
		70-75	5	100	2.6
		75-80	5	100	3.8
		80-85	5	100	3.6
		85-90	5	100	2.4
		90-95	5	100	1.2

(Continued)

TABLE EF-1 (Concluded)

BPL DETERMINATION ON CORES

Effingham County

Hole No.	Surface Elevation (Sea Level) Feet	Depth Feet	Core Recovery Feet	Core Recovery %	BPL %
Ef-6 cont.	50	95-100	5	100	0.8
		100-105	5	100	0.7
		105-115	8	80	1.4
		115-125	9	90	3.4
		125-135	8	80	2.9
		135-145	9	90	0.8
		145-150	5	100	1.2
		150-155	5	100	1.8
		155-165	6	60	0.3
		165-170	5	100	0.0
Ef-7	119	0-5	W.S.	-	0.0
		5-10	W.S.	-	0.0
		10-15	4	80	0.0
		15-20	4	80	0.0
		20-25	3.5	70	0.0
		25-30	5	100	0.7
		30-40	7	70	4.7
		40-45	3	60	3.5
		45-55	3	30	2.6
		55-60	4	80	4.2
		60-65	2	40	5.2
		65-70	3	60	2.7
		70-80	6	60	3.8
		80-90	8	80	3.0

Heavy Minerals and Sands

Table EF-1 shows that Effingham County contains extensive deposits of phosphorites but with a concentration such that they are not of economic interest for current phosphate mining procedures. Hence, no matrix samples were beneficiated for phosphate recovery data.

Examination of the cores and their lithology directed attention to an evaluation of the sands and heavy minerals. Similar examination of cores from counties previously drilled showed that a number deserved similar evaluation.

Accordingly, the sands and heavy minerals in about the top 50 feet of cores from Effingham County and selected cores from other counties have been processed, analyzed, and the results compiled and presented as Appendix I to this report.

APPENDIX I

SANDS AND HEAVY MINERALS

Introduction

While some data have been given in preceeding Project Reports on occurrence of heavy minerals and sand, an economic evaluation has not been presented. It appeared from early results of Effingham County operations that most promise of economic potential utilization of its mineral resources would be in its sands, and perhaps heavy minerals. In order to present more comprehensive information, intervals of cores from other counties having similar potential were selected, processed, and the results, compiled as reported herein.

Economic Criteria

Sand

The economy of the sand industry depends primarily upon the construction, highway, and glass industries. The outlook for Georgia for the next decade is for an increasing demand. Sands are widely distributed throughout Georgia. In 1960, there were 33 companies in 22 counties producing commercial sands: building, filter, blast (abrasive), foundry and molding, glass, paving, fill, and others.

There are so many sand uses under broad size distribution specifications, with quality factors expressed in generalized terms, that a complete listing in this report is not feasible. In fact, one wishes he could consider a specification as the one attributed to Vitruvius Pollio in the first century B.C. (see p. 780, Bulletin 630, U. S. Bureau of Mines, 1965) who for a mortar sand said: "throw some sand upon a white garment and then shake it out, if the garment is not soiled and no dirt adheres to it, the sand is suitable." For some uses of sand and aggregate, specifications include tests for soundness (abrasion resistance), freedom from deleterious particles, size gradation, adhesion of bitumens, and many others.

For "ordinary" sands, relative freedom from clays, size gradation, deposits within about 50 feet of the surface, and proximity of market are the major factors influencing economic utilization. For "glass" sands, quality characteristics are rather rigidly specified, with uniformity being of importance as regards both quality and size gradation.

Examples of "generalized" quality specifications as shown in the U. S. Bureau of Mines Bulletin 630, 1965, are:

Highway construction sands: clean, hard, and free from clay. Totally free from organic impurities if for concrete pavement. For bituminous paving, small amounts of organic material are allowable.

Filtration sands: uniform grade, free from clay, loam, dust, or other foreign matter, and must possess good permeability.

Blast sands: ability to resist shattering and uniform grade are most important.

Glass sands: less than 20 mesh.

Flat, Table, and Optical: 99+ percent silica, with minimum of coloring substances such as iron or chromium and of uniform grade. High percentages of alumina, calcium, or magnesium oxides are undesirable.

Common colored-glass: less rigid specifications and may contain as much as one percent of coloring materials.

An attempt is made below to show representative specifications to permit preliminary evaluation of the sands reported herein.

Construction sands:

Size Gradations

Weight Percent Passing Through Indicated Screen Size

Screen	Concrete(1)	Mortar(2)	Fine Aggregate(3) For Underdrain	Roadway Materials(4) I	II	III
3/8 in.			100			
4 mesh	95-100	100				
16 mesh	45-95	90-100	45-95			
50 mesh	8-30	15-40	0-8			
60 mesh				15-85		
100 mesh	1-10	0-10				
200 mesh				0-35	0-45	0-75

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(1) Of natural or manufactured (ground) sand of not more than 1% clay lumps; 1% coal, shale, soft or flaky material; nor more than 1% total detrimental substances. Clay and silt up to 3% permitted if evenly distributed. Color must not exceed "standard."

(2) Natural or manufactured sand: shall have hard, clean, durable, uncoated surfaces; detrimental substances: same as (1).

(3) Natural sand or siliceous sandstone of hard, durable, rounded to angular particles with no flat or elongated particles.

(4) The percent clay may be 0-16% for Type I and Type II.

Foundry sands:

According to "Foundry Sand Handbook (American Foundrymen's Society - 1953 Edition)," there is no generally accepted purchase specification for foundry sand. Some specifications may call for fineness test, moisture content, permeability, green strength, clay percent, etc. Methods of testing have been adopted. "AFS Clay" refers to particles "which, when suspended in water, fail to settle one inch per minute and which consists of particles less than 20 microns...in diameter," i.e., 625 mesh.

"Standard" sands have the following size distribution:

U. S. Standard Sieve No.	Weight Percent Retained Standard	Weight Percent Retained Secondary Standard
20		0-1
30		0-1
40	0	0-1
50	0-5	2-4
70	95-100	18-25
100	*	32-43
140		23-29
200		6-11
270		0-2
Pan		0-1

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*Any particle passing 70 mesh shall be retained on 100 mesh.

Type of Sand	Silica Content	"AFS Clay" Content
Molding		8-20
Silica sand	95% min.	
Bank sand		5 max.
Dune sand	90% min.	

Glass sands:

Uniformity of chemical composition and, somewhat less important, size distribution from shipment to shipment is a "quality" highly desired by industry. This means that relatively large and uniform deposits are needed and that mining programs should be planned on the basis of extensive sampling of the deposits to meet the desired uniformity of products. The data below are from "Silica for Glass Manufacture in South Carolina," Buie, B. F., and Robinson, G. C., Bulletin 23, 1958, Division of Geology, State Development Board, Columbia, South Carolina.

Chemical Composition

		Suggested Specifications of American Ceramic Society			
		SiO_2	Al_2O_3	Fe_2O_3	CaO+MgO
Quality	Use	Minimum	Maximum	Maximum	Maximum
First	Optical	99.8	0.1	0.02	0.1
Second	Flint, glass, and tableware	98.5	0.5	0.035	0.2
Third	Flint	95.0	4.0	0.035	0.5
Fourth	Sheet, rolled, polished plate and window	98.5	0.5	0.06	0.5
Fifth	Same as fourth	95.0	4.0	0.06	0.5
Sixth	Green containers	98.0	0.5	0.3	0.5
Seventh	Green glass	95.0	4.0	0.3	0.5
Eighth	Amber glass	98.0	0.5	1.0	0.5
Ninth	Amber glass	95.0	4.0	1.0	0.5

The Georgia Department of Mines, Mining, and Geology's Information Circular No. 11, 1960, presents results of 600 analyses of different glass sands being used by glass manufacturers. Some of the ranges of components within these sands, with comments, were:

Component	Range Percent by Weight	Comments
SiO_2	88.5 - 99.7	
Fe_2O_3	0.05- 0.3	As high as 7% used in bottle manufacture
Al_2O_3	0.1 - 0.7	2.2% was average for bottles, but 6-10% may be used.

Quoting the same American Chemical Society chemical specifications, size specifications are shown in "The High Silica Resources of Tennessee," R. E. Hershey, Tennessee Division of Geology, Report of Investigation No. 10, 1960.

Mesh	Percent by Weight
On 20	0
On 40	40-60
On 60	30-40
On 100	10-20
Thru 100	0-5

Poole* indicates that minus 150 mesh material can be used. For fiber-glass, the Owens-Corning Fiberglass Corporation has expressed a preference for material of which 99.5 percent will pass 200 mesh and 95.5 percent will pass 325 mesh (ct. Buie and Robinson, ibid, p.11).

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*Poole, J.P., "Glass Sand Beneficiation Process." Jour. Amer. Ceram. Soc. 32, pp. 237-241, 1949.

Current specifications of both size and chemical composition were available from a few manufacturers and are shown in the following table:

INDUSTRIAL GLASS SAND SPECIFICATIONS
(Representative)

Screen Analysis

Mesh	Percent by Weight		
On 20		.1 max	
On 30	.7	5.0 max	none
On 40	10.1	20.0 max	3.0 max
On 50	38.7		
On 70	39.8		
Thru 80			35.0 max
On 100	0.9	15.0 max	
Thru 140			5.0 max

Chemical Analysis (1)

Component	Percent by Weight		
Fe_2O_3	0.19	.03 max(2) .15 max(3)	.025 max
Al_2O_3	.12	*	
$Fe_2O_3+Al_2O_3$			.5 max
TiO_2	.009		.03 max
CaO	Trace		
MgO	Trace		
CaO+MgO			.10 max
Organics			Trace
Loss on Ignition (LOI)	.08		
SiO_2	99.88	99.3 min	Bal.

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*As low as possible and controllable.

(1) Other impurities: coloring minerals and refractory materials, as low as possible and controllable. For example, refractory particles, such as sircon and kyanite, from a 500 gram sample should not have over 2 particles on the 40 mesh screen, nor more than 19 particles on the -40+60 mesh screen.

(2) For crystal glass.

(3) For colored glass.

It is obvious that manufacturers differ widely in their specifications, which makes it very difficult to present an evaluation of the economic potential of a given deposit. It does appear that uniformity of size and composition is important; but this can be determined only through extensive core drilling of a deposit.

Heavy Minerals

"Heavy minerals"--those with a specific gravity of over about 2.8--include many minerals. Feasibility of economic utilization involves consideration of all minerals present in the mixture, and separations into useful concentrates of a specific mineral may be quite complex. Current practice generally involves the sands in the size range of 35 to 150 mesh. Industrial attention in Georgia has been focused on (1) the titanium group: ilmenite, leucoxene, and rutile (35-100% TiO_2), (2) zircon (67% ZrO_2) and at times a source of hafnium; (3) monazite--amisture of rare earths and also a source of thorium oxides.

There do not seem to be well-defined criteria for the economic evaluation of a heavy mineral bearing sand deposit. Generalized standards employed by industry for titanium-rich heavy mineral sands in the past have been:

1. The sand should contain a minimum of one percent titanium dioxide.

2. In most cases, the total heavy mineral content of a typical Georgia heavy mineral sand must be more than three percent of the total sand to meet standard no. 1.

3. The titanium concentrate to be sold should be at least half the heavy mineral fraction of the sand.

4. The titanium concentrate should contain at least 60-65 percent titanium dioxide.

Thickness of overburden also is an important factor.

The demand and prices for specific minerals fluctuate widely. Thorium, europium, and vanadium are in increasing demand. It is not feasible, however, to set forth economic criteria of explicit value for this reconnaissance investigation. Attention herein was paid to the composition of "sands" occurring within about 50 feet of the surface and in the particle size range of 35 to 150 mesh.

Experimental

Procedure

To obtain data for preliminary evaluation of economic potential, core samples were selected on the basis of lithologic descriptions and prior chemical analyses. The samples were washed free of the fine clays, screened, and the -35+150 mesh material--the size range of interest--used for evaluation. It was considered that only those layers which contained "sands" of at least 70 percent plus 150 mesh material would be of possible economic interest. This arbitrary "cut-off" was used in selecting matrices for study.

A sample of the -35+150 mesh fraction was chemically analyzed; another sample was examined visually and microscopically for mineral species contaminants. Size distribution was obtained by screening with 35, 48, 65, 100, and 150 mesh screens. Of the cores available, the sample from Ef-2 was considered as having most promise for possible rare earth materials and a sample was analyzed by dc arc emission spectrography.

Results

The weight percent of "sands" in matrices and weight composition of washed -35+150 mesh fractions are given in Table AI-1. Inspection of the data from the same hole provides an estimation of the uniformity of particle sizes. For example, Ef-4 shows fairly uniform distribution in the washed -35+150 mesh material from surface to 35 feet and a rather sharp change to -100+150 mesh below 35 feet.

Visual

Visual and microscopic examinations were carried out to obtain a qualitative evaluation of the shape, color, and "purity" of the -35+150 mesh material. The information is presented in Table AI-2. In all samples reported herein, the shape of the sands are satisfactory with no, or but minor, elongated shapes. Observations as reported from visual and microscopic examinations require definition of the terms: "clear," "coated," and "colored."

"Clear": refers to relatively transparent quartz grains whose surfaces are not coated with other minerals or organic contaminants, and do not exhibit colored contaminants within the grain.

"Coated": refers to grains that appear stained with iron oxides, clays, occasionally organic matter, or other minerals.

"Colored": refers to clear, transparent grains that are contaminated with some mineral material that imparts a color to the grain: orange (or), reddish (red), yellow (y), tan (tan), white (wh), cream (cr), pale (pa), dark (dk), light (lt).

Spectrography

Semi-quantitative analyses of elements, including rare earths, the -35+150 mesh fraction of the core from hole Ef-2 at various depths are presented in Table AI-3. These data were obtained by arc emission spectrography. In most cases three lines of the spectra were examined for each element to preclude errors by interfering lines of other elements.

In the case of tungsten, two of the lines scanned were definitely due to tungsten, but the third line usually obtained was absent. Some of the results are surprising, to say the least. As examples, presence of tungsten, cobalt, and antimony, and the similar ranges for iron and aluminum.

Some additional preliminary analyses were carried out on samples from Ec-2 in the +35, -150+200, -200+325, and -325 mesh ranges to determine if elemental concentrations were a function of particle size, but results did not indicate desirability of more detailed study.

About all that can be said at this time is that the data must be considered as rough estimates, but that a few cases merit further attention.

Chemical

Chemical analyses of the processed -35+150 mesh sand fractions are given in Table AI-4 in terms of the compounds usually contained in commercial specifications for glass sand. The table is arranged to facilitate addition of compounds where their combined value is sometimes expressed in specifications; e.g., "Iron and Aluminum" (Fe_2O_3+Al_2O_3) and "Calcium and Magnesium" (CaO+MgO). The indicated variation in "Loss on Ignition" (LOI) prompted microscopic examination of the samples. It was found that LOI's up to about 0.6 percent were related to the presence of trace organic matter and mica; while above 0.6 percent, the LOI was closely related to the amount of clay present.

Considerable, and to date, inexplicable, difficulties were experienced in the determinations of silica in high grade sands. Colorimetric procedures, in accordance with the U.S.G.S. Bulletin No. 1144A (considered to be the "Bible" for analyses of rocks and minerals) and calibrated using NBS 70a sample, results in obviously high SiO_2 analyses; even to over 100 percent in some samples. Fresh samples were taken and run by both the U.S.G.S. colorimetric method and by gravimetric methods. Results from gravimetric procedures gave 2 to 4.8 percent lower values. A reagent grade silicic acid was analyzed and the two methods checked within 0.3 percent. The discrepancy noted in the "natural" sand samples is thought to be due to very small amounts of phosphates, and perhaps arsenic, present; each of which would increase the apparent silica as determined by the colorimetric method.

The values shown for SiO_2 in Table AI-4 have been adjusted to conform with gravimetric procedures. It is considered that the reported values are correct, but may, if anything, be one or two percent low.

Summary

A. Potential Glass Sand Use

Deposits which merit attention are limited through a review of the data above on the bases of:

1. Quality specifications as listed under the paragraph "Glass Sands" above.

2. Application of an arbitrary limit that the matrix contains at least 70 percent by weight of sands larger than 150 mesh.

3. Uniformity - No definite figures as to the degree of "uniformity" are available, so this must be expressed as judgement-qualitative factor.

As the specification of "at least 70 percent by weight of sands larger than 150 mesh" was arbitrarily chosen, layers which appeared to be of interest otherwise are included in the summary with the actual percent of +150 mesh material also listed.

Hole No	Layer Depth Feet	Percent +150 Mesh Sands In core	Meets Approx. Chemical Specifications For Quality No	Uniformity(1)	
				Chemical	Size
ATKINSON COUNTY					
At-1	0-29	75.2	3(2), 5(2), 7, 9	G	F+
At-3	5-20	52.7	7, 9	P	P
BEN HILL COUNTY					
BH-2	0-10	52.5	3(2), 5(2), 7, 9	G	G
BH-3	0-10	62.8	7(4), 9	F	G
BERRIEN COUNTY					
Be-1	10(5)-31	53.2	7, 9	F	G
Be-3	0-10	61.4	7, 9	P	G
EFFINGHAM COUNTY					
Ef-1	8(5)-15	98.6	9	-	-
Ef-2	0-10	98.1	5, 6, 7, 8, 9		
	20(5)-30	69.7	7, 9		
Ef-3	0-20	58.6	9(4)	F	P
Ef-4	0-35	64.1	7, 9	F	P
Ef-5	0-10	91.5	6(4), 7, 9	G	F
IRWIN COUNTY					
Ir-2	5-15	69.6	7(4), 9(4)	G	F
	15-35	30.8(7)	9(4)	G	F
Ir-3	0-16	94.8	6, 7, 8, 9	G	G
LOWNDES COUNTY					
Lo-4	0-10	86.1	5, 7, 9	G	G
	10-15	68.1	7, 9	-	-
	15-20	78.7	7, 9	-	-

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(1) G=good; F=fair; P=poor
(2) Fe_2O_3 somewhat high for quality number
(3) Thin layer
(4) SiO_2 may be somewhat low
(5) Note overburden to be removed
(6) Practically all +35 mesh
(7) Finely sized sand

Several factors should be considered in evaluation of deposits of sands for glass manufacture:

a. Some deposits which exceed the arbitrary 70 percent of +150 mesh material may have a substantial amount in the +35 mesh size, which may require grinding to meet size specifications. (See Table AI-1.)

b. Sands showing relatively low quality grade specifications may be beneficiated by attrition scrubbing to remove surface contaminants and/or by flotation procedure to produce higher grade material. It is believed that at least second-quality grade could be produced from some of these deposits. Further laboratory investigations would be required to demonstrate this.

c. Transportation costs to a market may make a deposit uneconomical in the light of competition by similar or lower grade deposits which can be transported to the market at less cost.

d. Obviously, results from one hole does not indicate the areal extent of a deposit. The results do provide a guide to deposits that merit additional sampling to determine quantity and quality data of a deposit on which to base, with other factors, economics of utilization.

B. Other Sand Uses

By simple washing and screening to size distribution specifications, it is apparent that most of the sands would be suitable for relatively local use for highway construction, concrete and foundry use.

C. Heavy Minerals

The chance of interest in heavy minerals in the areas reported herein were slim but the reported occurrence of five "old beach-ridges" in Effingham County made it desirable not to overlook a possibility. Analyses of samples from Ef-2 indicate no feasibility of commercial utilization. (See Table AI-3.)

TABLE AI-1

PARTICLE SIZE - WEIGHT (%) DISTRIBUTION

	Layer Depth, Feet		Matrix				-35+150 Mesh Washed Fraction			
Hole No.	From	To	+35	-35 +150	-150 +400	-400	-35 +48	-48 +65	-65 +100	-100 +150
ATKINSON COUNTY										
At-1	0	5	23.7	45.5	11.2	19.6	25.9	28.1	24.6	21.3
	5	10	33.5	36.0	4.9	25.6	39.2	34.1	16.2	10.5
	10	23	26.8	51.4	5.7	16.1	26.3	36.2	23.3	14.2
	23	29	31.5	38.7	2.6	27.2	39.0	32.5	20.2	8.3
	29	31	8.8	19.7	7.5	64.0	14.7	24.9	25.7	34.7
At-2	0	5	40.6	29.5	8.7	21.2	28.6	27.1	22.2	22.1
	5	10	44.4	24.5	0.8	30.3	19.2	29.0	28.7	23.1
	10	20	23.9	26.6	12.0	37.5	15.0	20.5	29.3	35.2
At-3	0	5	27.4	47.7	7.9	17.0	31.0	26.7	23.0	19.3
	5	10	10.2	35.2	9.2	45.4	22.2	26.4	26.3	25.2
	10	20	28.2	28.2	8.6	35.0	40.0	27.9	17.4	14.7
	20	25	22.9	29.5	5.1	42.5	41.3	28.6	17.9	12.2
BEN HILL COUNTY										
BH-1	0	5	20.9	53.8	5.9	19.4	21.7	25.8	23.7	28.8
	5	10	18.8	50.0	12.1	19.1	25.6	27.4	22.8	24.2
	10	18	17.5	54.8	9.6	18.1	26.0	26.9	25.6	21.5
	18	30	14.6	45.7	7.7	32.0	28.0	32.2	23.1	16.7
BH-2	0	5	43.0	18.0	12.1	26.9	36.6	30.7	19.6	13.1
	5	10	16.4	27.6	5.7	50.3	30.1	31.7	24.6	13.6
BH-3	0	5	14.5	46.5	7.0	32.0	21.5	29.0	26.8	22.7
	5	10	18.8	45.8	8.4	27.0	18.7	30.3	31.3	19.7
BERRIEN COUNTY										
Be-1	0	5	27.9	44.6	10.0	17.5	24.8	28.1	25.7	21.4
	5	10	16.4	55.5	3.8	24.8	18.9	26.4	26.2	28.5
	10	22	12.2	48.5	10.0	29.3	18.2	27.2	28.8	25.8
	22	31	10.8	32.5	14.2	42.5	22.9	26.9	24.2	26.0
Be-2	0	5	15.2	33.0	10.7	41.1	20.6	26.5	25.7	27.2
	5	10	3.3	7.7	15.0	74.0	12.5	18.7	22.5	46.3
	10	15	6.7	38.9	12.1	42.3	11.8	18.9	28.1	41.2

(Continued)

TABLE AI-1 (Continued)

PARTICLE SIZE - WEIGHT (%) DISTRIBUTION

Hole No.	Layer Depth, Feet		Matrix				-35+150 Mesh Washed Fraction			
	From	To	+35	-35 +150	-150 +400	-400	-35 +48	-48 +65	-65 +100	-100 +150
Be-3	0	5	30.7	38.5	12.9	17.9	24.5	26.8	23.9	24.8
	5	10	26.1	27.6	11.1	35.2	22.5	26.0	24.7	26.8
	10	19	10.0	25.8	11.8	52.4	14.5	20.0	27.1	38.4
EFFINGHAM COUNTY										
Ef-1	8	15	95.2	3.4	0.1	1.3	not enough material for screen analyses			
	38	42	6.2	61.8	16.3	15.7	1.1	4.1	26.0	68.8
	45	49	5.7	72.9	17.7	3.7	1.2	3.3	24.5	71.0
Ef-2	0	10	63.8	34.3	0.5	1.4	not enough material for screen analyses			
	10	20	24.1	51.6	7.6	16.7	18.3	20.1	20.6	41.0
	20	30	36.2	33.5	12.2	18.1	37.0	38.7	20.6	3.7
	30	40	38.3	46.3	2.3	13.5	26.2	22.9	30.7	20.2
	40	45	34.0	37.8	5.2	23.0	16.6	27.8	41.7	13.9
	45	50	38.3	44.1	6.1	11.5	4.6	13.7	41.6	40.1
Ef-3	0	5	17.2	34.6	7.9	40.3	10.9	12.2	28.8	48.1
	5	10	2.2	36.7	5.2	55.9	2.0	6.0	45.6	46.4
	10	20	4.5	67.3	5.3	22.9	2.8	15.2	38.1	43.9
	20	30	12.6	29.4	35.3	22.7	7.1	6.6	9.0	77.3
Ef-4	0	5	14.4	52.3	5.5	27.8	15.7	21.0	25.0	38.3
	5	10	19.4	36.2	6.8	37.6	16.7	21.4	34.2	27.7
	10	20	15.7	54.1	6.9	23.3	18.5	25.4	26.6	29.5
	20	30	36.3	34.2	6.2	23.3	26.2	15.6	25.1	33.1
	30	35	17.0	29.3	18.6	35.1	25.9	20.6	12.8	40.7
	35	40	0.9	59.3	24.5	15.3	2.1	6.3	21.0	70.6
Ef-5	0	5	56.5	35.0	1.6	6.9	31.4	27.6	21.7	19.3
	5	10	49.7	44.0	0.9	5.4	27.8	32.6	25.6	14.0
	10	23	9.3	63.2	12.8	14.7	4.7	10.4	34.9	50.0
	23	33	0.0	74.9	14.0	11.1	0.5	3.9	35.0	60.6
	33	43	0.0	72.1	16.2	11.7	0.7	4.1	34.1	61.1
	43	48	0.0	63.3	23.0	13.7	1.6	4.3	24.2	69.9

(Continued)

TABLE AI-1 (Concluded)

PARTICLE SIZE - WEIGHT (%) DISTRIBUTION

Hole No.	Layer Depth, Feet		Matrix				-35+150 Mesh Washed Fraction			
	From	To	+35	-35 +150	-150 +400	-400	-35 +48	-48 +65	-65 +100	-100 +150
Ef-6	0	5	67.4	23.3	2.1	7.2	40.8	31.8	17.1	10.3
	5	10	66.1	14.8	3.0	16.1	42.7	30.5	16.0	10.8
	10	20	19.7	46.7	11.8	21.8	23.2	34.6	26.3	15.9
	20	30	16.8	54.4	6.5	22.3	9.5	13.4	41.7	35.4
	30	40	12.7	72.3	3.2	11.8	17.1	31.5	34.6	16.8
	40	51	2.7	76.1	4.0	17.2	15.9	33.1	35.7	15.3
Ef-7	0	5	9.1	59.1	8.8	23.0	10.9	20.5	34.1	34.5
	5	10	8.1	63.0	5.2	23.7	10.3	21.4	35.0	33.3
	10	15	6.0	58.9	5.9	29.2	10.5	26.0	37.9	25.6
	15	20	6.8	63.2	9.1	20.9	10.5	23.7	29.0	36.8
	20	28	8.9	61.9	10.9	18.3	13.7	28.0	37.0	21.3
	28	33	2.8	74.9	6.1	16.2	4.0	27.7	44.4	23.9
IRWIN COUNTY										
Ir-1	0	5	25.9	21.4	11.7	41.0	23.0	24.6	23.5	28.9
	5	10	6.0	21.1	14.5	58.4	15.0	21.0	26.4	37.6
	10	15	8.0	21.0	13.7	57.3	16.1	21.0	26.7	36.2
	15	20	7.1	22.9	19.4	50.6	15.9	20.2	24.8	39.1
	20	31	25.8	27.3	8.7	38.2	35.7	27.0	18.0	19.3
Ir-2	0	5	12.4	27.6	11.5	48.5	16.0	19.4	27.7	36.9
	5	10	24.6	39.5	4.3	31.6	35.7	35.4	18.7	10.2
	10	15	37.1	38.1	4.2	20.6	48.3	33.9	11.8	6.0
	15	25	4.6	29.8	20.9	44.9	10.1	17.0	30.1	42.8
	25	35	0.0	27.3	22.2	50.5	9.9	18.6	28.9	42.6
Ir-3	0	5	28.5	68.3	0.8	2.4	51.0	32.8	12.3	3.9
	5	10	32.1	65.5	1.6	0.8	44.9	34.7	14.6	5.8
	10	16	28.9	61.4	4.9	4.8	33.0	40.5	15.7	8.5
	16	26	38.5	45.2	7.1	9.2	28.5	29.0	24.5	18.0
	26	30	26.4	53.2	7.9	12.5	19.2	27.8	33.1	19.9
	30	35	11.2	36.1	12.9	39.1	29.3	31.9	20.6	18.2
	35	50	19.8	42.8	10.5	27.0	30.8	31.7	20.5	17.0
LOWNDES COUNTY										
Lo-4	0	5	24.4	59.6	10.8	5.2	16.2	32.9	25.7	25.2
	5	10	20.1	68.1	11.3	0.5	20.7	32.2	23.3	23.8
	10	15	7.4	61.7	3.1	27.8	18.3	57.0	19.1	5.6
	15	20	11.2	67.5	3.7	17.6	22.0	46.8	20.4	10.8

TABLE AI-2

VISUAL AND MICROSCOPIC OBSERVATIONS

Note: Color abbreviations are: Br = brown, Cr = cream, Lt = light, Or = orange, Wh = white, Y = yellow

Hole No	Layer Depth (Feet) From	Layer Depth (Feet) To	+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction: Visual Color	Observations of -35+150 Mesh Fraction: Microscopic
ATKINSON COUNTY					
At-1	0	29	75.2	Or, Lt Gray to Wh	
	29	31	28.5	Or	
	0	5			Clear, coated, colored feldspar, some organic matter
	5	10			Clear, coated, colored, traces of feldspar and clay
	10	23			Mostly clear, some colored, sparse heavy minerals
	23	29			Same; some mica
	29	31			Mostly colored, some clear, some iron oxides, feldspar and organics
At-2	0	20	60.0	Y Or to Pale Or	
	0	5			Clear, coated, colored, iron oxides
	5	10			Clear, coated, colored, feldspar
	10	20			Mostly clear, some coated and colored, some feldspar, mica, and iron oxides

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet)		+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction	
	From	To		Visual Color	Microscopic
At-3	0	25	56.3	Pale Or to Lt Gray	
	0	5			Mostly clear, some coated or colored, feldspar and organics
	5	10			Clear, coated, colored, some feldspar
	10	20			Mostly clear, mod pink feldspar
	20	25			Mostly clear, some yellow coating, iron oxides
BEN HILL COUNTY					
BH-1	0	30	67.3	Buff to Cr	
	0	5			Many clear, some stained, iron oxides, organic matter
	5	10			Many clear, some stained, iron oxides, feldspar
	10	18			Clear, coated, abundant colored, feldspar, iron oxides
	18	30			Many clear, some coated and colored, feldspar, iron oxides, mica, heavy minerals
BH-2	0	10	52.5	Pale Or	
	0	5			Many coated and colored, clear, iron oxides, feldspar, organic matter
	5	10			Many coated, clear, iron oxides, feldspar

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet)		+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction	
	From	To		Visual Color	Microscopic
BH-3	0	10	62.8	Pale Y to Cr	
	0	5			Clear, many colored or coated, clay
	5	10			Clear, many colored or coated, some feldspar, mica
BERRIEN COUNTY					
Be-1	0	22	65.9	Pale Or to Lt Gray	
	22	31	43.3	Pale Buff	
	0	5			Clear and coated, some colored, some mica, heavy minerals
	5	10			Clear, coated and colored grains, some iron oxides, mica, heavy minerals
	10	22			Mostly clear and coated, some faintly colored, some heavy minerals
	22	31			Clear and coated, some colored, iron oxides, feldspar
Be-2	0	15	34.9	Buff to Lt Br to Buff	
	0	5			Clear, coated and colored, abundant iron oxides
	5	10			Mostly clear grains, large amount of iron oxide, mica, clay
	10	15			Mostly clear, clay, iron oxides , feldspar, mica

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet) From	To	+150 Mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction: Visual Color	Microscopic
Be-3	0	10	61.5	Buff to Pale Y	
	10	19	35.8	Or to Buff	
	0	5			Clear, coated and yellow colored, iron oxides, organic matter, heavy minerals
	5	10			Many coated, some clear, feldspar, iron oxides
	10	19			Most coated, abundant iron oxides, some mica, feldspar
EFFINGHAM COUNTY					
Ef-1	8	15	98.6	Clear	
	38	48	72.0	Cr-Gray	
	8	15			Majority clear, few coated, some heavy minerals
	38	42			Majority clear, few coated, scarce heavy minerals, mica, and clay present
	45	48			Clear, clay, mica, feldspar, phosphorite present
Ef-2	0	50	81.0	Or-Y to Buff	
	0	10			Clear, few coated with iron oxide, few feldspar and heavy minerals, some organic material
	10	20			Clear with many iron oxide coated, some vermiculite present, heavy minerals and feldspar

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet)		+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction	
	From	To		Visual Color	Microscopic
Ef-2 (Cont'd)	20	30			Clear, few coated, few colored, heavy minerals, scarce agglomerated with clay, feldspar and mica present
	30	40			Mostly clear, some coated and colored, heavy minerals, mica and feldspar present
	40	45			Mostly clear, more coated and colored than 30-40 ft. interval, feldspar and iron oxides more abundant than shallow depths; mica and heavy minerals present
	45	50			Mostly clear, clay, mica, iron oxides, feldspar, heavy minerals present
Ef-3	0	30	53.1	Or, Pink, Buff	
	0	5			Mostly clear, coated and colored, abundant, iron oxides, mica, feldspar present
	5	10			Coated and colored, mostly clear limonite and mica present
	10	20			Approximately half are coated, half clear, feldspar, mica, heavy minerals present
	20	30			Most clear or colored, few coated, clay, mica, some iron oxides

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet) From	Layer Depth (Feet) To	+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction: Visual Color	Observations of -35+150 Mesh Fraction: Microscopic
Ef-4	0	40	63.3	Pale Buff to Lt Gray	
	0	5			Clear, feldspar, limonite, heavy minerals present
	5	10			Mostly clear, few coated, few colored, mica, feldspar, iron oxides, heavy minerals
	10	20			Clear and coated, feldspar, mica, limonite, heavy minerals
	20	30			Clear and coated, feldspar, mica, iron oxides, very few heavy minerals
	30	35			Clear, coated and colored, large amounts of agglomerate of sand with iron oxide cement, feldspar, mica, heavy minerals
	35	40			Clear and coated, large amounts of clay, feldspar, mica, heavy minerals
Ef-5	0	48	76.2	Gray, Lt Gray	
	0	5			More clear than coated, feldspar some iron oxides, heavy minerals, mica
	5	10			Mostly clear, some with yellowish coating, feldspar, some mica
	10	23			Clear and coated, some clay, feldspar, limonite, mica

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet) From	Layer Depth (Feet) To	+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction: Visual Color	Observations of -35+150 Mesh Fraction: Microscopic
Ef-5 (Cont'd)	23	33			Mostly clear, some feldspar, mica, abundant grains of opaque dark gray mineral-clay
	33	43			Clear, abundant clay, mica, feldspar, some heavy minerals
	43	48			Clear, abundant clay, feldspar, heavy minerals
Ef-6	0	51	77.5	Buff, Lt Gray to Buff	
	0	5			Clear and coated, feldspar, mica, some heavy minerals
	5	10			Mostly clear, some yellow coated, feldspar, mica, heavy minerals
	10	20			Mostly clear, very few coated, very little iron oxides, abundant mica, some feldspar and heavy minerals
	20	30			Clear and coated, very few iron oxides, mica, many organic materials
	30	40			Clear, coated and colored, abundant clay, feldspar, mica, heavy minerals
	40	51			Clear, coated and colored, rounded gray (phosphate), feldspar, mica, heavy minerals

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet) From	Layer Depth (Feet) To	+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction: Visual	Observations of -35+150 Mesh Fraction: Microscopic
Ef-7	0	33	70.5	Buff, Clear, Clear to Lt Gray	
	0	5			Mostly clear, some colored, some mica and heavy minerals
	5	10			Clear, some feldspar, mica, traces organic material
	10	15			Clear, few colored, feldspar
	15	20			Clear, and colored, some feldspar, iron oxides, mica
	20	28			Clear, coated, and colored, feldspar, mica, heavy minerals
	28	33			Clear, coated, and colored, feldspar, mica, iron oxides
IRWIN COUNTY					
Ir-1	0	31	40.4	Y Brown to Pale Buff	
	0	5			Clear, many coated, abundant iron oxides
	5	10			Clear and coated, very abundant iron oxides
	10	15			Clear and slightly coated, abundant iron oxides, some feldspar
	15	20			Clear and coated and colored, iron minerals, feldspar
	20	31			Clear and coated, some iron oxides, feldspar

(Continued)

TABLE AI-2 (Continued)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet) From	Layer Depth (Feet) To	+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction: Visual Color	Observations of -35+150 Mesh Fraction: Microscopic
Ir-2	0	15	59.8	Or to Cr	
	15	35	30.9	Buff to Lt Gray	
	0	5			Most colored or coated, iron oxides
	5	10			Clear, many colored or coated, some iron oxides
	10	15			Most clear, some iron colored, some feldspar
	15	20			Most clear, some coated, iron oxides, feldspar, mica
	25	35			Almost all clear, some clay and mica
Ir-3	0	50	89.0	Pale Or, Clear, Lt Tan	
	0	5			Clear, many colored, some feldspar and organic matter
	5	10			Approximately half clear and half colored, some feldspar
	10	16			Mostly clear, very few coated, some feldspar, traces of iron oxides, heavy minerals
	16	26			Mostly clear, very few coated, feldspar, traces of heavy minerals
	26	30			Mostly clear, very few coated, feldspar, traces heavy minerals
	30	35			Mostly clear, clay, feldspar
	35	50			Mostly clear, feldspar, clay

(Continued)

TABLE AI-2 (Concluded)

VISUAL AND MICROSCOPIC OBSERVATIONS

Hole No	Layer Depth (Feet)		+150 mesh Sands in Core Weighted Average Percent	Observations of -35+150 Mesh Fraction	
	From	To		Visual Color	Microscopic
LOWNDES COUNTY					
Lo-4	0	20	80.0	Clear to Lt Gray-Clear	
	0	5			Clear, few colored, some feldspar
	5	10			Clear and coated, few colored, some organic matter
	10	15			Clear, scarce feldspar and heavy minerals
	15	20			Clear and coated, some clay

TABLE AI-3

ELEMENT ANALYSES OF +35-150 MESH FRACTION OF SANDS FROM HOLE Ef-2

(Results are in Weight Percent)

		Layer Depth, Feet				
Element	Symbol	10-20	20-30	30-40	40-45	45-50
Aluminum	Al	.3-.8	.05-.08			
Antimony	Sb		.005-.01			
Boron	B	.002-.005	.005-.01			
Calcium	Ca	.1-.3	.05-.1			
Chromium	Cr	.002-.005				
Cobalt	Co		.1-.5			
Copper	Cu	.005-.05	.005-.008			
Dysprosium	Dy		.001-.005			
Gadolinium	Gd					.005-.01
Hafnium	Hf			.005-.01		
Holmium	Ho			.005-.01		
Iron	Fe	.3-.8	.03-.08	.05-.1	.05-.1	.1-.3
Lead	Pb	.002-.005				
Luticium	Lu					
Magnesium	Mg	.1-.3	.05-.5			
Manganese	Mn	.1-.3	.05-.1			
Nickel	Ni		.05-.1			
Phosphorous	P	.08-.1	.001-.005			
Potassium	K	.002-.005				
Scandium	Sc	.002-.005				
Silicon	Si*	VS	VS	VS	VS	VS
Silver	Ag		.0001-.0005			
Sodium	Na					
Thorium	Th			.002-.005	.005-.05	.002-.005
Tin	Sn					
Titanium	Ti	.3-.8	.1-.3	.005-.05	.05-.1	.1-.3
Tungsten	W	.002-.005	.01-.1			
Vanadium	V	.002-.005				
Ytterbium	Yb					.005-.01
Zinc	Zn	.005-.05	.005-.001			

*Very Strong (VS) lines were obtained as expected. Actual weight percents were all over eighty percent, the data being given in Table AI-4.

TABLE AI-4

CHEMICAL COMPOSITION OF PROCESSED SANDS -35+150 MESH FRACTION

(Results are in Weight Percent)

Hole No.	Layer Depth, Feet From	To	Loss on Ignition (LOI)	SiO_2	P_2O_5	Fe_2O_3	Al_2O_3	CaO	MgO	TiO_2
ATKINSON COUNTY										
At-1	0	5	.26	94.5	.0	.08	.11	.0	.01	.0
	5	10	.24	94.5	.0	.16	.22	.0	.04	.0
	10	23	.15	94.5	.0	.16	.0	.0	.03	.1
	23	29	.17	94.5	.0	.18	.0	.0	.01	.1
	29	31	.23	94.5	.0	.05	.05	.0	.0	.0
At-2	0	5	.58	92.1	.0	.18	.05	.0	.03	.0
	5	10	.35	92.1	.0	.68	.18	.0	.02	.18
	10	20	.27	93.4	.0	.68	.11	.0	.0	.0
At-3	0	5	.15	94.5	.0	1.28	.32	.0	.03	.0
	5	10	.16	94.5	.0	.08	.02	.0	.0	.0
	10	20	.44	94.5	.0	.49	.17	.0	.0	.18
	20	25	.16	91.4	.0	.16	.43	.0	.01	.21
BEN HILL COUNTY										
BH-1	0	5	.17	97.5	.0	.14	.17	.0	.03	.1
	5	10	.17	92.6	.0	.13	.17	.0	.01	.0
	10	18	.05	92.6	.0	.11	.12	.0	.0	.1
	18	30	.06	92.6	.0	.09	.03	.0	.0	.18
BH-2	0	5	.30	95.5	.0	.26	.48	.0	.02	.3
	5	10	.07	94.1	.0	.28	.13	.0	.0	.1
BH-3	0	5	.31	95.5	.0	.31	.35	.0	.02	.1
	5	10	.14	92.6	.0	.23	.42	.0	.01	.1
BERRIEN COUNTY										
Be-1	0	5	.18	91.4	.15	.08	.08	.07	.01	.0
	5	10	.14	92.9	.0	.08	.0	.0	.01	.0
	10	22	.17	94.3	.0	.06	.24	.0	.01	.0
	22	31	.44	96.4	.0	.23	.28	.03	.01	.0

(Continued)

TABLE AI-4 (Continued)

CHEMICAL COMPOSITION OF PROCESSED SANDS -35+150 MESH FRACTION
(Results are in Weight Percent)

Hole No.	Layer Depth, Feet		Loss on Ignition (LOI)	SiO_2	P_2O_5	Fe_2O_3	Al_2O_3	CaO	MgO	TiO_2
	From	To								
Be-2	0	5	.38	95.3	.0	1.94	.24	.0	.01	.38
	5	10	1.63	88.7	.0	6.81	1.39	.0	.09	.5
	10	15	.53	96.6	.0	.61	.64	.03	.08	.22
Be-3	0	5	.17	96.4	.0	.24	.08	.0	.02	.1
	5	10	.25	94.5	.0	.96	.17	.0	.02	.0
	10	19	.30	92.1	.0	2.34	.36	.0	.01	.22
EFFINGHAM COUNTY										
Ef-1	8	15	.08	94.5	.0	.46	.26	.04	.02	.55
	38	42	.25	87.5	.0	.15	2.51	.1	.02	.28
	45	48	.71	92.6	.34	.84	5.58	.74	.22	.38
Ef-2	0	10	.12	97.6	.0	.07	.06	.0	.09	.34
	10	20	1.35	90.8	.0	.13	.19	1.67	.04	.0
	20	30	.44	95.4	.25	.23	.24	.29	.02	.0
	30	40	.0	91.0	.27	.19	.28	.0	.0	.0
	40	45	.27	95.6	.4	.22	.49	.1	.0	.0
	45	50	.35	92.1	.37	.17	1.82	.03	.02	.0
Ef-3	0	5	.13	94.1	.0	.42	.17	.0	.01	.34
	5	10	.13	92.6	.0	.7	.17	.0	.01	.49
	10	20	.11	95.5	.0	.26	.22	.0	.01	1.04
	20	30	1.97	77.7	.28	.99	4.90	.96	.69	.49
Ef-4	0	5	.05	92.6	.0	.16	.0	.0	.05	.38
	5	10	.19	92.6	.0	.36	.31	.0	.02	.38
	10	20	.64	95.5	.0	.26	.09	.0	.01	.14
	20	30	.04	91.4	.0	.23	.02	.0	.01	.24
	30	35	.42	95.2	.0	.82	.74	.01	.04	.34
	35	40	1.35	78.4	.17	.97	6.21	.7	.45	.55
Ef-5	0	5	.21	96.4	.0	.15	.11	.0	.01	.28
	5	10	.03	96.4	.0	.14	.02	.0	.01	.49
	10	23	.49	89.9	.0	.28	2.81	.1	.06	.55
	23	33	.45	85.1	.25	.38	4.28	.52	.09	.38
	33	43	.5	83.9	.37	.42	4.6	.68	.12	.49
	43	48	.91	82.8	.45	.54	5.07	.78	.29	.34

(Continued)

TABLE AI-4 (Concluded)

CHEMICAL COMPOSITION OF PROCESSED SANDS -35+150 MESH FRACTION

(Results are in Weight Percent)

Hole No.	Layer Depth, Feet From	To	Loss on Ignition (LOI)	SiO_2	P_2O_5	Fe_2O_3	Al_2O_3	CaO	MgO	TiO_2
Ef-6	0	5	.11	92.6	.0	.11	.02	.0	.01	.38
	5	10	.14	92.6	.0	.19	.17	.0	.01	.49
	10	20	.12	91.4	.0	.14	.08	.0	.01	.24
	20	30	.08	89.0	.0	.16	.08	.0	.01	.0
	30	40	.44	89.4	.96	.27	.96	.7	.09	.0
	40	51	.64	86.8	1.65	.22	1.60	1.11	.13	.0
Ef-7	0	5	.06	92.6	.0	.07	.08	.03	.01	.0
	5	10	.04	90.2	.0	.06	.0	.0	.06	.0
	10	15	.03	94.1	.0	.06	.44	.0	.02	.0
	15	20	.05	92.6	.0	.21	.06	.0	.02	.0
	20	28	.10	92.6	.0	.34	.09	.0	.02	.0
	28	33	.36	90.2	.4	.24	.85	.48	.04	.21
IRWIN COUNTY										
Ir-1	0	5	.61	92.6	.0	2.14	.48	.0	.01	.3
	5	10	.61	90.2	.0	3.46	.48	.0	.01	.38
	10	15	.33	90.2	.0	1.64	.29	.0	.0	.38
	15	20	.34	91.5	.0	.76	.18	.0	.03	.0
	20	31	.2	90.2	.0	.59	.6	.0	.0	.0
Ir-2	0	5	.28	90.2	.0	.86	.11	.0	.01	.0
	5	10	.18	95.5	.0	.28	.18	.0	.01	.0
	10	15	.15	92.6	.0	.11	.24	.0	.01	.0
	15	25	.27	92.6	.0	.44	.29	.0	.01	.0
	25	35	.68	95.5	.0	.47	.68	.0	.11	.0
Ir-3	0	5	.11	95.5	.0	.13	.0	.0	.0	.0
	5	10	.13	97.6	.0	.06	.0	.0	.0	.18
	10	16	.07	97.6	.0	.16	.03	.0	.01	.0
	16	26	.15	92.6	.0	.26	.04	.0	.01	.1
	26	30	.05	91.5	.0	.06	.0	.0	.0	.3
	30	35	.83	92.6	.0	.44	1.08	.01	.13	.0
	35	50	.36	92.6	.0	.24	.65	.0	.05	.0
LOWNDES COUNTY										
Lo-4	0	5	.11	95.5	.0	.07	.02	.0	.02	.16
	5	10	.05	97.5	.0	.05	.02	.0	.0	.0
	10	15	.09	94.5	.0	.08	.08	.0	.01	.0
	15	20	.23	94.5	.83	.25	.54	.0	.01	.1

APPENDIX II

ELECTRICAL RESISTIVITY AND GAMMA-RAY LOGS

HOLES LOGGED IN BROOKS, CAMDEN, CLINCH, ECHOLS, LANIER, LOWNDES, WARE COUNTIES

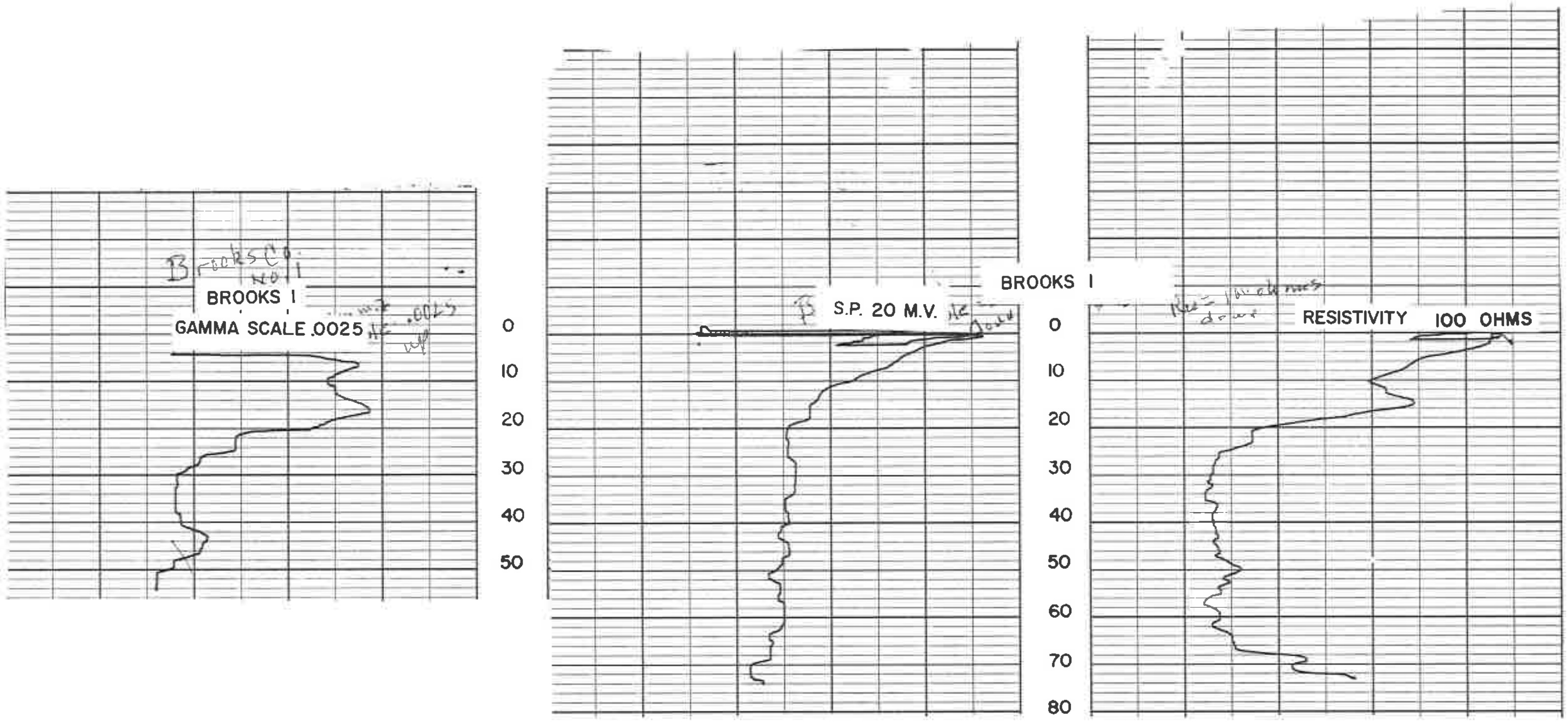

Figure AII-1. Electric and Gamma-Ray Logs - Brooks County
Hole No. Br-1

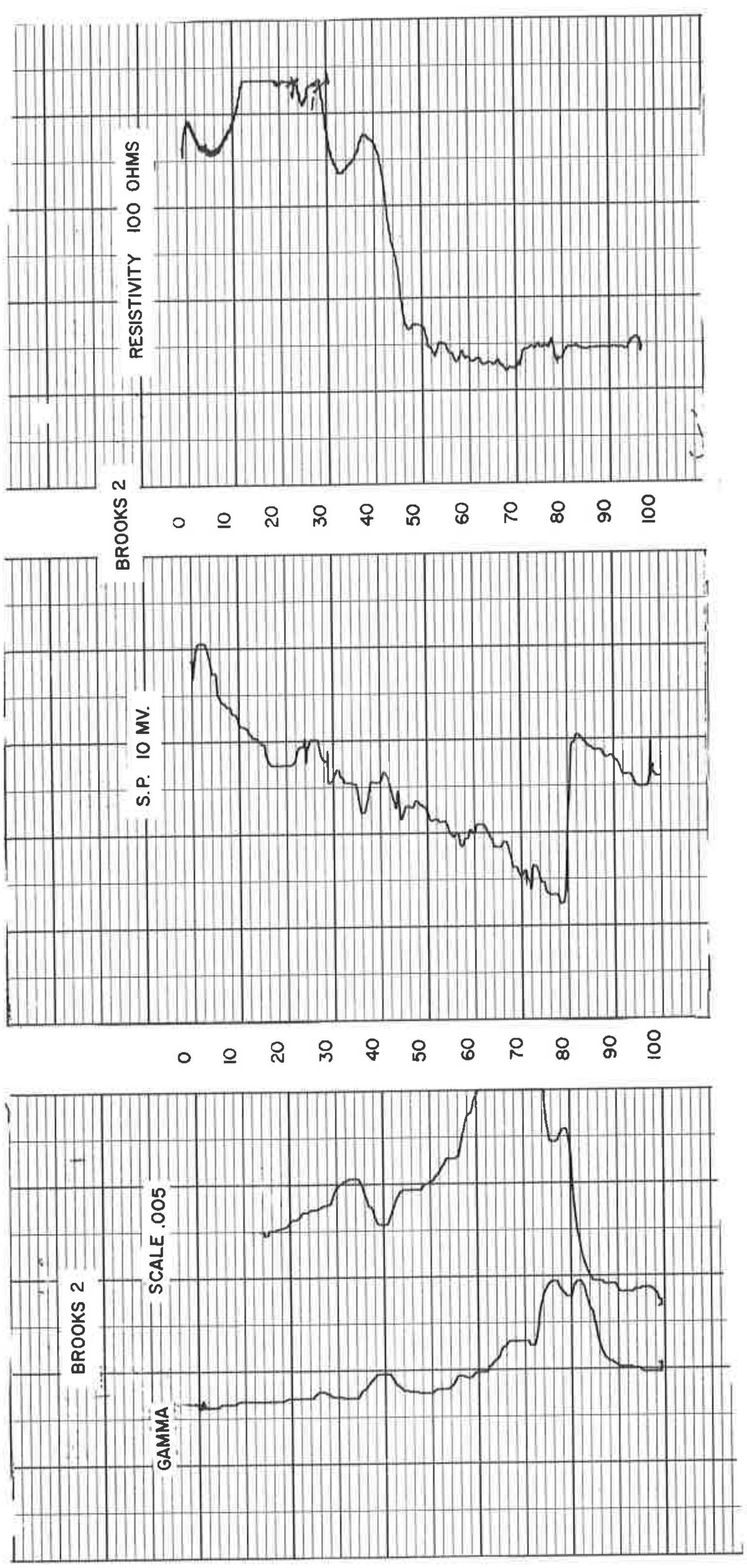

Figure AII-1. Electric and Gamma-Ray Logs - Brooks County
Hole No. Br-2

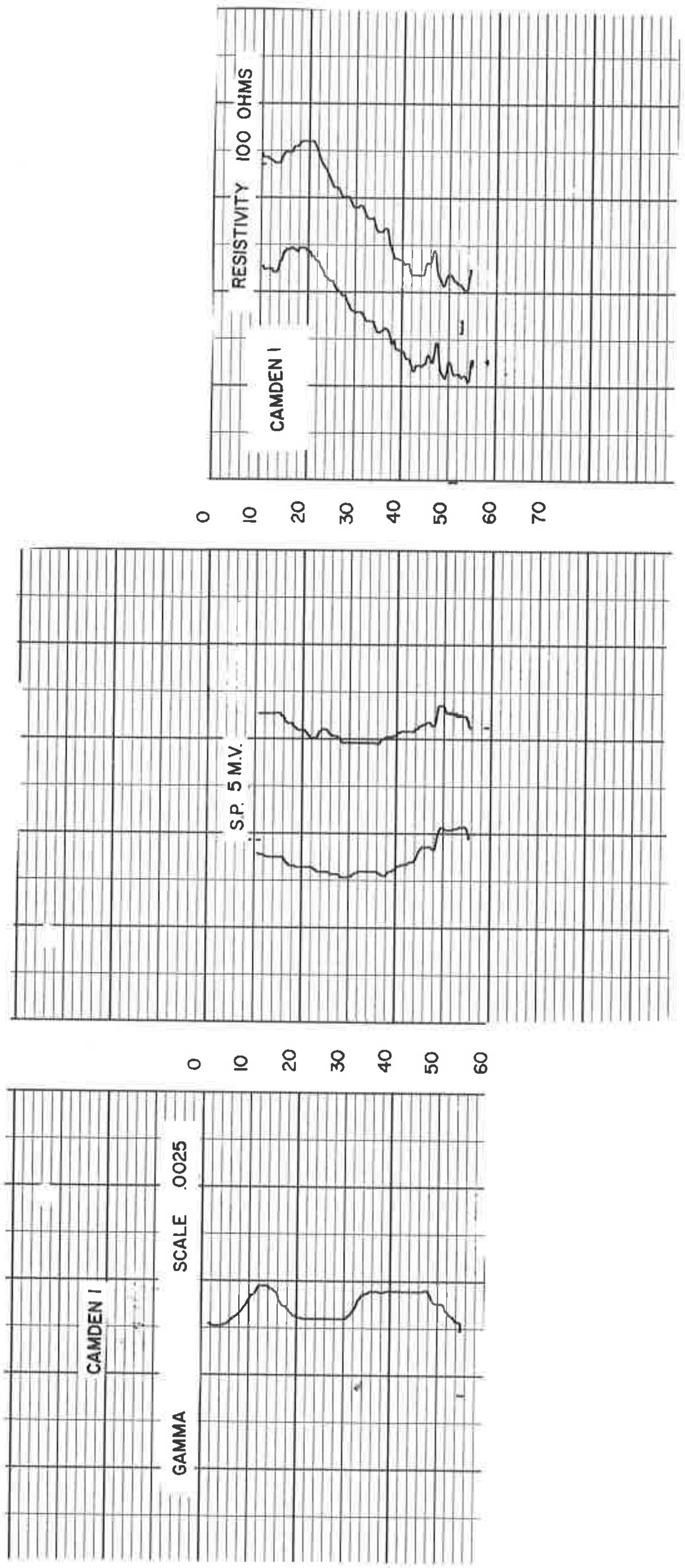

Figure AII-2. Electric and Gamma-Ray Logs - Camden County Hole No. Ca-1

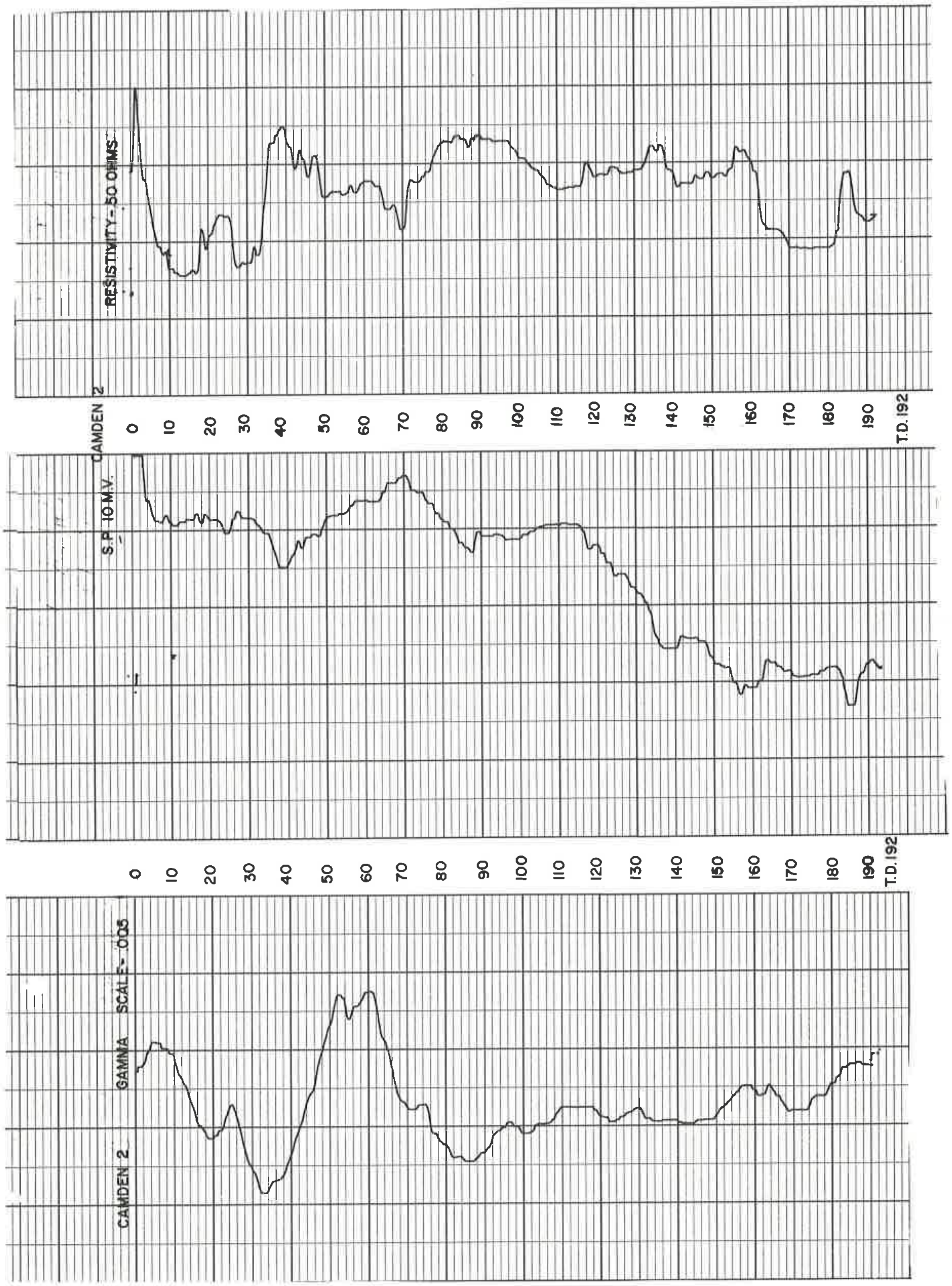

Figure AII-2. Electric and Gamma-Ray Logs - Camden County
Hole No. Ca-2

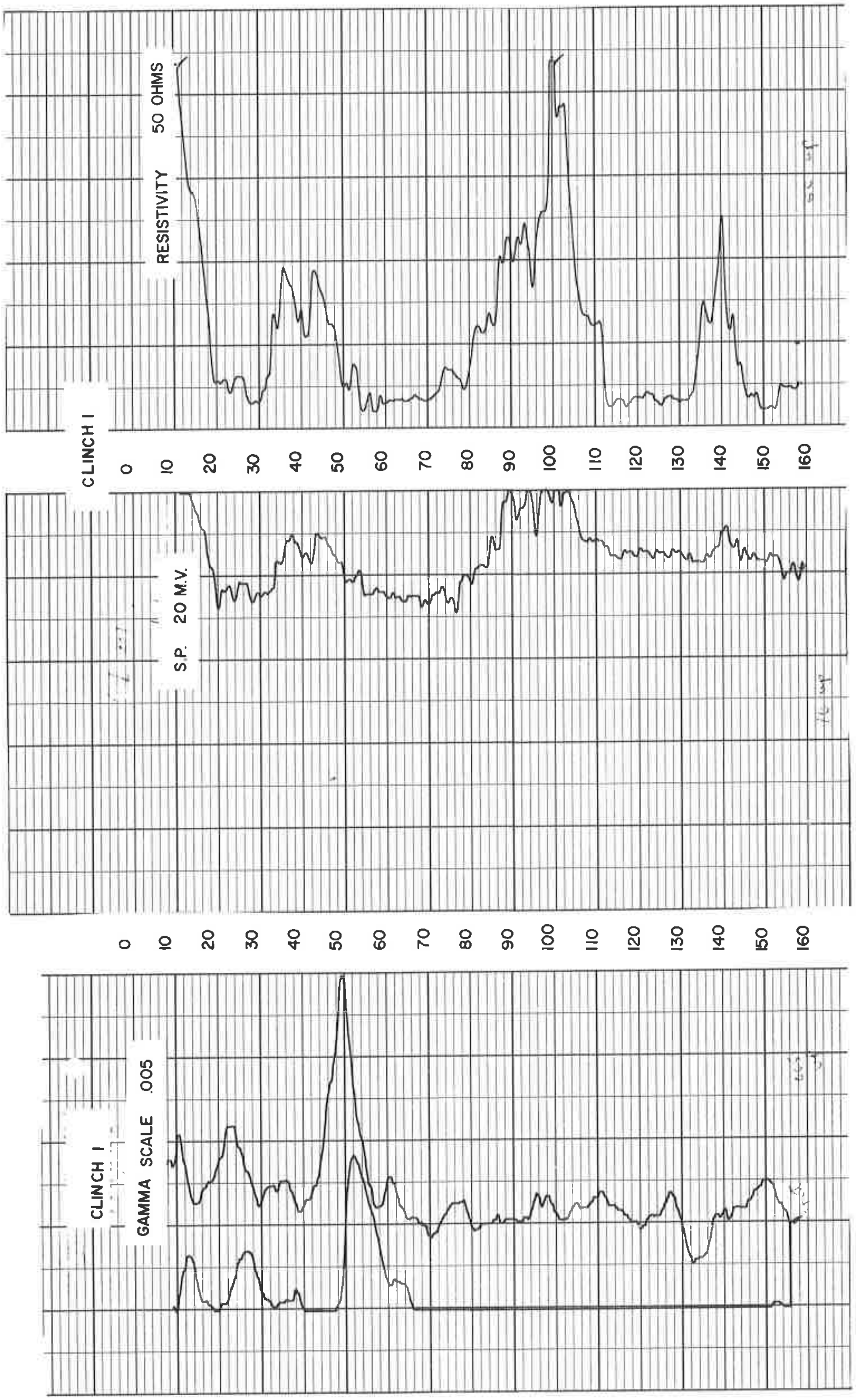

Figure AII-3. Electric and Gamma-Ray Logs - Clinch County
Hole No. Cl-1

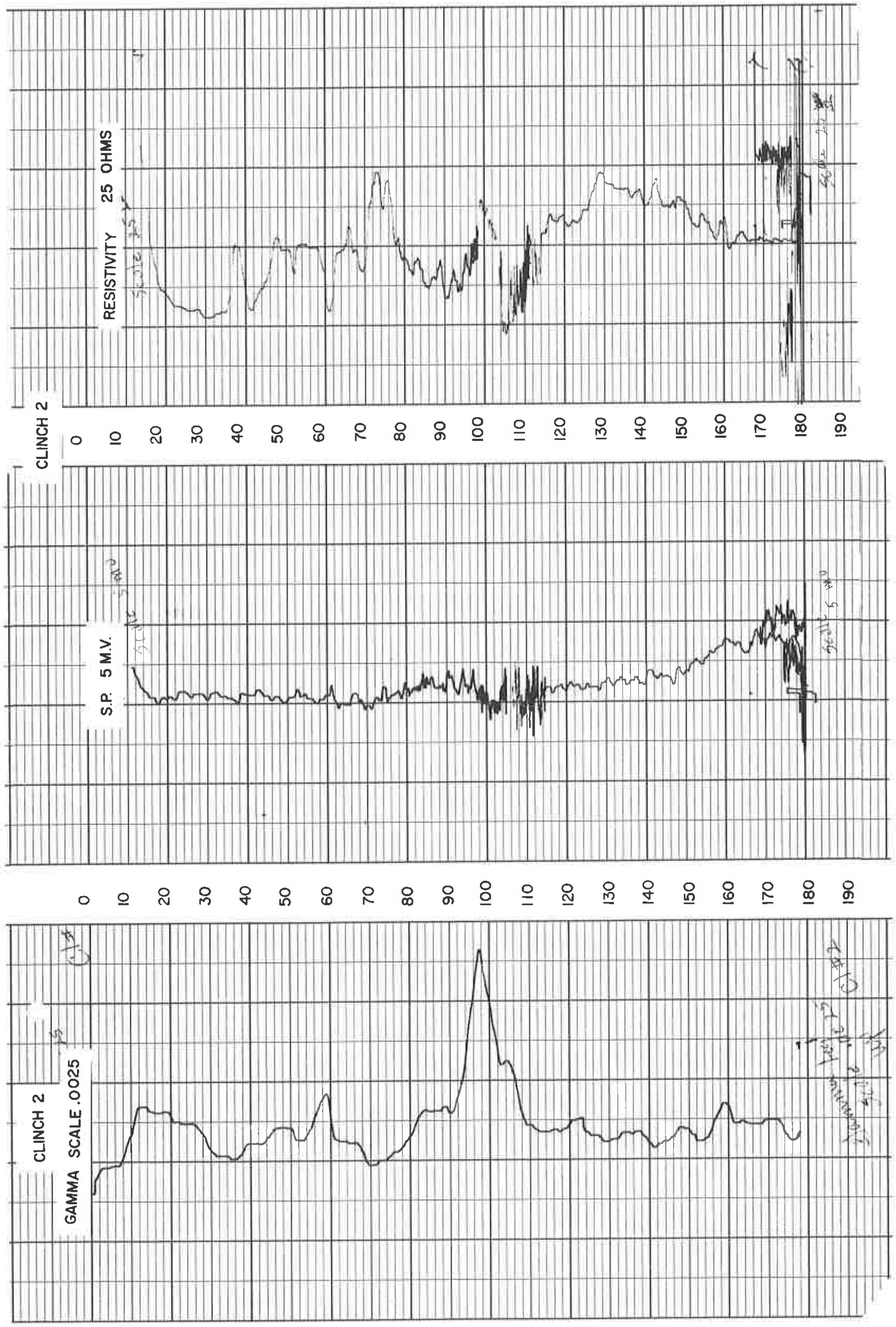

Figure AII-3. Electric and Gamma-Ray Logs - Clinch County Hole No. Cl-2

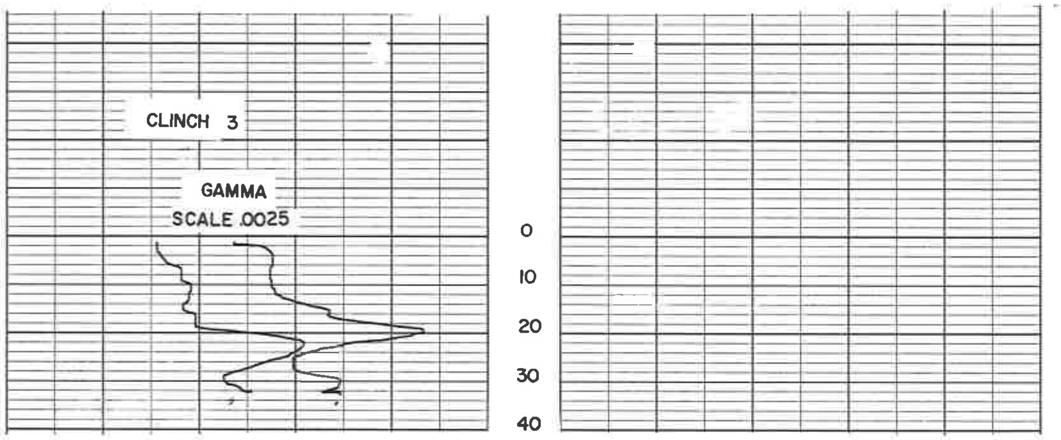

Figure AII-3. Electric and Gamma-Ray Logs - Clinch County Hole No. Cl-3

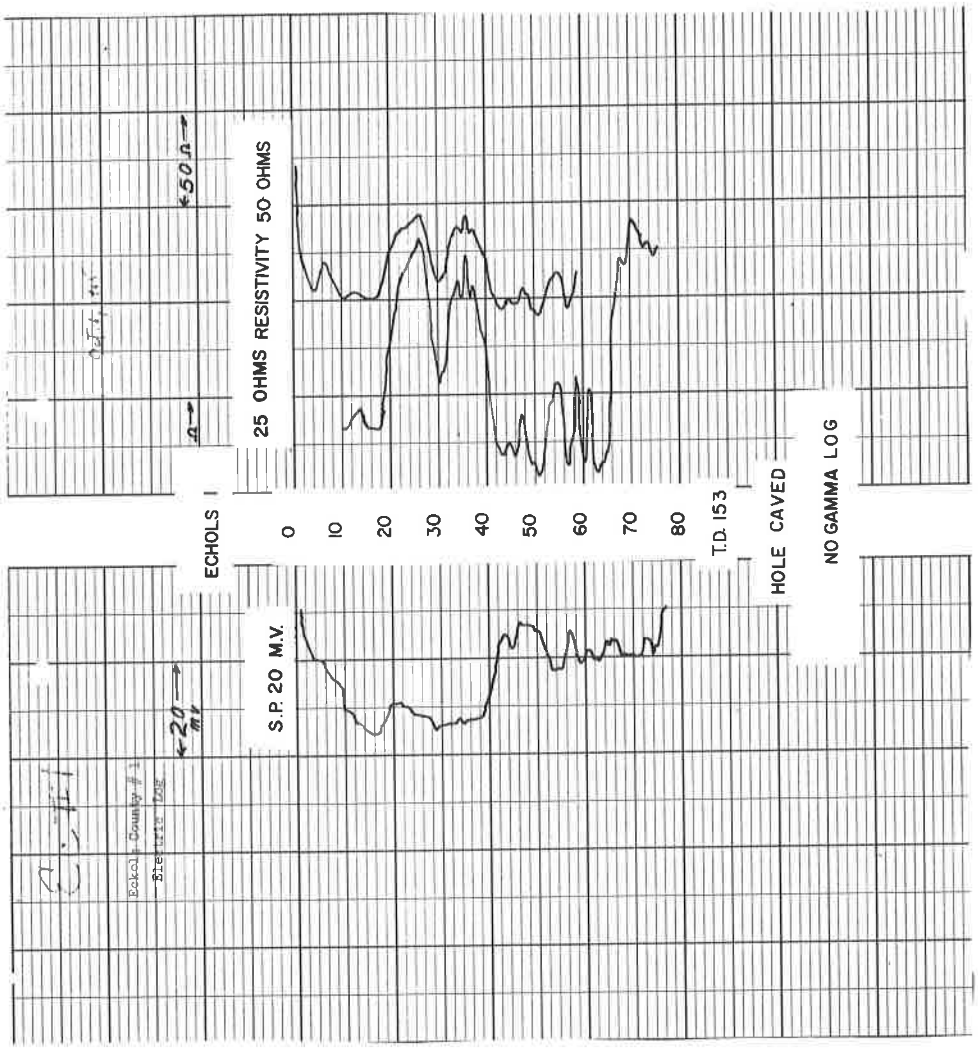

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-1

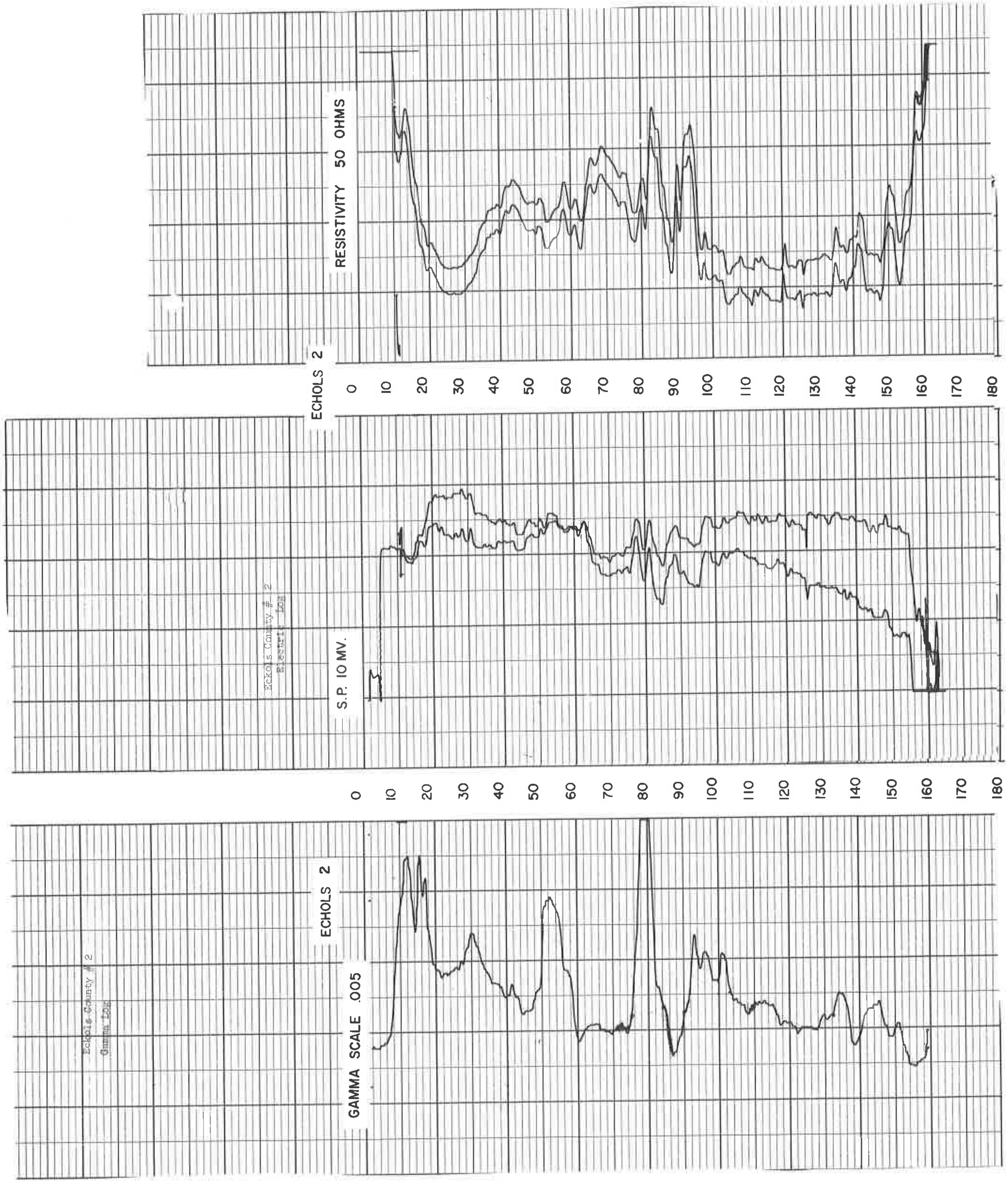

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-2

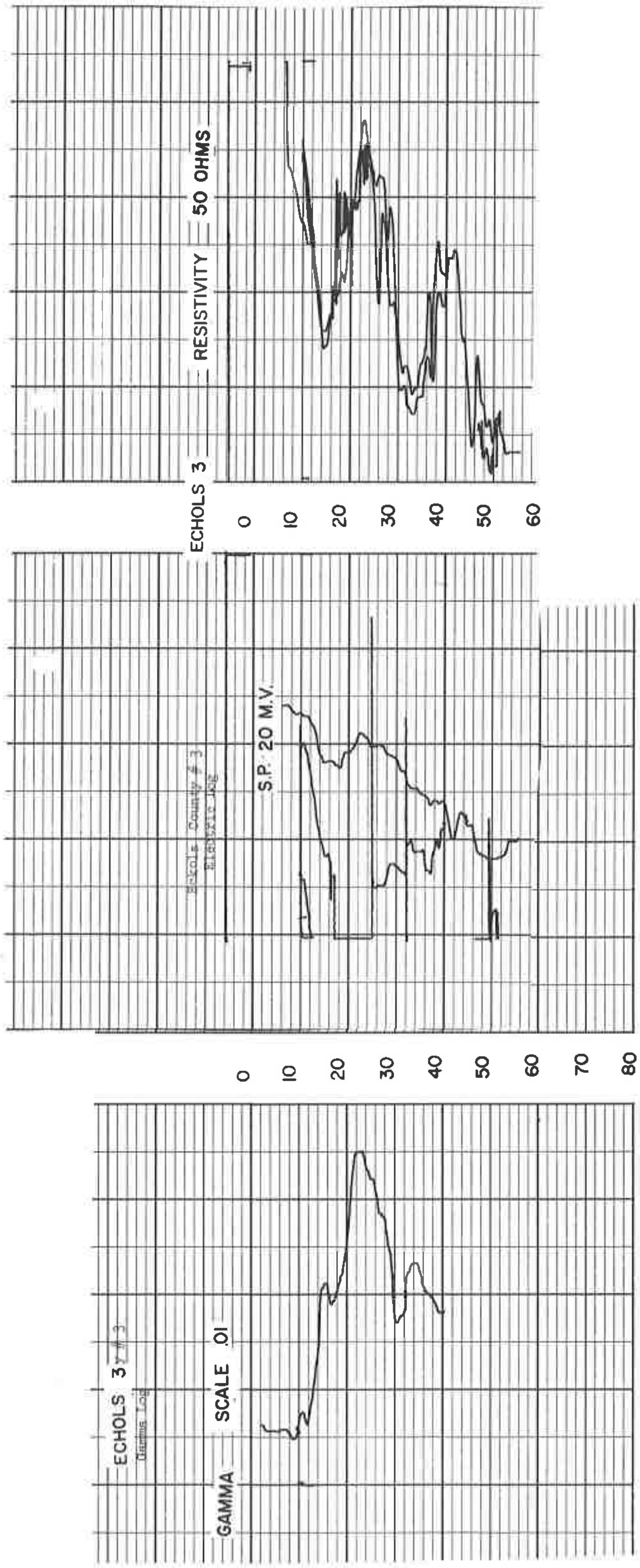

Figure AII-4. Electric and Gamma-Ray Logs - Echols County
Hole No. Ec-3

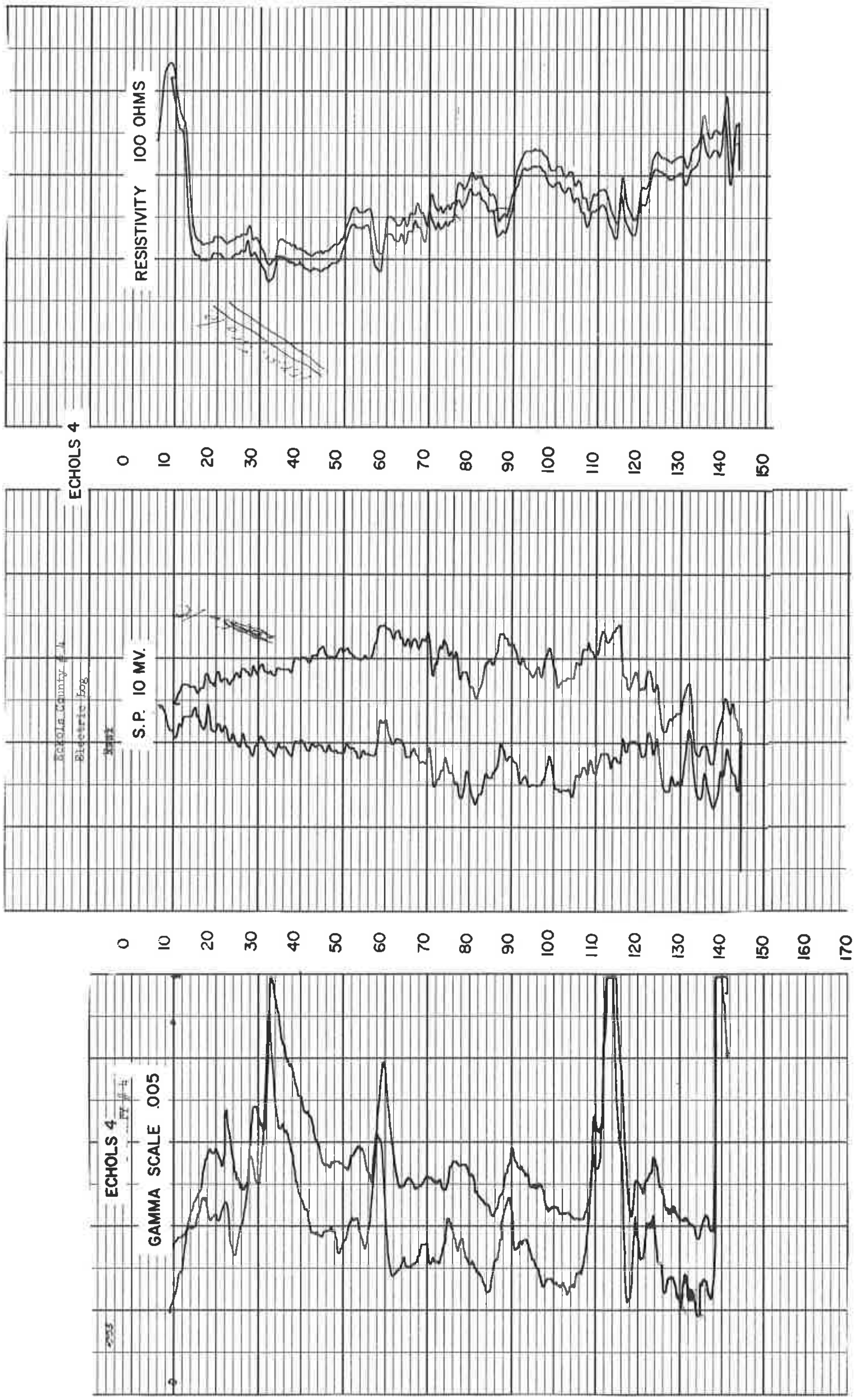

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-4

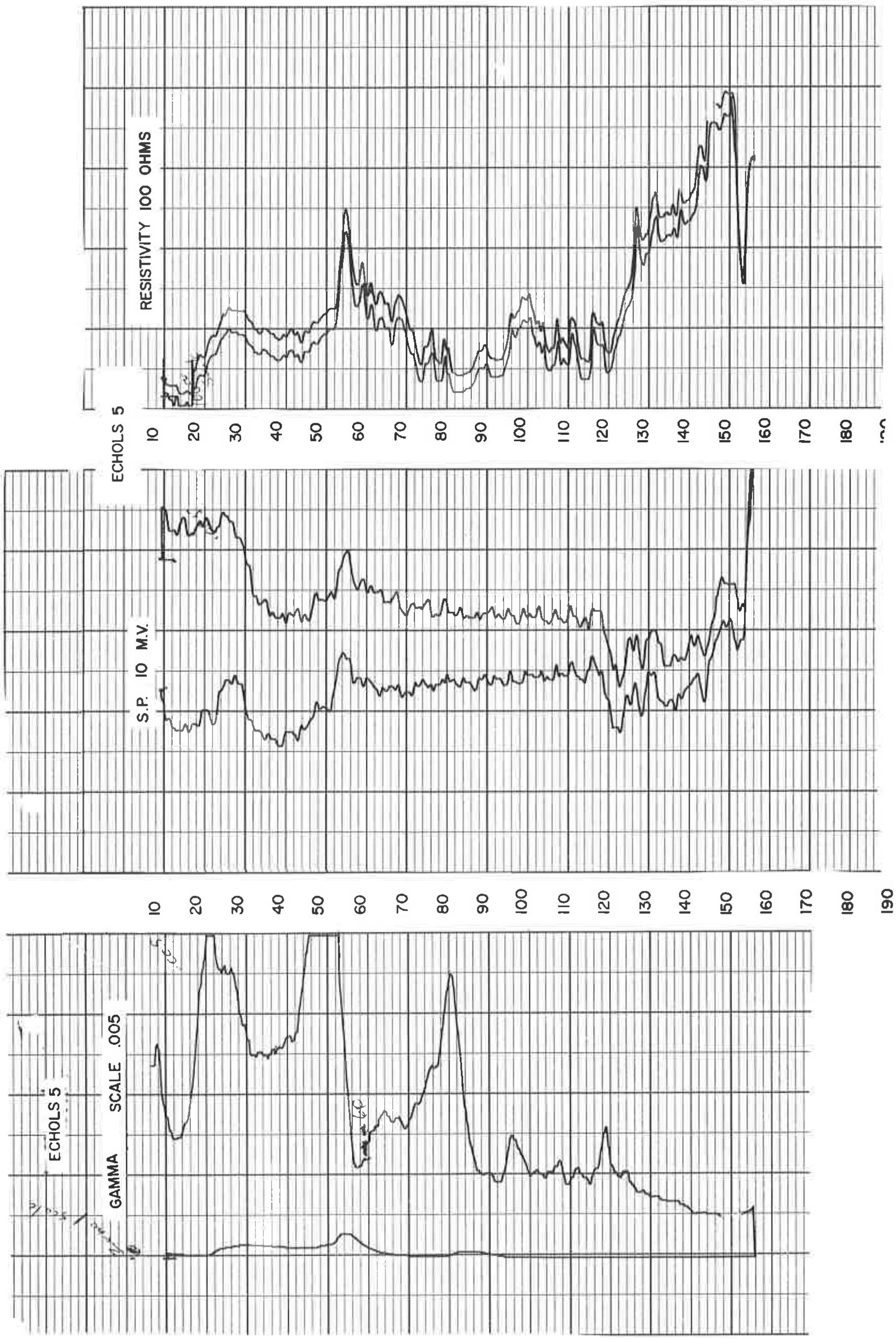

Figure AII-4. Electric and Gamma-Ray Logs - Echols County
Hole No. Ec-5

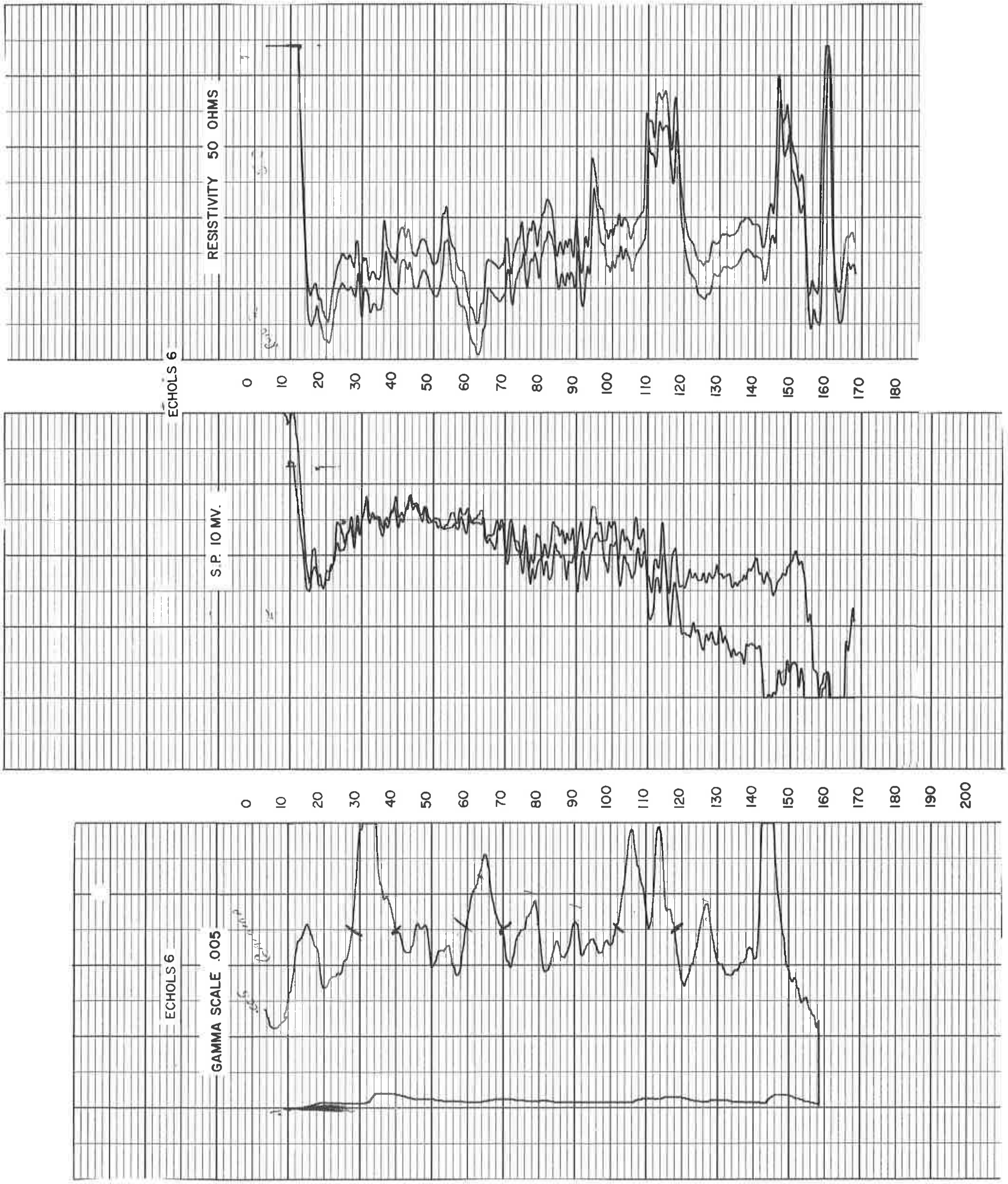

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-6

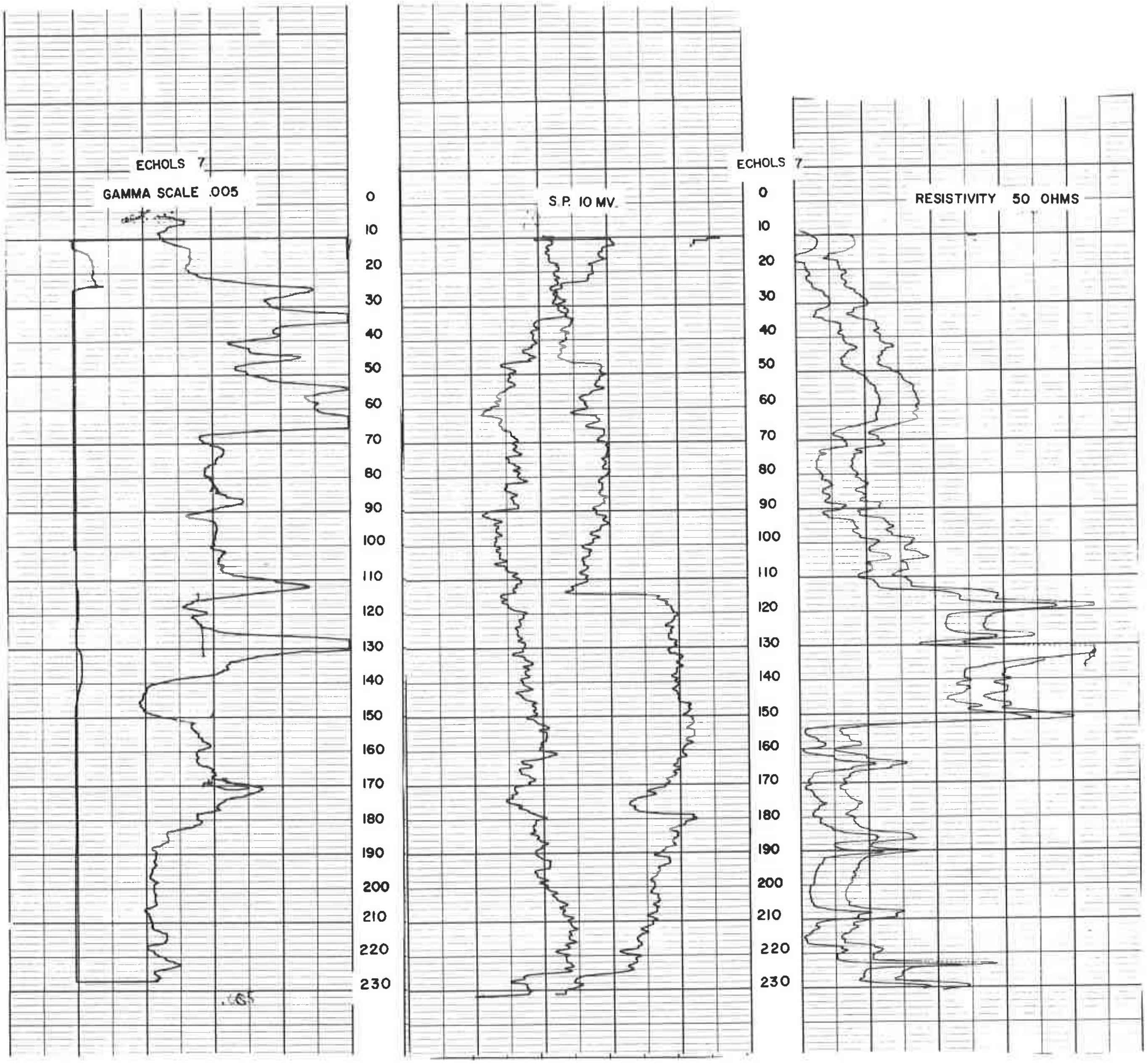

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-7

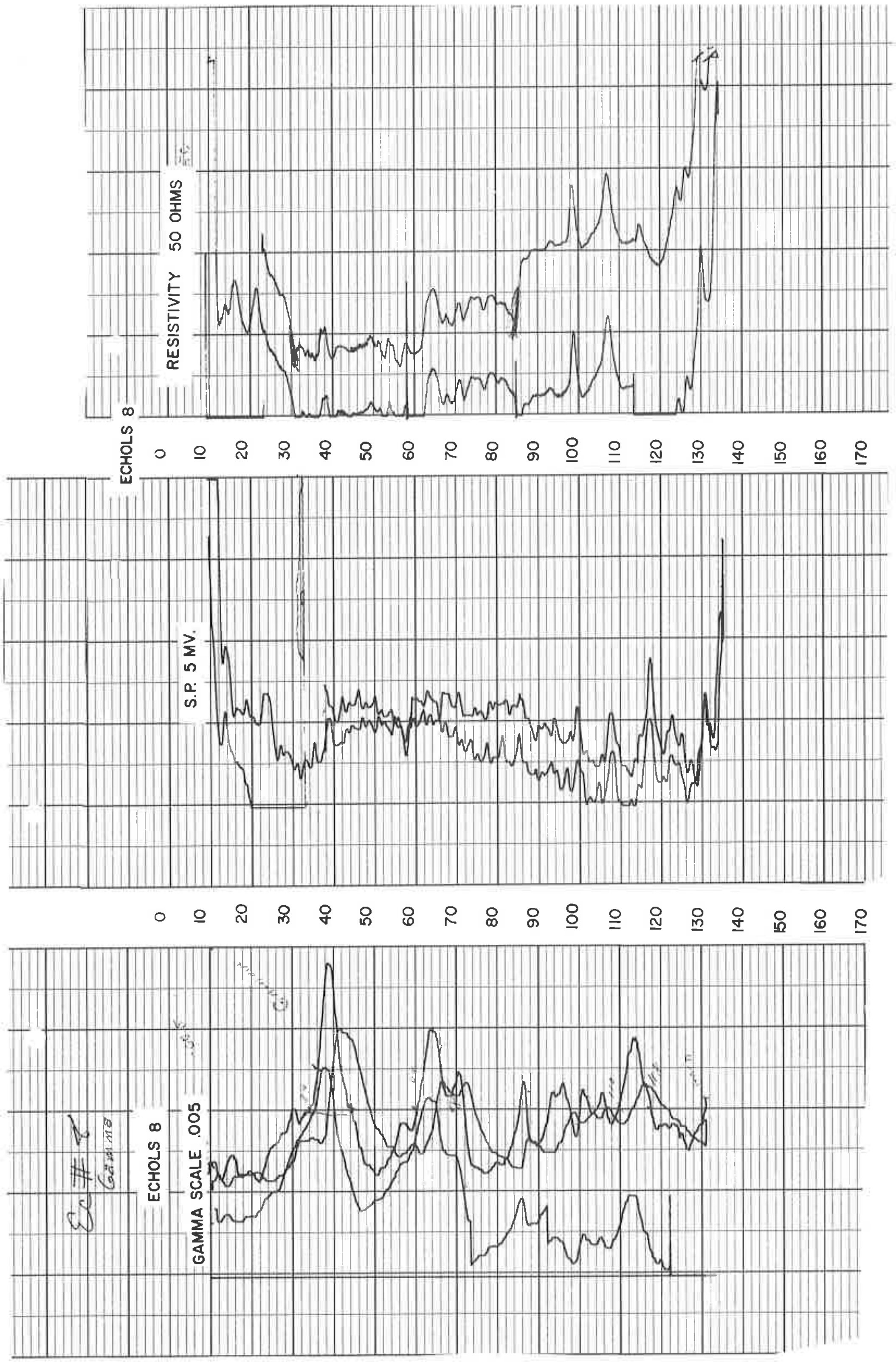

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-8

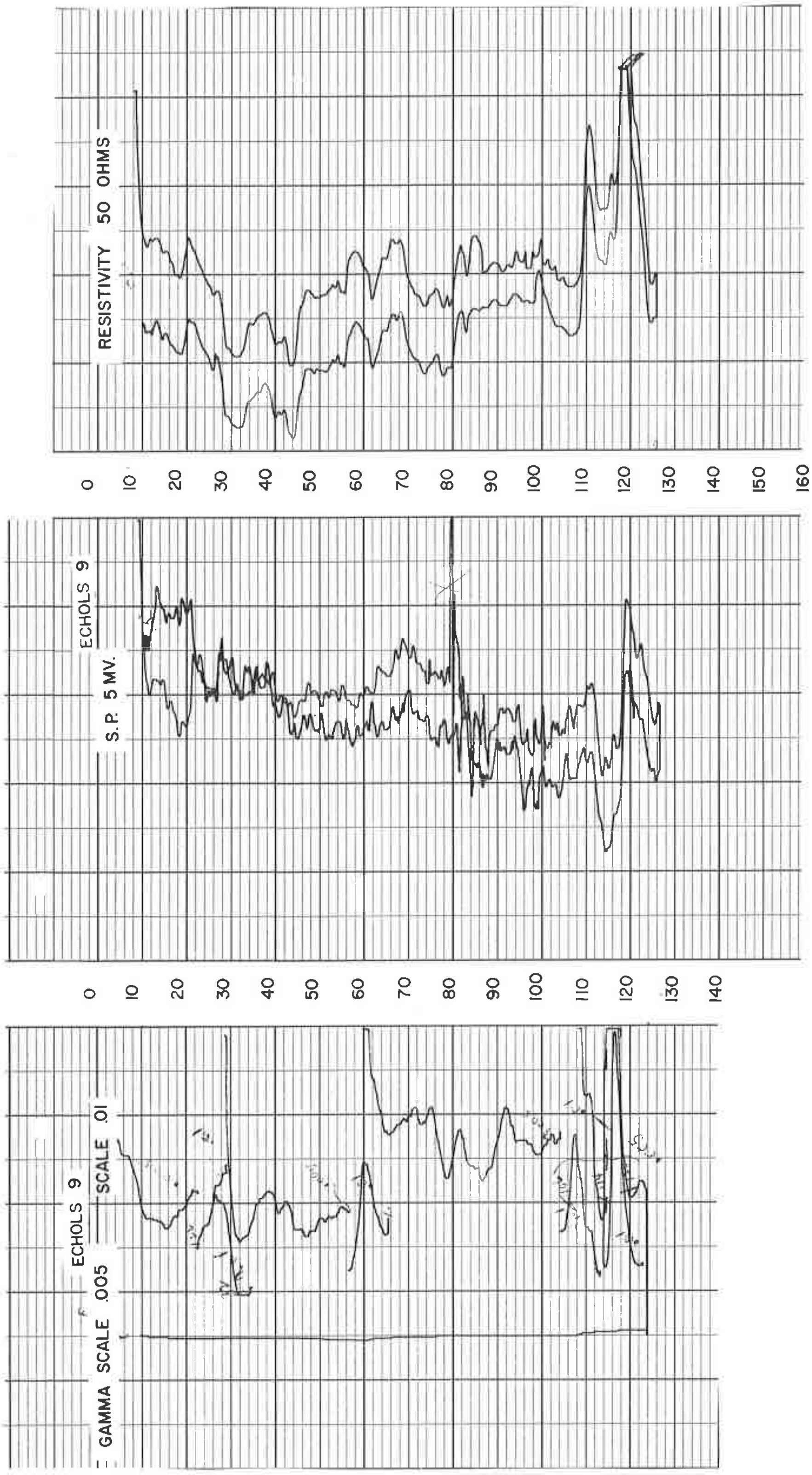

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-9

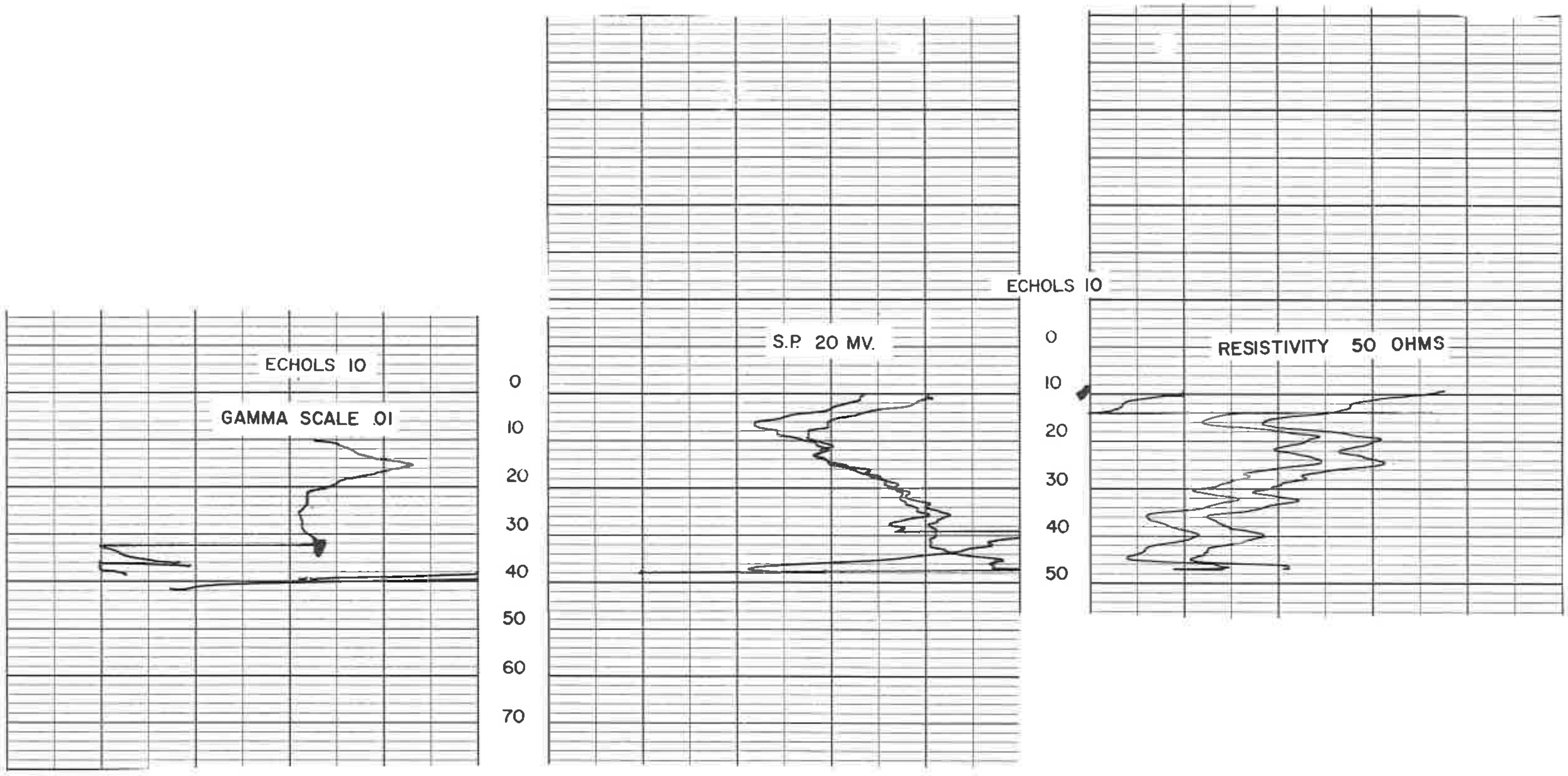

Figure AII-4. Electric and Gamma-Ray Logs - Echols County
Hole No. Ec-10

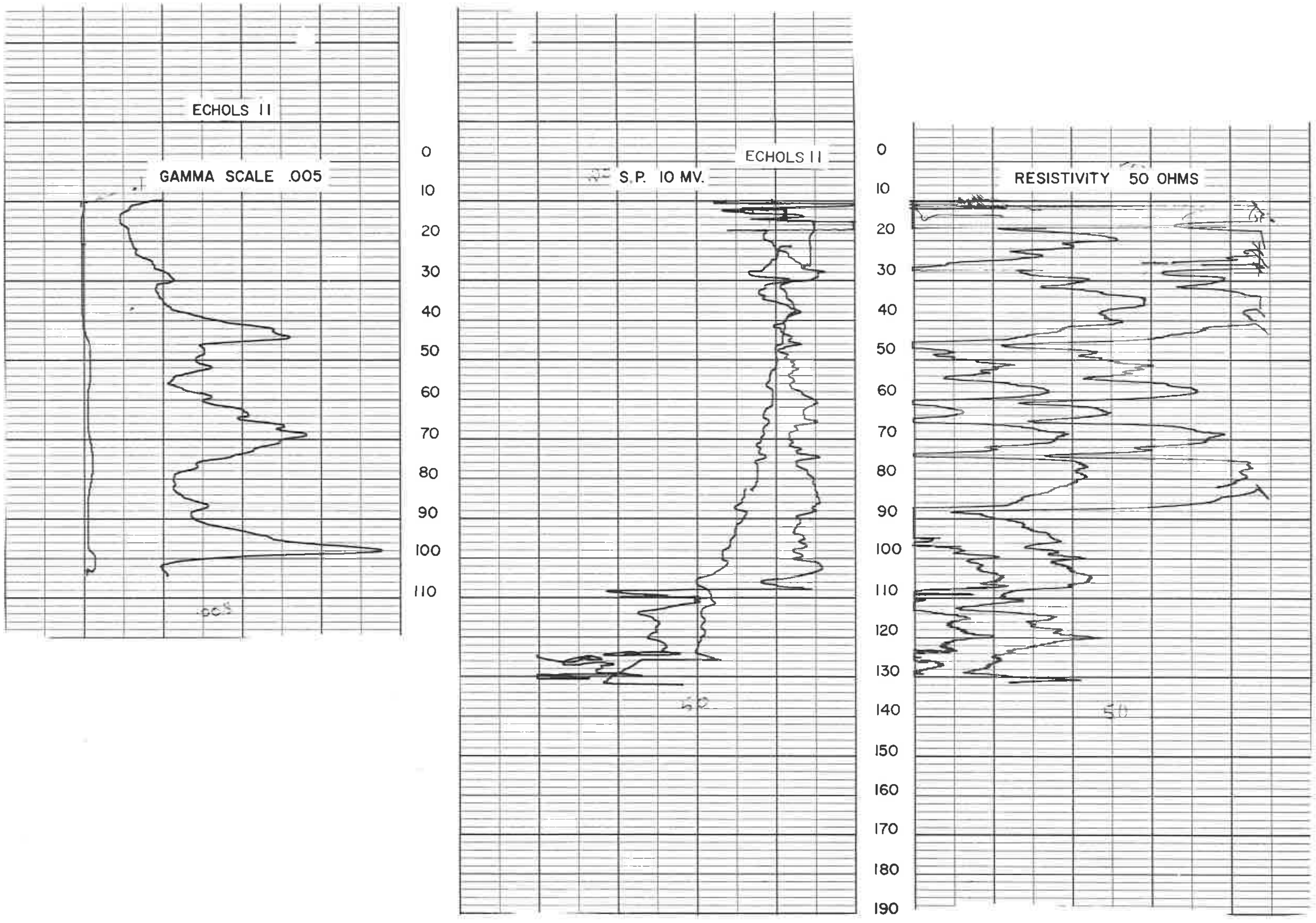

Figure AII-4. Electric and Gamma-Ray Logs - Echols County
Hole No. Ec-11

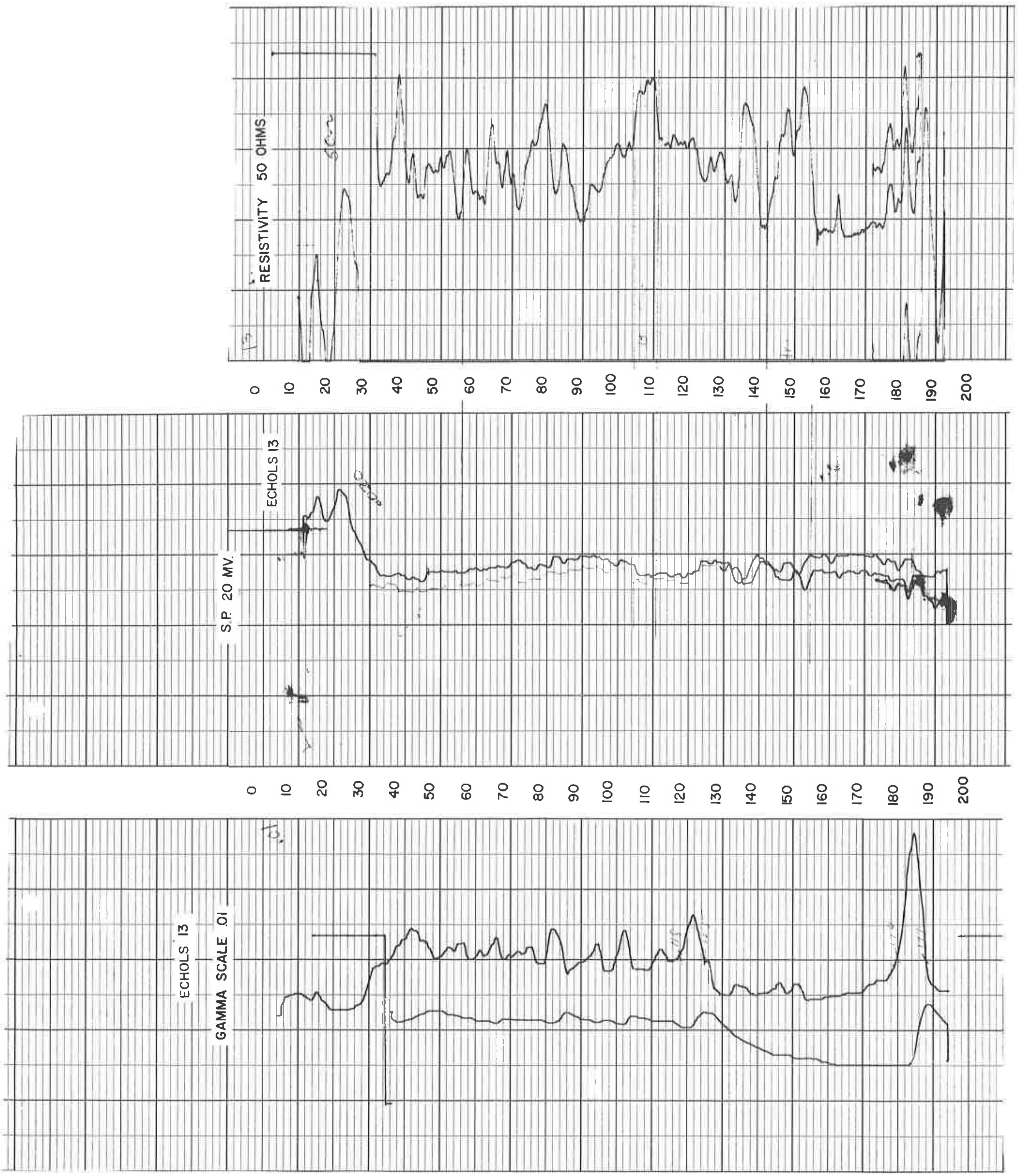

Figure AII-4. Electric and Gamma-Ray Logs - Echols County Hole No. Ec-13

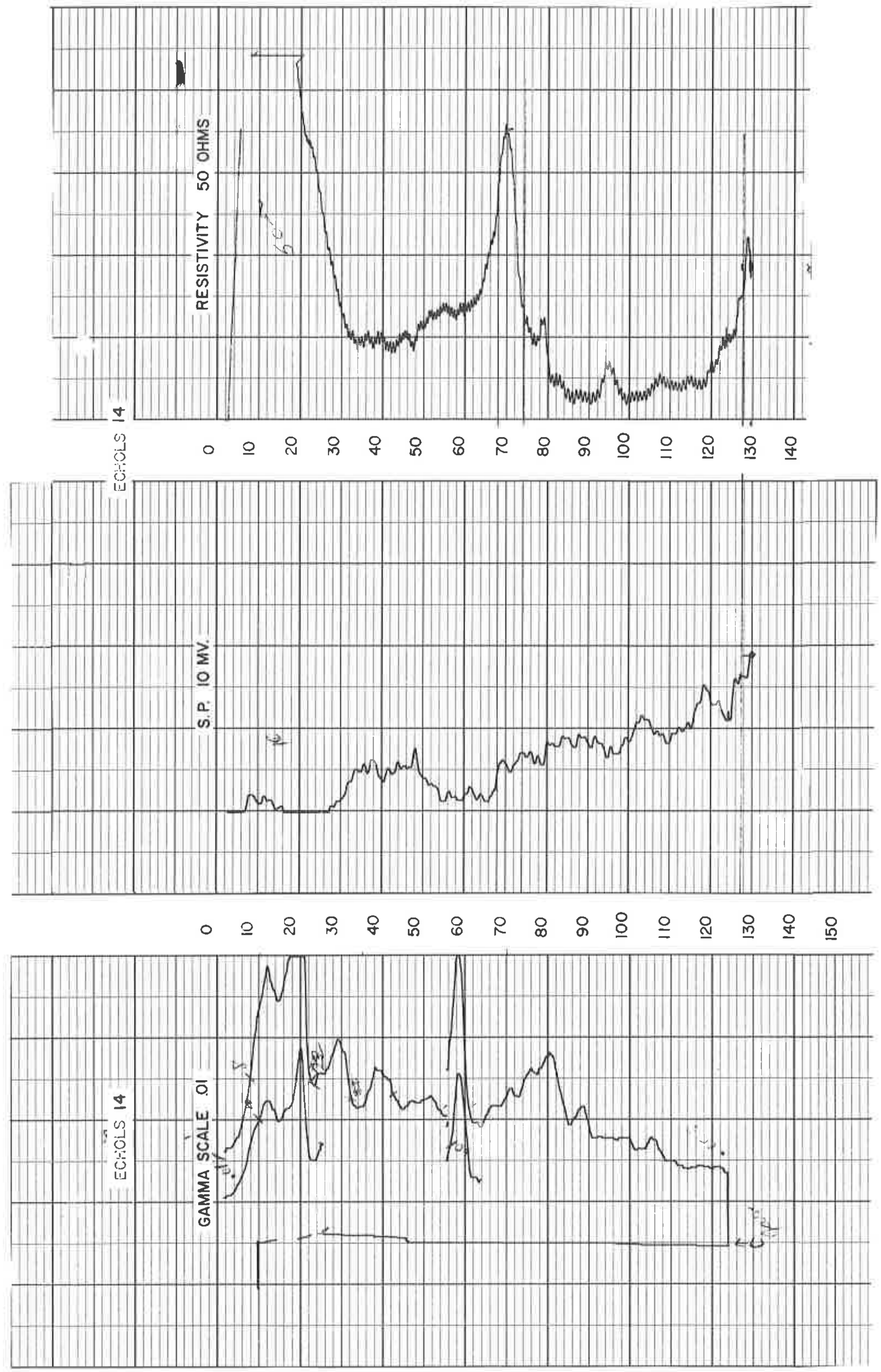

Figure AII-4. Electric and Gamma-Ray Logs - Echols County
Hole No. Ec-14

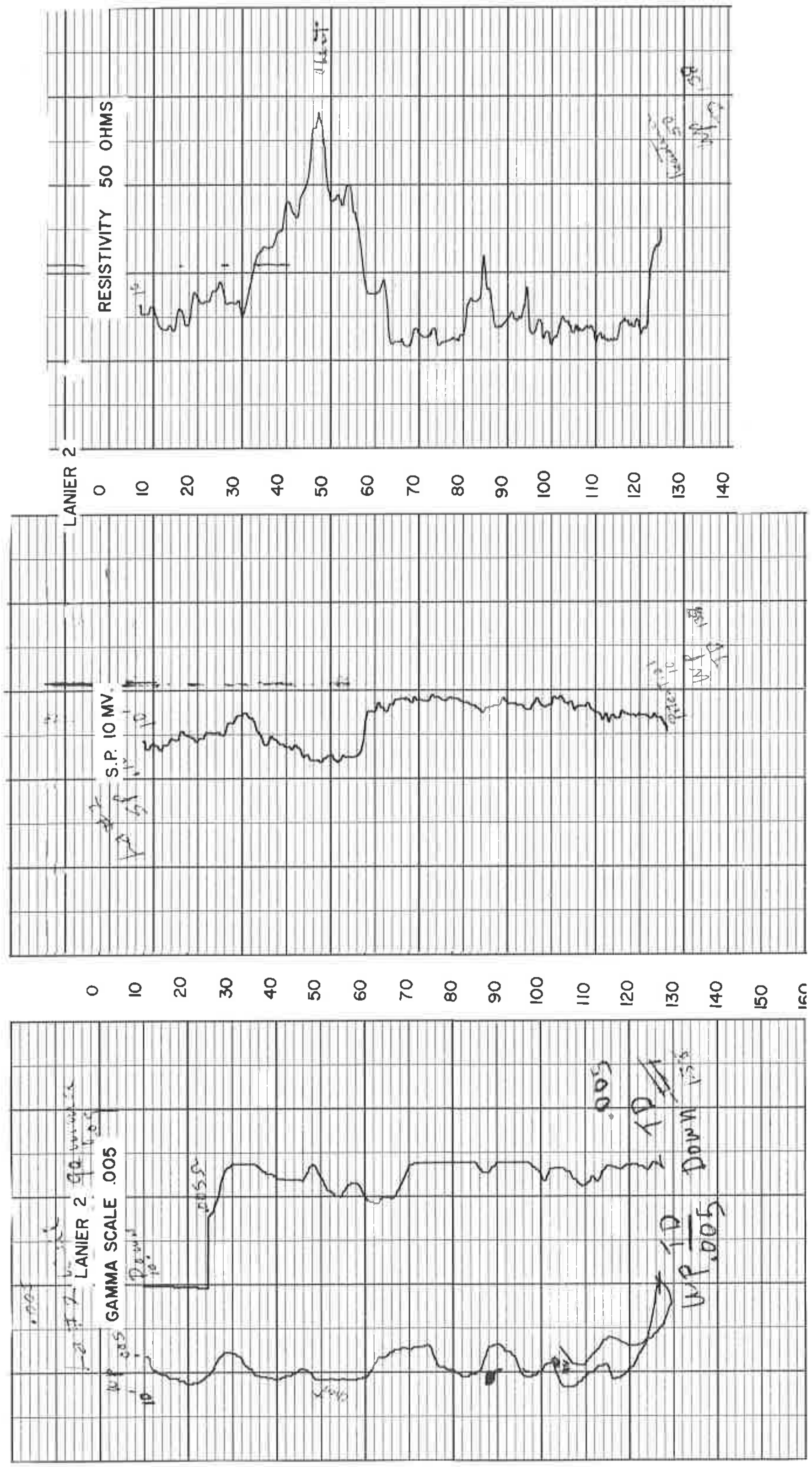

Figure AII-5. Electric and Gamma-Ray Logs - Lanier County Hole No. La-2

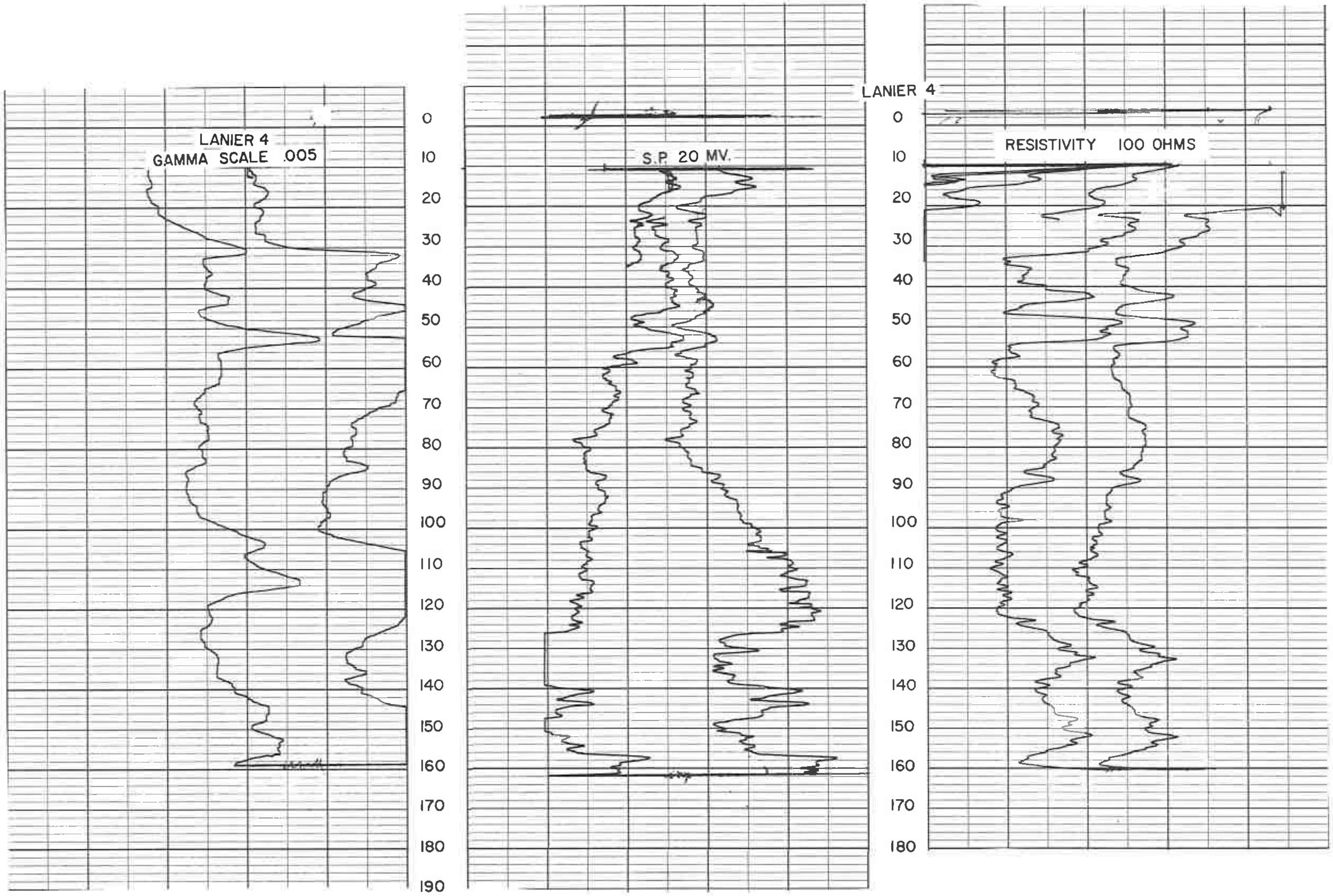

Figure AII-5. Electric and Gamma-Ray Logs - Lanier County
Hole No. La-4

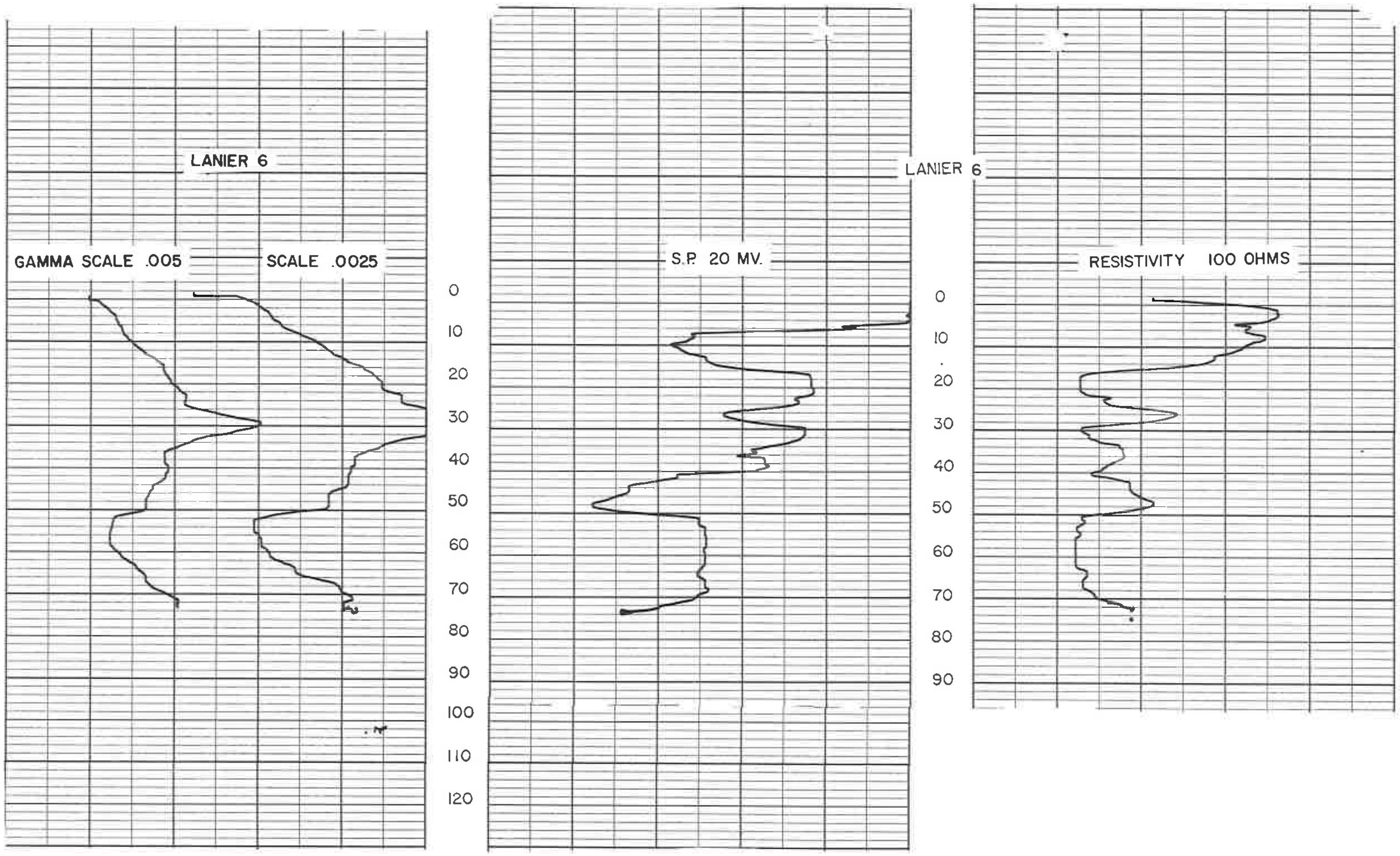

Figure AII-5. Electric and Gamma-Ray Logs - Lanier County
Hole No. La-6

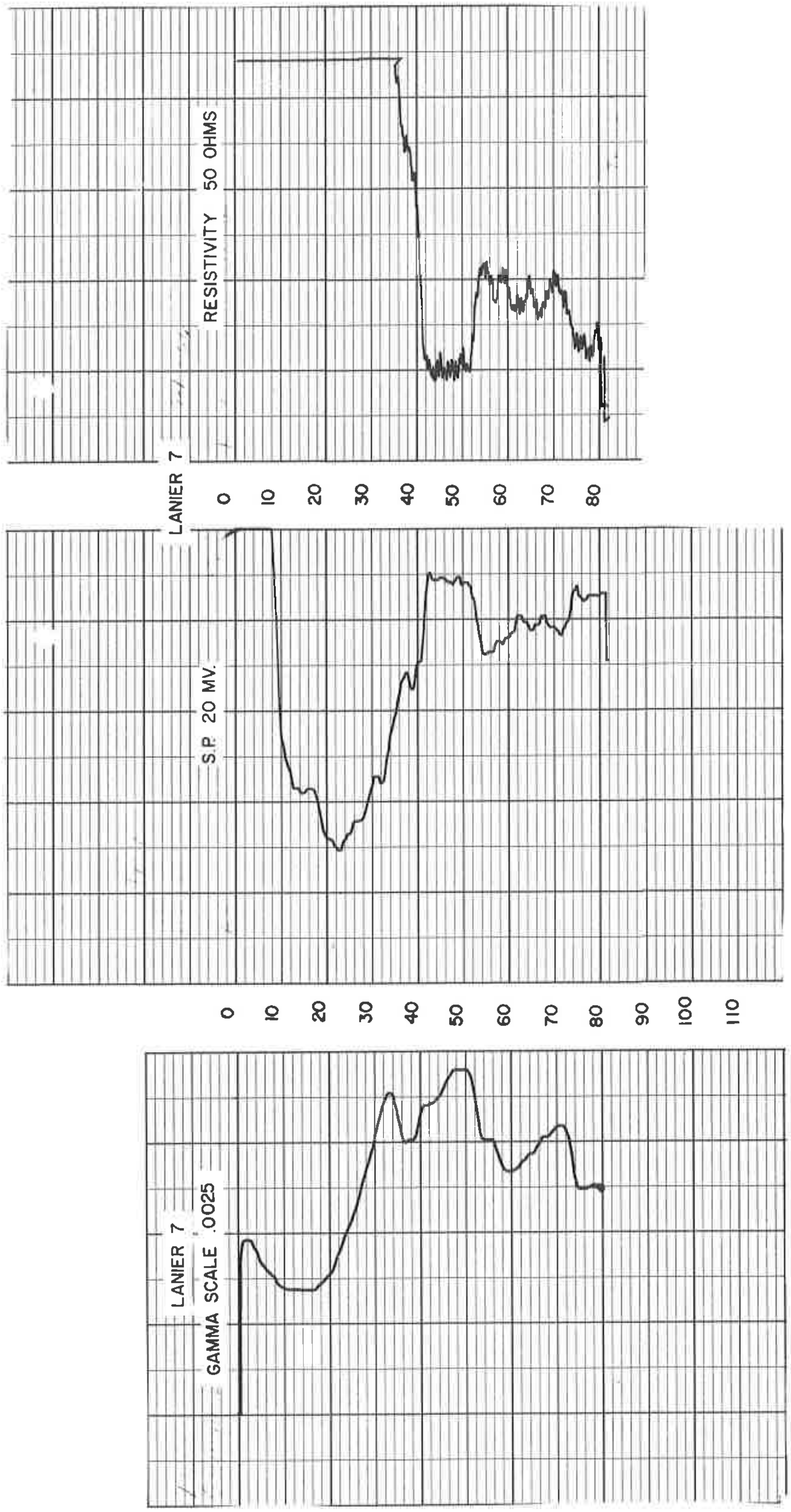

Figure AII-5. Electric and Gamma-Ray Logs - Lanier County
Hole No. La-7

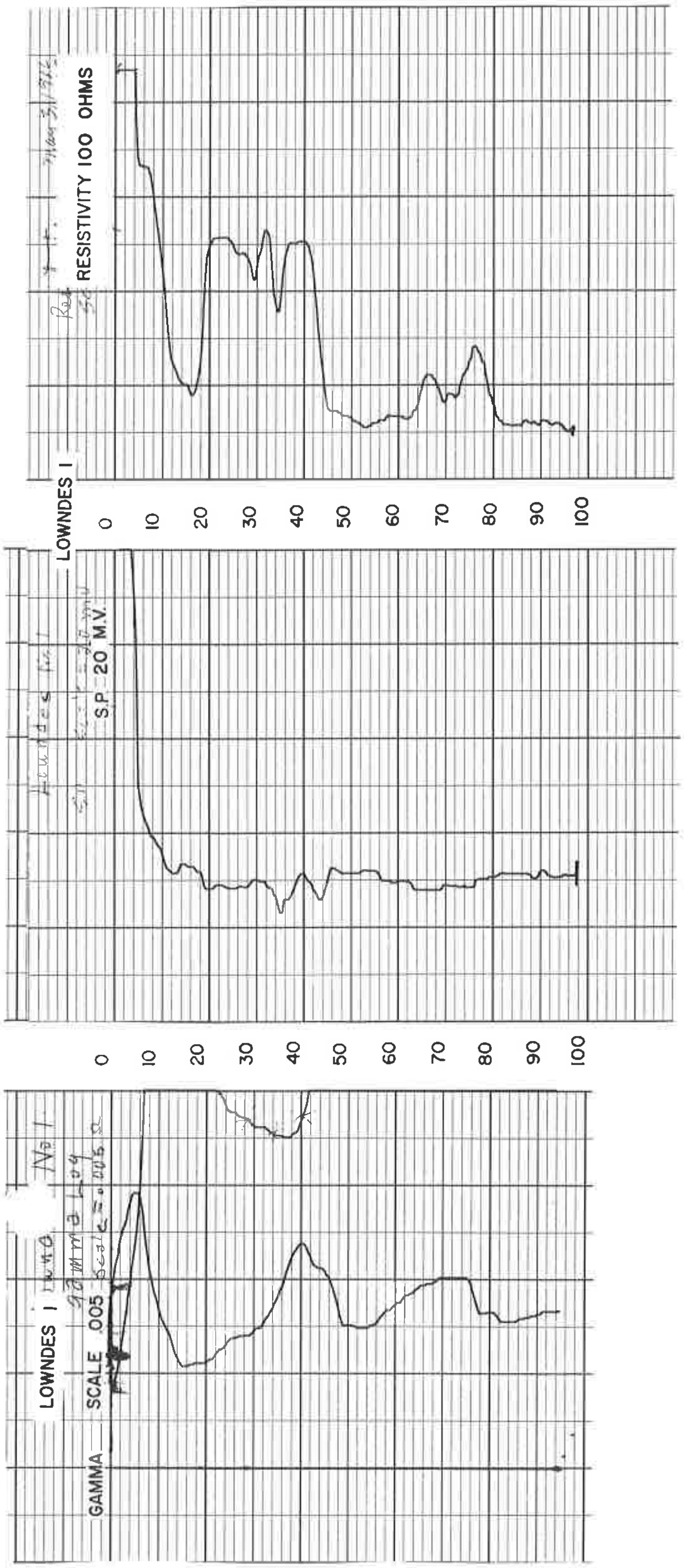

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County Hole No. Lo-1

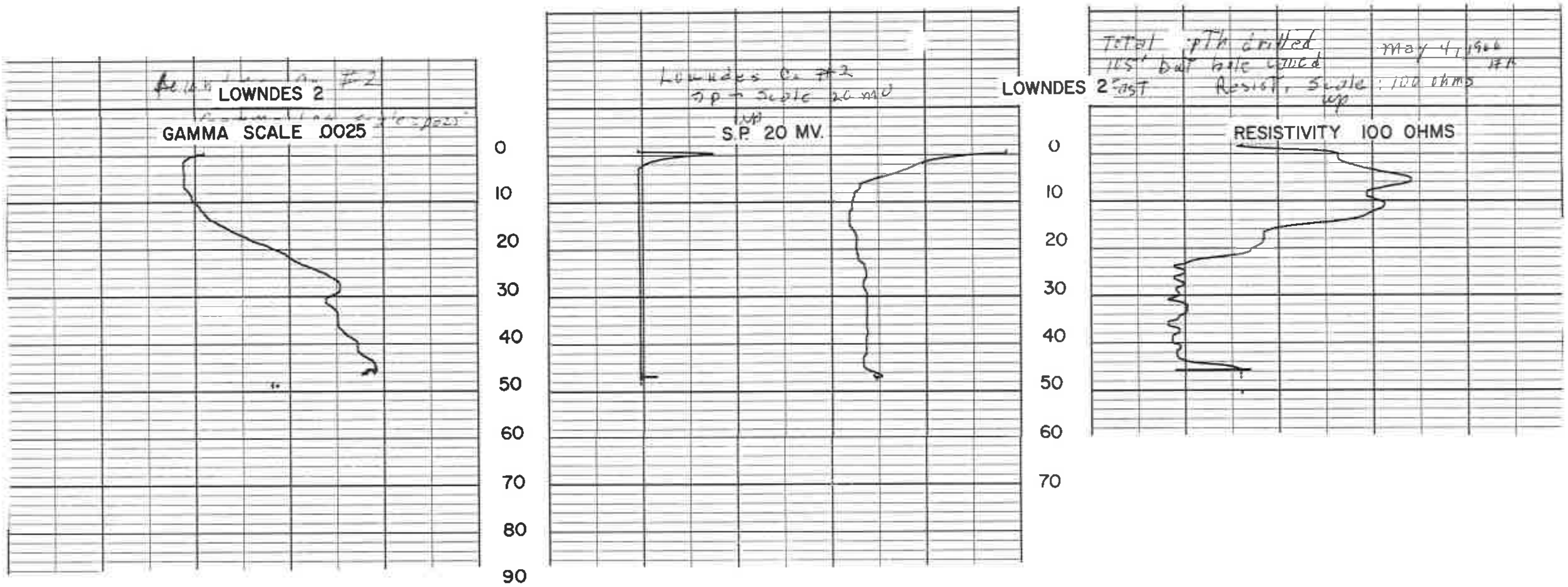

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County
Hole No. Lo-2

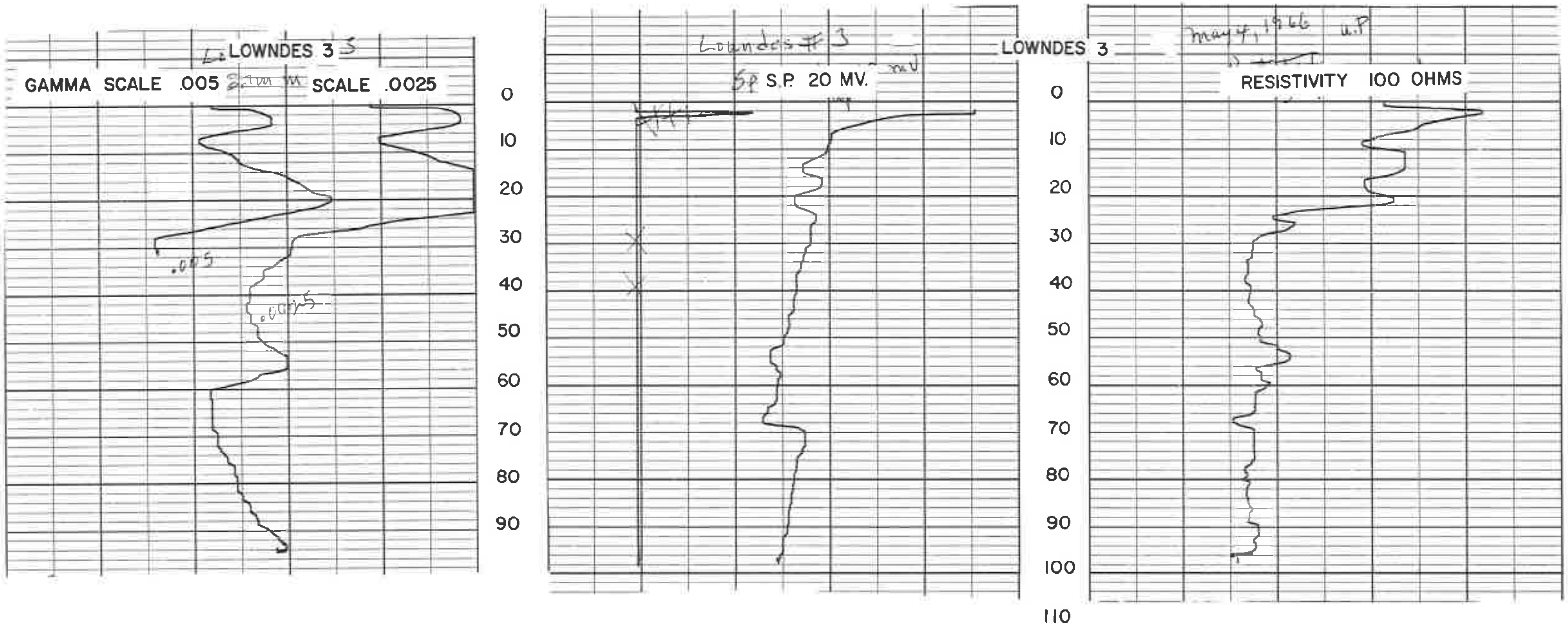

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County
Hole No. Lo-3

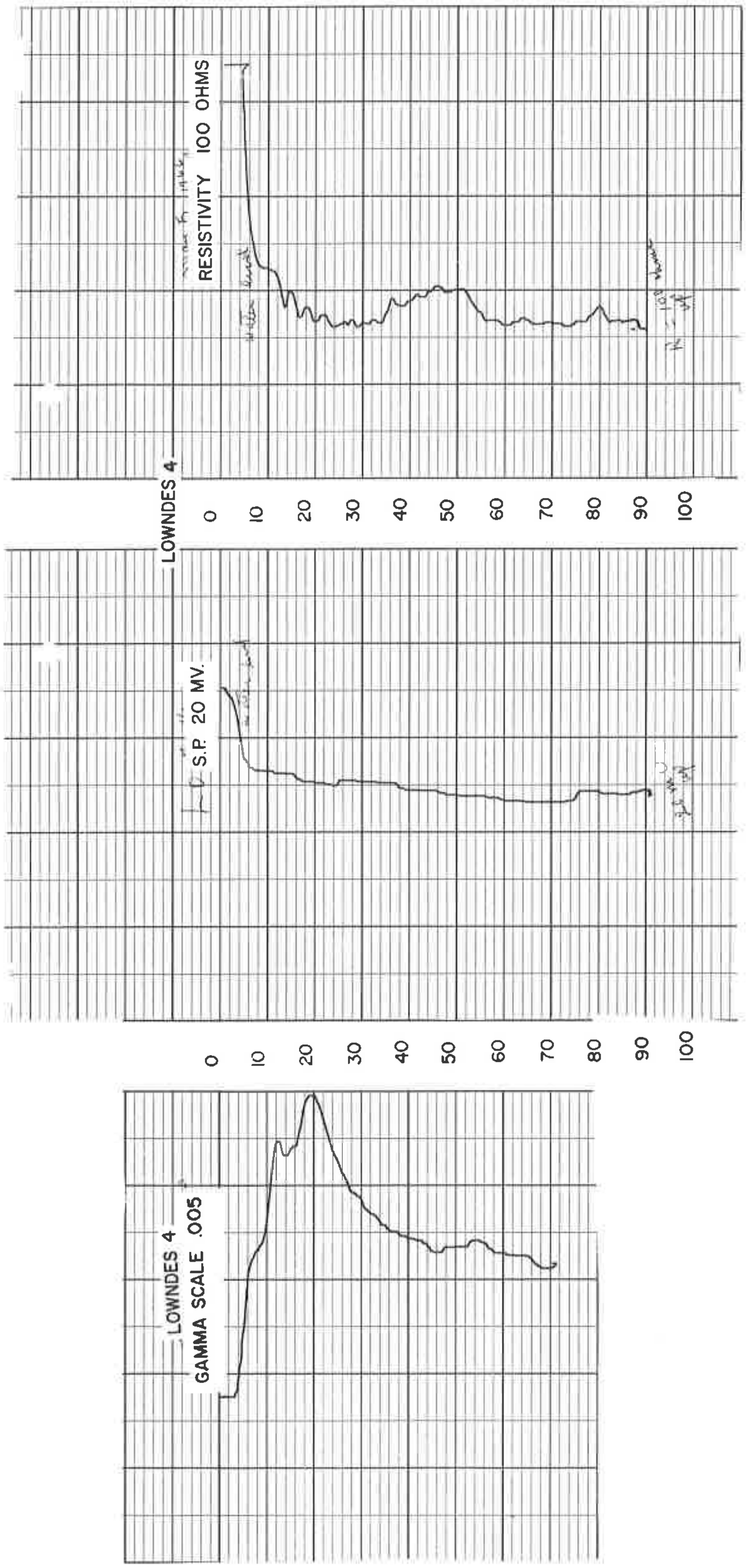

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County
Hole No. Lo-4

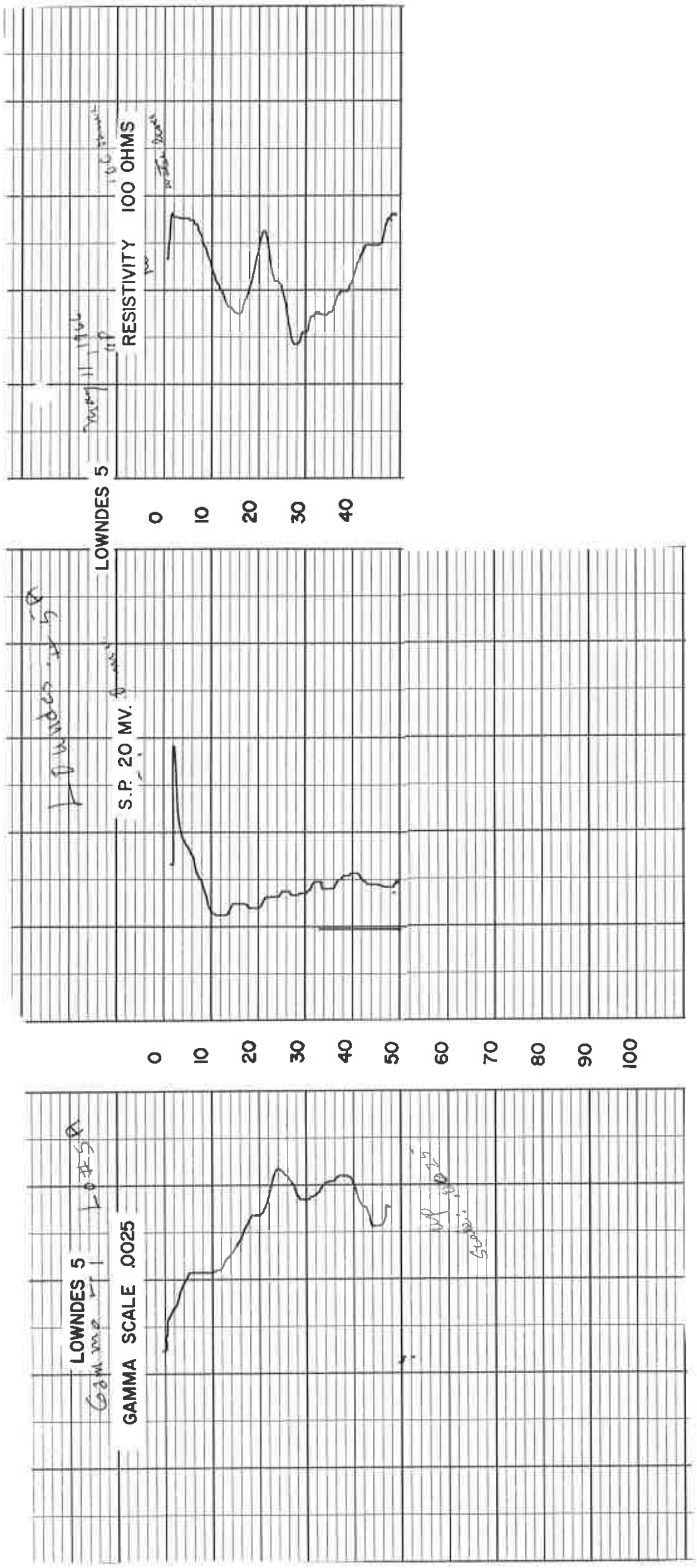

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County Hole No. Lo-5

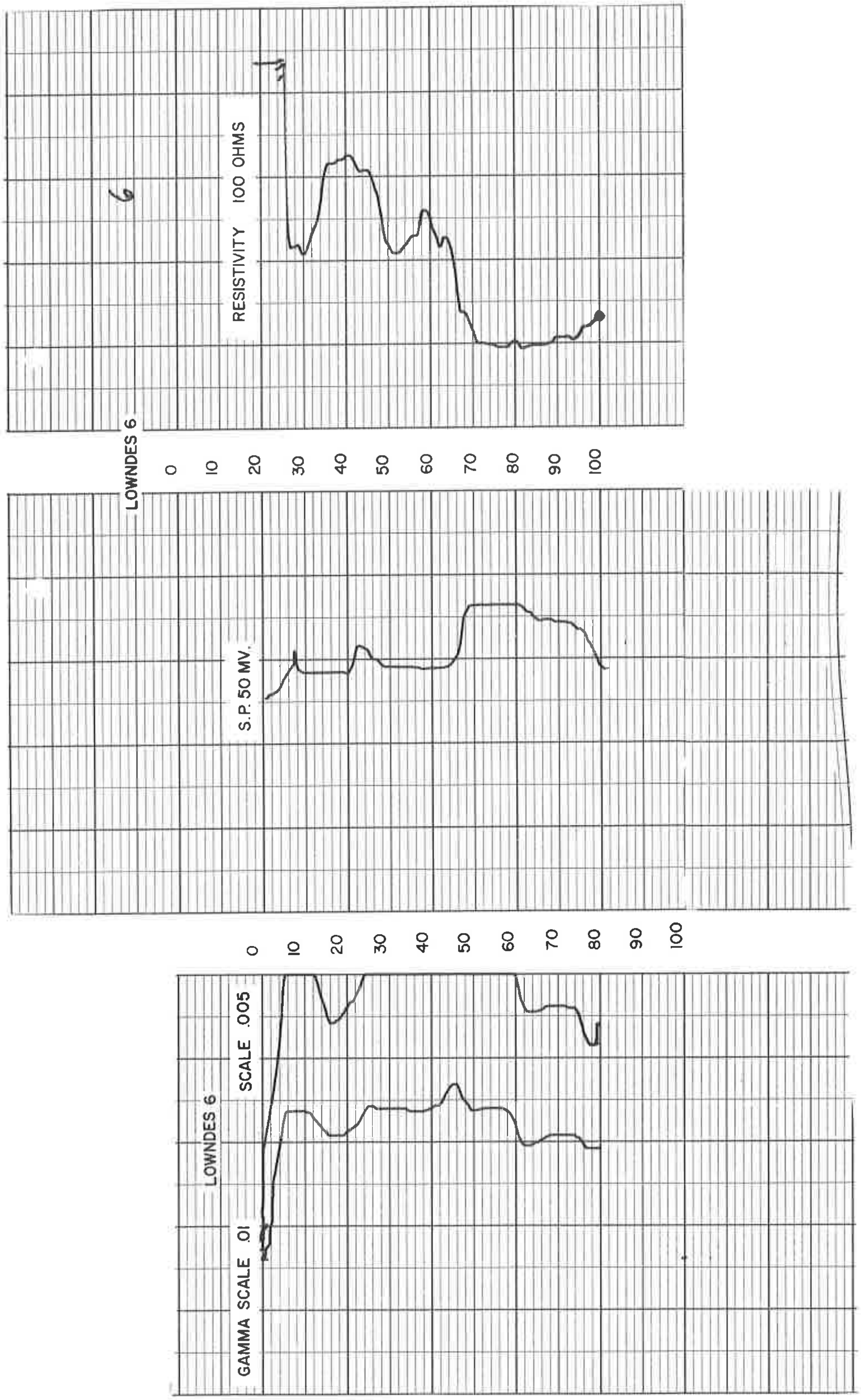

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County
Hole No. Lo-6

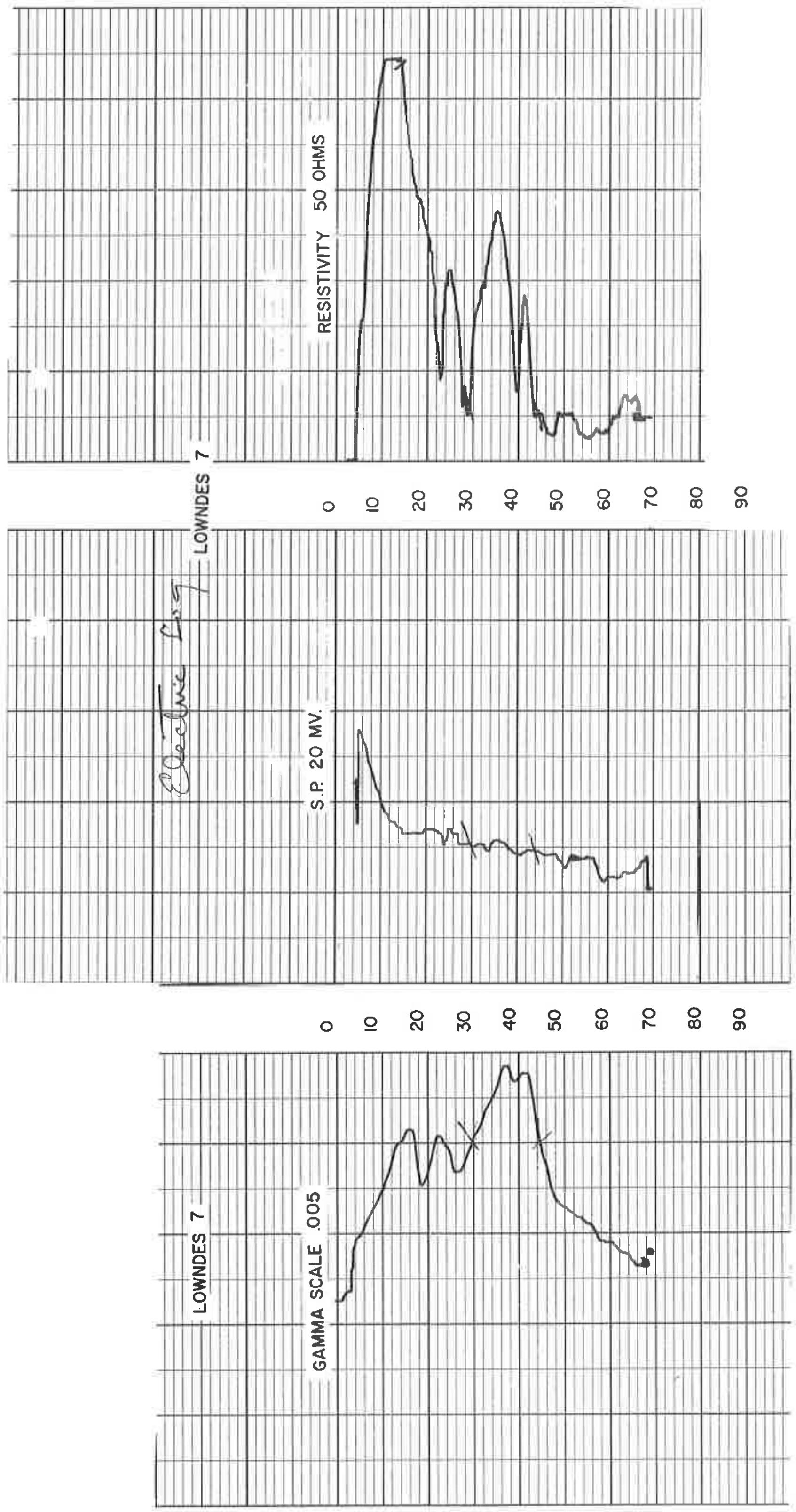

Figure AII-6. Electric and Gamma-Ray Logs - Lowndes County
Hole No. Lo-7

APPENDIX III

ANALYTICAL PROCEDURES

Introduction

Prior reports have noted several difficulties experienced in laboratory analytical work and investigations which resulted in modifications of procedures. Development of a lithium metaborate-carbon crucible fusion to replace sodium carbonate-platinum crucible fusion for analysis with an atomic absorption spectrophotometer has been particularly noteworthy.

It was considered desirable to compile and present complete details of the procedures as determined most appropriate to date for this South Georgia Minerals Program. In summary:

a. Wet chemical-titration techniques were used for acid insoluble residue (sand and insoluble silicates) and bone phosphate of lime (BPL).

b. Wet chemical-colorimeter techniques were applied to the determination of silica (SiO_2) and titania (TiO_2).

c. Atomic absorption spectrophotometry (with lithium metaborate-carbon crucible fusion) was found appropriate for lime (CaO), iron (Fe_2O_3), alumina (Al_2O_3), and magnesia (MgO).

It should be noted that to date the apparent advantages from using "as is" solid samples with an x-ray fluorescent unit have not outweighed the difficulties in preparing truly representative samples and obtaining satisfactory reproducible results. Technique modifications are being investigated, and when resolved, the use of an x-ray unit may be resumed. At that time, detailed procedures will be presented.

Note: For convenience of reproduction, each section is started on a new page.

General Laboratory Apparatus and Furniture

Balances

a. Analytical balance, ± 0.03 mg. precision
b. Semi-rough balance, Ohaus Dial-O-Gram 310
c. Rough balance, ± 0.01 gram precision

Furniture

a. Large fume hood
b. Water still, 1 gallon per hour or greater capacity
c. Gas, water, 110 v. AC, water aspirators

Heating Apparatus

a. Hot plates
b. Steam bath with polypropylene cover inserts to suit special teflon beakers*
c. Water bath, constant temperature
d. Large drying oven
e. Muffle furnace

Spectrophotometers

a. Spectrophotometer, Spectronic 20 or equivalent
b. Atomic absorption spectrophotometer with conventional and high temperature burners, lamps for elements of interest
    (1) acetylene, welding grade
    (2) air, breathing quality
    (3) nitrous oxide, medical grade

General Apparatus

a. Pipet washer with basket, for 36-inch pipets
b. Burets, Class A, 50 ml. with stands
c. Magnetic stirrers with glass or plastic coated bars, stirring bar retriever
d. Polyethylene reagent dispensers
e. Wash bottles, assorted sizes and types
f. Desiccator (large)
g. Platinum crucibles and dishes, 30 ml., 250 ml. with covers as budget permits
h. Teflon beakers* (approximately 100 ml.) with covers

Safety Equipment

Rubber gloves, safety goggles, laboratory aprons, asbestos gloves

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*L. Shapiro and W. W. Brannock, United States Geological Survey Bulletin No. 1144-A (1962) p. A5.

Acid Insoluble Residue

Apparatus

150 ml. beakers, 75 mm. long stem funnels, 250 ml. volumetric flasks, porcelain crucibles with covers, stirring rods, plastic bottles with caps, Whatman No. 40 filter paper 12.5 cm. dia.

Reagents (ACS Reagent Grade, except where noted otherwise)

Nitric acid

Hydrochloric acid

Procedure

1. Weigh 1.000 gram sample into 150 ml. beaker.

2. Add 30 ml. nitric acid and 10 ml. hydrochloric acid. Heat for 30 minutes or until solution is clear.

3. Dilute to about 100 ml. and filter through Whatman No. 40 paper catching filtrate in 250 ml. volumetric flask.

4. Transfer all of solid residue to filter paper and wash at least five times with distilled water, catching washings in the volumetric flask.

5. Dilute the filtrate to the mark with distilled water, mix, and reserve for phosphate analysis. The filtrate may be stored in tightly capped plastic bottles.

6. Place the filter paper and residue in a weighed porcelain crucible, cover, and burn off filter paper in muffle furnace at 700-800° C. When smoking stops remove cover and continue ignition for at least one hour.

7. Cool crucibles, preferably in a desiccator, weigh, and calculate per cent acid insoluble residue.

$$\text{Per cent acid insoluble residue} = \frac{\text{weight of ignited residue} \times 100}{\text{weight of sample}}$$

The acid insoluble residue consists of sand and insoluble silicates. This quantity is of interest in evaluating phosphate ores, where a more precise determination of silica is not required.

References

Methods Used and Adopted by the Association of Florida Phosphate Chemists (1960).

Discussion

Determination of sand and insoluble silicates is accomplished with little additional effort during sample preparation for phosphate analysis. Filtration of the digested sample during transfer to 250 ml. volumetric flasks improves the precision of the phosphate analysis and simplifies cleaning of volumetric ware between successive determinations. Further economy of effort with no significant sacrifice of precision can be achieved by igniting several samples in succession before cleaning and re-igniting the porcelain crucibles. The weight of the ignited crucible and ash may be taken as the empty crucible weight in the next determination.

Bone Phosphate of Lime (BPL)

Apparatus

Vacuum pump or aspirator and bench connections, filter flasks, 250 ml. beakers, 250 ml. volumetric flasks, 25 ml. pipets stirring rods, hot plate, water bath, burette assembly, Hirsch funnels, size 0. Closed system for filtering by vacuum, plastic dispensers for reagents, dropping bottles, glass fiber filter paper, 4.5 cm. dia.

Reagents (ACS Reagent Grade, except where noted otherwise)

Nitric acid

Hydrochloric acid

Ammonium molybdate solution

Mix 489 ml. nitric acid with 1148 ml. water and cool to room temperature. Dissolve 100 gm. molybdic acid 85 per cent in a mixture of 144 ml. ammonium hydroxide and 275 ml. water. Pour the ammoniacal molybdate solution gradually into the dilute nitric acid solution with constant stirring. Allow solution to stand at least 24 hours and filter immediately before using.

Ammonium nitrate solution

Dissolve 200 grams of ammonium nitrate in 1 liter of distilled water.

Standard sodium hydroxide solution

So-S-270 sodium hydroxide solution N/2 (Fisher Scientific Company) standardized against primary standard grade potassium acid phthalate as each container is opened. Normalities of these solutions have varied from 0.4998 to 0.5002 among the containers received. An arithmetical factor, the observed normality of the standard alkali divided by 0.3240 will be used in the calculations at the end of the determination.

Standard nitric acid solution

The standard acid is prepared to the exact normality of the standard alkali. For each liter of standard solution use 31.6 ml. ACS Reagent Grade concentrated nitric acid. When diluted to volume, mix thoroughly and determine the strength by titration against the standard alkali using phenolphthalein as the indicator. Correct to the exact normality of the standard alkali.

Phenolphthalein indicator solution

Dissolve one gram of phenolphthalein in 100 ml. of 95 per cent ethanol.

Sodium alizarin sulfonate indicator solution

Dissolve one gram of sodium alizarin sulfonate in distilled water.

Asbestos

Macerate acid washed and ignited asbestos with water in a Waring blender for one minute.

<u>Procedure</u>

1. Transfer 25 ml. aliquot portions of the filtrates from the acid insoluble residue determinations and from a similarly treated 0.5000 gram portion of National Bureau of Standards sample No. 120a into 250 ml. beakers. Add a glass stirring rod to each beaker.

2. To each beaker add 25 ml. of ammonium nitrate solution and stir well with a glass rod.

3. Add ammonium hydroxide from a dropping bottle until the solution is just alkaline, using sodium alizarin sulfonate as the indicator.

4. Add nitric acid from a dropping bottle until the solution is just acid.

5. Adjust the volume to at least 100 ml. with distilled water.

6. Add, gradually and with constant stirring, 25 ml. of ammonium molybdate reagent filtered immediately before use.

7. Place the beakers in a water bath for 30 minutes at 43° C, stirring intermittently.

8. Prepare funnels for vacuum filtration by placing a 4.5 cm. glass filter paper in a Hirsch funnel and adding sufficient asbestos slurried in water to form a continuous mat. Turn on aspirator and pull water into filter flask, leaving a thin but continuous asbestos mat on the filter paper.

9. At the end of 30 minutes pour the supernatant liquid through the prepared filter, wash the precipitate in the beaker twice by decantation, and transfer the yellow precipitate to the filter, using a fine stream of water from a wash bottle. Small amounts of precipitate adhering to the beaker and stirring rod can be ignored.

10. Wash the precipitate 20 times with small portions of water, allowing the funnel to pull dry after each addition of water.

11. Break the vacuum on the system, wipe the liquid, if any, from the outside of the funnel, and loosen the precipitate and filter paper with the stirring rod. Transfer the filter paper and precipitate to the original beaker.

12. Rinse the inverted funnel with a small stream of water, allowing the water to pass into the beaker, then wipe the inside of the funnel with a small piece of filter paper. Add the paper to the beaker.

13. To each beaker add about 25 ml. of water and stir to break up the cake of ammonium phosphomolybdate and asbestos.

14. From a burette add sufficient standard sodium hydroxide solution to dissolve the yellow precipitate. Stir vigorously while adding the standard alkali and use a small excess to insure that no yellow solid remains. With the stirring rod raise a piece of the filter paper to wipe the walls of the beaker with the alkaline solution. Rinse the walls of the beaker with a small quantity of water to flush any adhering alkali into the solution.

15. Add two drops of phenolphthalein and titrate the excess alkali with standard nitric acid. The end point is the last faint pink or the first dead white. One or two drops of a 48 per cent aqueous formaldehyde solution may be used to make the end point appear sharp. (The apparent end point may appear to differ by as much as 0.1 ml. among several analysts, so it is desirable that one analyst titrate the samples and standard which make up the set of determinations.)

16. Calculate the per cent bone phosphate of lime as follows:

$$\text{Per cent } P_2O_5 = \frac{\text{normality of base}}{0.3240} (\text{ml. standard NaOH} - \text{ml. standard } HNO_3)$$

$$\text{Corrected Per cent } P_2O_5 = \text{Per cent } P_2O_5 \times \frac{34.4}{\text{apparent per cent } P_2O_5 \text{ in standard}}$$

$$\text{Per cent BPL} = \text{Corrected Per cent } P_2O_5 \times 2.185$$

Notes

a. On matrix samples, steps 3, 4, and 5 of this procedure may be omitted. Should the BPL concentration be greater than 8 per cent, an additional aliquot portion of the sample should be carried through the entire procedure.

b. Overnight precipitation at room temperature will give results similar to those obtained from 30 minutes at 43° C.

c. Glass fiber filter paper will permit more rapid filtration without loss than will conventional paper.

d. A thin, continuous asbestos mat will permit less solid loss during filtration than will a thick mat.

e. The full force of the aspirator is required during filtration. Operating with a lesser flow of water or with air leaks in the closed filtration system produces a less compact asbestos mat and consequent loss of precipitate through the filter.

f. Maceration of acid washed and ignited asbestos fiber for one minute with water in a Waring blender produces a uniform slurry which can be used to form a uniform filter mat. Excessive maceration leads to a dense mat and slow filtration.

References

1. Methods Used and Adopted by the Association of Florida Phosphate Chemists (1960).

2. Official Methods of Analysis of the Association of Official Agricultural Chemists, 8th edition (1955).

3. R. D. Duncan, and J. A. Brabson, "A Critical Evaluation of the Ammonium Molybdophosphate Method for Phosphorous," Journal of the Association of Official Agricultural Chemists, 48, 1092-95 (December 1965).

Discussion

The volumetric phosphate determination is employed by a majority of the laboratories participating in the A.O.A.C. Magruder Check Sample Program. Its principal disadvantage is a large standard deviation. Its advantages are simplicity, rapidity, economical apparatus and reagents, and ease of training technicians to use the method.

While the apparent end point of the titration may differ by as much as 0.3 per cent BPL among different analysts titrating aliquot portions from the same sample, a single operator can consistently obtain results which agree within 0.05 per cent BPL. Including aliquot portions drawn from National Bureau of Standards Sample No. 120a and applying an arithmetical correction factor derived from analysis of the standard sample leads to good agreement among results from several analysts.

Several procedures have been examined for precipitation of phosphate as ammonium phosphomolybdate. No difference has been observed between samples precipitated for 30 minutes at 35° C, 30 minutes at 43° C, 30 minutes at 55° C, and overnight at room temperature. Routine procedure is 30 minutes at 43° C or overnight at room temperature when a large number of samples must be processed. Standard samples are always included among sets of unknowns. Phosphate concentration in the aliquot portions has a marked effect on loss of ammonium phosphomolybdate through the filter and, consequently, on the analytical result. This observation is attributed to the classical principle that fewer precipitation nuclei form in a solution dilute with respect to either ingredient or precipitant than form in concentrated solutions. These relatively few nuclei grow to relatively large particles rather than the large number of small particles precipitated from more concentrated solutions. While this effect has been negligible in samples containing only a few per cent phosphate, it may lead to deviations as high as 4 per cent in samples where the BPL concentration approaches 70 per cent. Dilution of samples containing more than 8 per cent BPL before precipitation has produced an easily washed precipitate and results which agree within one per cent of the quantity of phosphate found.

Purchase of 0.500 Normal alkali solution (So-S-270 Sodium Hydroxide Solution N/2, Fisher Scientific Company) has eliminated the laborious preparation of 0.3240 N. carbonate free reagent from oily alkali. Standardization against primary standard grade potassium acid phthalate has indicated normalities of 0.4998 to 0.5002 on the several containers purchased. Standard nitric acid was prepared to the exact normality of the alkali in each container. A constant arithmetical factor to compensate for the difference of normality of the titrants used and the 0.3240 normality specified in the A.O.A.C. method was applied to all determinations by means of a desk calculator.

Sulfate interference was not observed in any of the analyses. Ten milliliter portions of 5 per cent barium chloride added to the filtrate from each of the matrix samples before dilution to 250 ml. did not produce any turbidity. Only faint turbidity was observed in a few of the processed samples. The barium sulfate precipitate, where observed, was allowed to settle overnight, and the aliquot portion was drawn from the clear liquid.

Silica (SiO_2)

Apparatus Required

Nickel crucibles, 75 ml. with covers; pipets, 1 ml., 2 ml., 4 ml., and 8 ml.; volumetric flasks, 100 ml., and 1000 ml.; spectrophotometer, polyethylene dispenser, matched cuvettes to suit spectrophotometer, long-stemmed plastic funnels, filter paper, Whatman No. 40, 12.5 cm.

Reagents (ACS Reagent Grade, except where noted otherwise)

NaOH solution, 30 per cent

Dissolve 1 pound of NaOH pellets in 1500 ml. of water in a polypropylene or stainless steel beaker. Cool and store in a plastic bottle.

HCl, 1:1

National Bureau of Standards Sample No. 70a, feldspar

Ammonium molybdate solution

Dissolve 7.5 of $(NH_4)_6\ Mo_7O_{24}\cdot 4H_2O$ in 75 ml. of water, add 25 ml. of 1 + 4 H_2SO_4, and mix. Store in plastic bottle.

Tartaric acid solution, 10 per cent

Prepare 500 ml. and store in plastic bottle. Discard when sediment forms.

Reducing solution

Dissolve 0.7 g. of sodium sulfite in 10 ml. of water. Add 0.15 g. of 1-amino-2-naphthol-4-sulfonic acid and stir until dissolved. Dissolve 9 g. of sodium bisulfite in 90 ml. of water, add this solution to the first solution and mix. Do not store this reagent for more than 3 days.

Procedure

1. Transfer 5 ml. portions of 30 per cent NaOH solution by means of a plastic dispenser into a series of 75 ml. nickel crucibles. (Use

one crucible for each sample, two for standards, and one for a blank). Crucibles should be cleaned with dilute HCl before use.

2. Evaporate solutions to dryness on a hot plate. Ignore a small amount of spattering (a convenient procedure is to perform the evaporation overnight).

3. Accurately weigh (to the nearest 0.1 mg.) 50 mg. of each sample, two 50 mg. portions of National Bureau of Standards Sample No. 70a (feldspar) and transfer to the crucibles containing NaOH.

4. Cover and heat the crucibles to dull redness for 5 minutes (a muffle furnace at 700-750° C is suitable) and allow the melts to cool.

5. Add about 50 ml. of water to each crucible, cover and allow to stand until the melt is disintegrated. (Overnight, or for about 1 hour if the solutions are stirred occasionally.)

6. Transfer the contents of each crucible to a 1000 ml. volumetric flask (previously rinsed with 1:1 HCl) containing about 400 ml. of water and 20 ml. of 1:1 HCl. A long-stemmed plastic funnel should be used so that the alkaline solution will drain directly into the dilute acid without contacting the walls of the flask. Police and wash each crucible to assure complete transfer of solution and precipitate.

7. Dilute each solution to the mark and mix well.

8. Filter approximately 100 ml. of the solutions through dry papers (Whatman No. 40 or equivalent) and store in tightly stoppered plastic bottles.

9. Transfer 8 ml. portions of the standards, samples, and blank to 100 ml. volumetric flasks. Use Class A 8 ml. transfer pipets. Add approximately 50 ml. of water to each flask.

10. Add 2 ml. of the ammonium molybdate solution with a pipet, swirling the flask during addition. Allow to stand for 10 minutes.

11. Swirl flask while adding 4 ml. of tartaric acid solution by pipet.

12. Add 1 ml. of the reducing solution while swirling the flasks.

13. Dilute to the mark, mix well, and allow to stand for at least 30 minutes.

14. Determine the per cent transmittance at 640 mμ for each solution using the blank as the reference (caution: <u>check cuvettes for match with single solution at 640 mμ</u>).

15. Calculate the per cent SiO_2:

a) Convert transmittance to absorbance by means of standard table.

b) Calculate the factor:

$$\frac{(\text{Per cent } SiO_2 \text{ in the standard}) \times (\text{weight of standard})}{\text{Average absorbance of the standard}} = \text{factor}$$

c) Calculate the per cent SiO_2:

$$\frac{(\text{Factor}) \times (\text{absorbance of the sample})}{\text{Weight of the sample}} = \text{per cent } SiO_2$$

Discussion

With only a few modifications for convenience, this method follows the procedure described by L. Shapiro and W. W. Brannock in <u>Rapid Analysis of Silicate, Carbonate, and Phosphate Rocks</u>, United States Geological Survey Bulletin No. 1144-A (1962), pp. A21-22, and A-24. Advantages of the method over the hydrofluoric acid procedure are elimination of platinum ware in the initial fusion step and elimination of prolonged filtration and evaporation steps. The volumetric ware must meet the tolerances prescribed by the National Bureau of Standards. While the procedure includes several transfers of precise quantities of sample and reagents, these operations are readily performed by careful technicians. Deviations greater than ± 2 per cent of the average amount of silica determined have not been observed. Deviations among most of the replicate determinations has been less than 1 per cent of the SiO_2 in the sample.

However, determinations of SiO_2 content of high quality sands from cores gave excessively high values; some being over 100 per cent, an obvious impossibility. Selected samples were analyzed by both the colorimetric method detailed above and by the slower but reliable gravimetric techniques. Very close checks were obtained with a sample of Reagent Grade Silic Acid. Natural sands samples showed the colorimetric method, which based upon NBS 70a standard sample, to give results averaging 4.7 per cent higher than by the gravimetric procedure.

A recognized authoritative laboratory in the United States, frequently used as a referee laboratory, was aware of the difficulties with the colorimetric procedure and used the slower-but-surer gravimetric method.

The cause of this discrepancy has not been determined. It is believed that but very small amounts of phosphates and arsenic would enhance the color developed and result in a fictitiously high value for SiO_2. Accordingly, the colorimetric method is useful for screening and preliminary evaluation, but for valid evaluation of specific areas, recourse must be had to gravimetric procedures.

Titanium Dioxide (TiO_2)

Apparatus

Hot plate; volumetric flasks, 100 ml. and 250 ml.; pipets, 1 ml., 2 ml., 5 ml., 10 ml., and 15 ml.; nickel crucibles, 7 ml.; Teflon stirring rods; filter funnels; Whatman No. 40 filter paper; wash bottles; beakers, 150 ml.; spectrophotometer, Bausch and Lomb Spectronic 20 or equivalent.

Reagents (ACS Reagent Grade, except where noted otherwise)

Sulfuric acid, 1:19*

Sodium hydroxide, 30 per cent

Dissolve 1 pound of ACS reagent grade pellets in 1500 ml. of distilled water.

Sodium carbonate solution, 1 per cent

Hydrogen peroxide, 3 per cent

Standard TiO_2 solution, prepared as follows:

1. To a 75 ml. nickel crucible thoroughly cleaned and rinsed with 1:19 H_2SO_4 add 5 ml. of 30 per cent sodium hydroxide solution and evaporate to dryness on a hot plate. (A small amount of spattering can be ignored.)

2. Weigh 0.1013 g. of National Bureau of Standards Sample No. 54a (99.6% TiO_2) and distribute over the dried NaOH in the crucible.

3. Fuse 5 minutes at 700° C in a muffle furnace and cool.

4. Add about 50 ml. of water and let stand overnight or stir intermittently with a Teflon rod until the melt is dissolved (at least 1 hour).

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*1 part H_2SO_4 added to 19 parts water.

5. Transfer the contents of the crucible to a 150 ml. beaker, using a Teflon rod to dislodge any solid material adhering to the crucible. Rinse the crucible with 1 per cent Na_2CO_3 solution and add the washings to the beaker.

6. Add about 25 ml. of 1:19 H_2SO_4 to the crucible and boil on hot plate.

7. Filter contents of the beaker through a dry filter paper, keeping as much of the residue as possible in the beaker. Rinse the beaker and residue on the paper with 1 per cent Na_2CO_3 solution. Discard filtrate and washings.

8. With a fine stream of 1:19 H_2SO_4 from a wash bottle transfer the insoluble residue from the filter paper back into the beaker.

9. Add the contents of the crucible to the beaker. Rinse the crucible with small portions of 1:19 H_2SO_4 and add the washings to the beaker. Add 1:19 H_2SO_4 to make a volume of approximately 50-75 ml.

10. Place the beaker on a hot plate and dissolve the residue. (It will probably be necessary to concentrate the acid by evaporation.)

11. When the solution is clear, remove the beaker from the hot plate, cool to room temperature, and make up the original volume (50-75 ml.) with distilled water.

12. Transfer the clear solution to a 250 ml. volumetric flask. Rinse the beaker with the dilute acid, adding the washings to the flask, and dilute to the mark with 1:19 H_2SO_4. Mix well and transfer to a tightly stoppered plastic bottle for storage.

Procedure

1. Weigh 0.1000 g. portions of National Bureau of Standards sample No. 69a (bauxite) and samples into a series of 75 ml. nickel

crucibles prepared as for the standard TiO_2 fusion. Reserve one crucible for a blank.

2. Fuse and dissolve the samples as in steps 3-11 of the standard solution preparation. Do not let the total volume of dilute acid exceed 75 ml.

3. Transfer the clear solutions from the beakers to a series of 100 ml. volumetric flasks.

4. To 100 ml. flasks containing about 50 ml. of 1:19 H_2SO_4 transfer 1, 2, 5, 10, and 15 ml. aliquot portions of the Standard TiO_2 solution, using Class A pipets.

5. Add 5 ml. of 3 per cent H_2O_2 solution to each of the flasks, dilute to the mark, and shake well.

6. After 10 minutes read the per cent transmittance of the standard and sample solutions using a Bausch and Lomb Spectronic 20 Spectrophotometer, or equivalent, at a wave length of 425 millimicrons, setting 100 per cent T by means of the blank solution.

7. Convert the per cent transmittance to absorbance by means of a standard table.

8. Construct a standard curve of absorbance vs. concentration from the absorbances of the blank and the diluted TiO_2 standard solutions. The 1, 2, 5, 10, and 15 ml. aliquot portions of TiO_2 standard solution correspond to 0.4, 0.8, 2.0, 4.0, and 6.0 per cent TiO_2, respectively.

9. Check the apparent per cent TiO_2 of the NBS 69a standard solution against the standard curve. The per cent TiO_2 found should be 2.78 ± 0.1 per cent.

Notes

1. The alkaline filtrate and washings may be added to dilute HCl in a liter flask and used for determination of SiO_2 (see procedure for SiO_2).

2. Identical results have been obtained from 0.1013 g. portions of NBS Sample No. 154a (99.6 per cent TiO_2) fused with Na_2CO_3 in platinum crucibles rather than with NaOH in nickel crucibles. The plot of absorbance vs. concentration was linear over the range of zero to six per cent TiO_2.

References

1. I. M. Kolthoff and E. B. Sandell, Textbook of Quantitative Inorganic Analysis, revised edition, Macmillan, New York, (1947), pp. 742-744.

2. L. Shapiro and W. W. Brannock, U. S. Geological Survey Bulletin 1144A, (1962), pp. A21-A22.

Atomic Absorption Spectrophotometry (Iron and Aluminum)

Apparatus

Graphite crucibles, Spex No. 7152 (Spex Industries, Inc., Metuchin, N. J.); tri-pour beakers, 100 ml., with caps; diamonite mortar and pestle, volumetric flasks, Class A, 50 ml., 100 ml., 250 ml., 500 ml., 1000 ml.; pipets, Class A, 1 ml., 2 ml., 4 ml., 5 ml., 8 ml., 10 ml., 15 ml., 25 ml., 50 ml., 100 ml.

Note: All volumetric glassware must be scrupulously cleaned, preferably with chromic acid and distilled water.

Reagents

Purified Lithium Metaborate

Dissolve 100 grams of lithium metaborate (K and K Industries, Inc., Plainview, N. Y.) in 1000 ml. hot water and filter immediately. Allow the solid to crystallize for several days then decant off supernatant liquid. Transfer the crystals to a polypropylene container and dry at 105° C. (Caution: Glass will break because of the expanding solid, or a polyethylene container may melt at this temperature.) Heat small quantities of the dried solid at 300° F in a platinum dish until expansion is complete, then crush the expanded solid and dry for two hours at 400° F. (The expansion step is conveniently accomplished in a muffle furnace with the door open. It is preferable to break the material into small lumps and expand only small quantities at one time, as larger lumps tend to expand vigorously, scattering the material over the inside of the furnace.) The dried material is stored in a desiccator. Recovery is only about 50 per cent of the initial solid. The yield may be increased to some extent by harvesting additional crystals from the supernatant liquid after further evaporation. Grind small quantities of the purified material to a fine powder with a "diamonite" mortar and pestle as needed.

Four per cent nitric acid

Dilute 40 ml. concentrated nitric acid to 1 liter with distilled water. Store in tightly stoppered glass or plastic bottle.

Stock diluent solution

Dissolve 58.65 g. of La_2O_3 (American Potash and Chemical Corporation) in 250 ml. of concentrated HCl. (Add acid slowly until the material is dissolved.) Dilute this solution to 1000 ml. Dilute 50 ml. of concentrated nitric acid to about 500 ml., dissolve 25 g. of purified $LiBO_2$ in the dilute nitric acid, and add distilled water to make 1000 ml. Mix 200 ml. of La_2O_3 solution with 800 ml. of $LiBO_2$ solution to make the stock diluent. Store in a tightly stoppered plastic bottle.

Standard rock samples

a) National Bureau of Standards Sample No. 120a phosphate rock.
b) National Bureau of Standards Sample No. 70a feldspar.
c) National Bureau of Standards Sample No. 69a bauxite.
d) National Bureau of Standards Sample No. 1a limestone.
e) National Bureau of Standards Sample No. 104 burned magnesite.

Fusion Procedure

1. Weigh 0.1000 gram portions of each of the standards and unknown samples into evaporating dishes or watch glasses.

2. Weigh 0.50 grams finely ground purified $LiBO_2$ into each container and an additional 0.50 gram portion to serve as a blank.

3. Mix the flux and sample powders thoroughly and transfer to graphite crucibles. Store in desiccator until a furnace is available.

4. Ignite in muffle furnace for 15 minutes at 950° C.

5. Pour molten bead into tri-pour beaker containing about 25 ml. four per cent nitric acid, add stirring bar, cover, and place on magnetic stirrer to dissolve melt.

6. Dilute to 50 ml. with 4 per cent nitric acid solution.

7. Transfer solution to 100 ml. volumetric flask, rinsing original container with stock diluent and dilute to mark with stock diluent solution. Mix well.

8. Filter through Whatman No. 40 filter paper and store solution in tightly stoppered plastic bottle.

Notes

The atomic absorption spectrophotometer used is a Perkin Elmer Model 303. The manufacturer's operating instructuions are followed rigorously. Hollow cathode lamp types used are calcium-magnesium dual purpose, iron, and aluminum.

Analytical Procedures

Alumina (Al_2O_3)

Aliquot portions of the standard and sample solution were diluted to less than 100 parts of aluminum per million with the stock diluent. Readings are made at 3092 Å using an air-acetylene flame. Per cent Fe_2O_3 in the samples is calculated by comparing the sample absorbance with the standard curve taking into account the blank and the respective dilutions of the standard and sample solutions.

Iron Oxide (Fe_2O_3)

Aliquot portions of the blank, standard, and sample solutions are diluted to the zero to 20 parts of iron per million range with the stock diluent. Readings are made at 2483 Å using an air-acetylene flame. Per cent Fe_2O_3 in the samples is calculated by comparing the sample absorbance with the standard curve taking into account the blank and the respective dilutions of the standard and sample solutions.

Lime (CaO)

Portions of the treated standard, sample, and blank solutions are mixed with suitable precisely known quantities of stock diluent solution to bring the Ca concentration into the range of zero to ten parts per million. Readings are made at 4227 Å using an air-acetylene flame. Standard tables are used to convert instrument readings to absorbance. CaO concentrations in the unknown samples are calculated after comparison of their absorbances with a curve of absorbance vs. concentration in the standards, taking into account the blank and the respective dilutions of the samples and standards.

Magnesium Oxide (MgO)

Aliquot portions of the blank, standard, and sample solutions are diluted to the zero to 2 parts of Mg per million range with the stock diluent. Readings are made at 2852 Å using an air-acetylene flame. Per cent MgO in the samples is calculated by comparing the sample absorbance with the standard curve taking into account the blank and the respective dilutions of the standard and sample solutions.

References

1. Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer Corporation (1964).

2. Dr. Kevin C. Beck, Georgia Institute of Technology, private communications.

Discussion

While atomic absorption spectrophotometry offers the advantage of rapid determination of many elements in solution without laborious separation procedures, the method is susceptible to a number of errors unless precautions are taken. The most conspicuous of these disadvantages is matrix dependence, i.e., the apparent variation of the ratio of absorbance to concentration among various types of sample. Where the original sample consists of a single known type of mineral (as is seldom the case) a standard curve might be plotted from diluted solutions prepared from rock of known composition and similar type.

This procedure was explored in the early phases of the study by performing wet chemical analyses of mixtures believed to be representative of the samples and using the data to construct standard curves. As the variety of samples outstripped the wet analytical capacity of the laboratory, a search for "matrix independent" procedures was undertaken.

Various sample opening procedures, which included alkaline fusions, acid fusion with potassium bisulfate, peroxide fusions, and dissolving the samples in mixtures of hydrofluoric acid with other mineral acids in special covered teflon beakers on a steam bath left residues and led to inconsistent results. Fusion with purified lithium metaborate produced clear solutions containing only graphite flecks from the crucibles. Ignition of the solid materials filtered from these solutions indicated no undissolved mineral residue. Calcium determinations by atomic absorption on these solutions were still matrix dependent, whether performed with nitrous oxide-acetylene or air-acetylene flame. Aluminum determinations, with a nitrous oxide-acetylene flame, using samples fused and dissolved by this method did not show matrix dependence.

The use of lanthanum oxide to suppress formation of refractory compounds of calcium with phosphate and alumina has been suggested. (Analytical Methods for Atomic Absorption Spectrophotometry, Perkin Elmer Corporation, Norwalk, Connecticut, January 1964, Standard Conditions for Calcium.) Use of lanthanum oxide (1.0 per cent) solution as a stock diluent produced erratic results with an air-acetylene flame. With the nitrous oxide-acetylene flame the matrix effect persisted and became more pronounced with successive dilutions. Substitution of 1.0 per cent $LiBO_2$ and 1 per cent La_2O_3 in 4 per cent nitric acid for water as the stock diluent produced ratios of absorbance to concentration which were similar for equally concentrated solutions prepared from five different National Bureau of Standards rock samples. These samples are all used in preparing the standard curves constructed with each set of determinations. The method is reliable for the range of zero to ten parts of calcium per million. It is believed that the dissolved lithium acts as a buffer to suppress ionization of calcium at the high nitrous oxide-acetylene flame temperature. The stock diluent system developed for determination of calcium also leads to good results in the magnesium analyses.