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30. MAJOR ELEMENTS: ANALYTICAL CHEMISTRY ONBOARD AND PRELIMINARY RESULTS¹

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INTRODUCTION

In 1972, chemical analysis of rocks using a dispersive X-ray fluorescence system was shown to be practicable at sea during the "Gibraco cruise" of the R.V. *Jean Charcot* (Bougault and Cambon, 1973). The same equipment and techniques were used to perform analyses on *Glomar Challenger* during Leg 37. The aim of this report is to describe the instruments used, the basic routine operations, the problems encountered, and how they were solved. From the experience of analytical work onboard and the precision of the results which is discussed, some brief recommendations are made to IPOD for shipboard chemical analysis. The preliminary results obtained are discussed in terms of chemical units compared to lithological units defined onboard.

INSTRUMENTS

All instruments, including the X-ray fluorescence unit, the furnace, the balance, the motor-driven crusher mortar, and the necessary accessory laboratory tools were sent from France to Rio de Janeiro in a laboratory van. The van (Figure 1), which is a standard 8 × 8 × 16 ft shipping container, was fixed to the deck near the chemical laboratory, the X-ray fluorescence unit remaining in it. The furnace, the scale, the mortar, and all other equipment for sample preparation were set up in the ship's permanent chemical laboratory.

The Siemens X-ray fluorescence unit (Figure 2) is composed of: (1) a stabilized high-voltage power transformer supplying the X-ray tube; (2) the XRF instrument itself (Siemens type VRS; goniometer, gas flow detector, solid scintillation detector, and four dispersing crystals); (3) a measuring panel including facilities for possible energy separation; and (4) a print-out.

The stability of the complete unit onboard proved to be the same as on shore (Bougault and Cambon, 1973; Boucher et al., 1974), and no problems were encountered during Leg 37.

The balance (Figure 3) is a LM600 Beckman scale which has been shown to be suitable for precision weighing onboard (Boucher et al., 1974). The absolute precision at sea in the range of 500 to 700 mg is better than 1 mg. This balance was set up on the small gimbal platform of the chemical laboratory.

The furnace (Figure 4) is an ERSEM, with no special improvements. It was chosen because it has no mer-

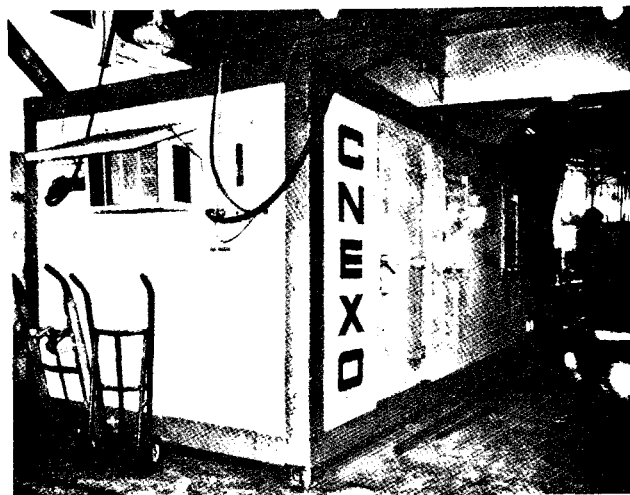


Figure 1. Laboratory van.

cury switches, which would not operate on a moving ship. Two fireproof holders (Figure 5) to hold the crucibles motionless can be put into the furnace simultaneously, thus allowing the preparation of six samples at the same time.

The crucibles used are "OPR crucibles" made of a platinum, gold, and rhodium alloy. The melt is cooled inside the crucibles and then the glass disks are removed. This procedure is important onboard because the ship's motion does not permit the melt to be cooled on a flat carbon or steel tray.

ROUTINE ANALYTICAL WORK

It is necessary to make glass disks to have good precision using an X-ray fluorescence method. The use of glass eliminates the grain size effect of a powder, and the matrix effect is eliminated by choosing a suitable composition for the flux. The flux used is made of 3.5 g of $\text{Li}_2\text{B}_4\text{O}_7$ and 0.5 g of La_2O_3 , a standard composition for the "heavy absorber method."

The first operation is to break the sample into small fragments and to powder it with the motor-driven agate mortar. At the beginning of the cruise samples were fragmented with a hammer until a more suitable Halbich mortar was found onboard. Tests previously made on shore indicate that no contamination for Ni, Fe, and Cr is introduced by use of this equipment.

As it is necessary to have the same quantity of material in each glass disk, the powdered samples were first ignited at 1050°C in platinum crucibles; the loss of weight at 1050°C was determined by weighing the sample before and after ignition. A half a gram of the ig-

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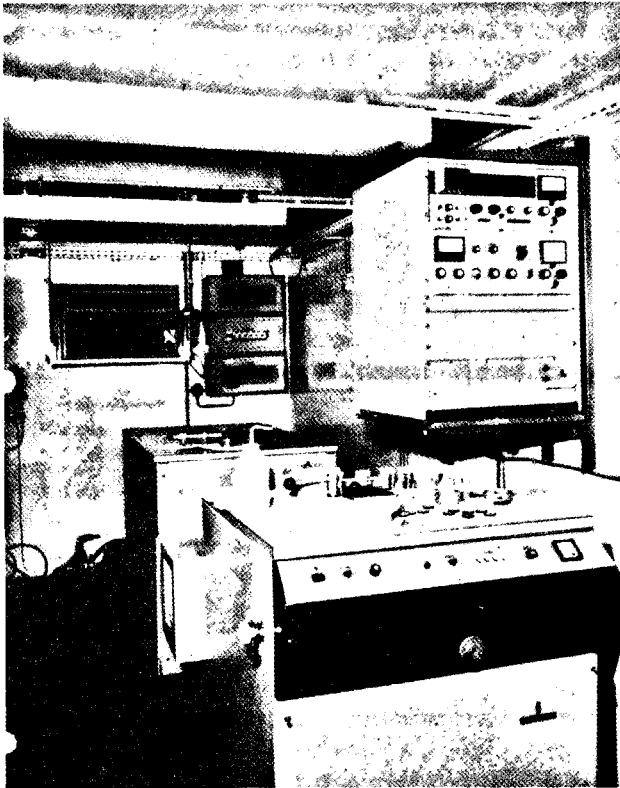


Figure 2. X-Ray fluorescence unit tied in the van.

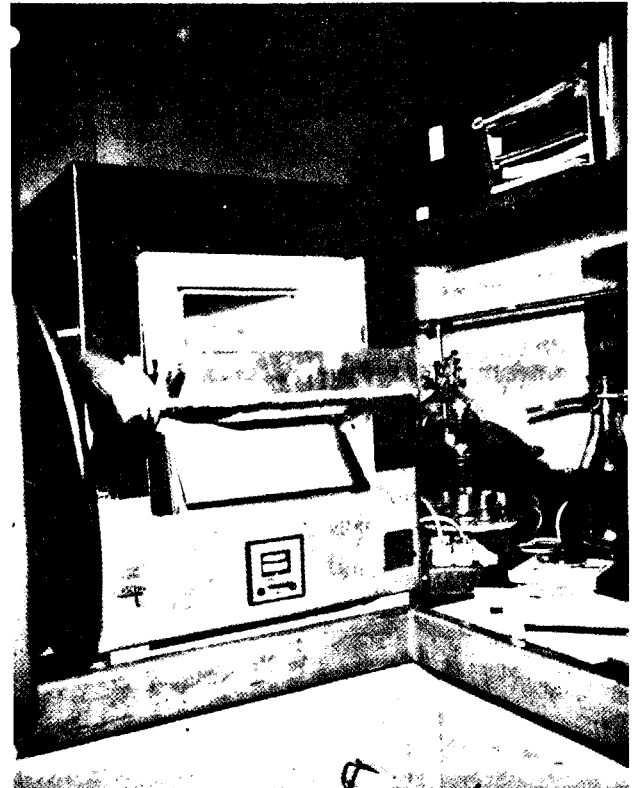


Figure 4. ERSEM furnace.

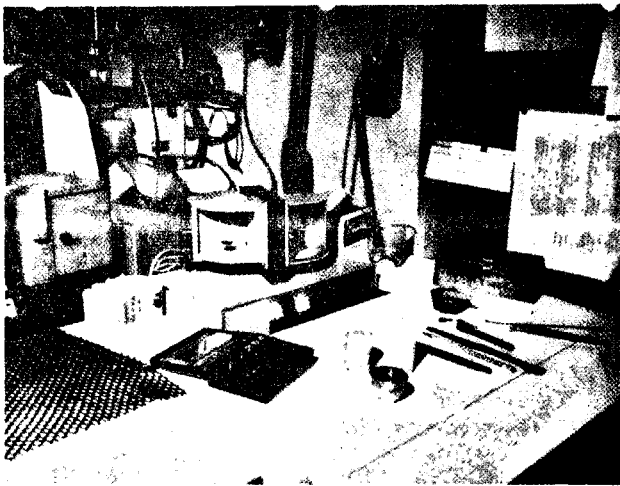


Figure 3. LM600 electromagnetic balance.

nited sample was weighed and mixed in the OPR crucible with the flux, which was previously prepared and weighed on shore. Six samples are simultaneously put into the furnace (with the fireproof holder) and melted at 1050°C. After 30 to 45 min they are removed from the furnace, allowed to cool, and unmolded. This operation was repeated three times to let possible bubbles escape and to ensure the homogeneity of the glass disks.

At the beginning of the cruise some problems developed with the shape of the glass disks. Because the

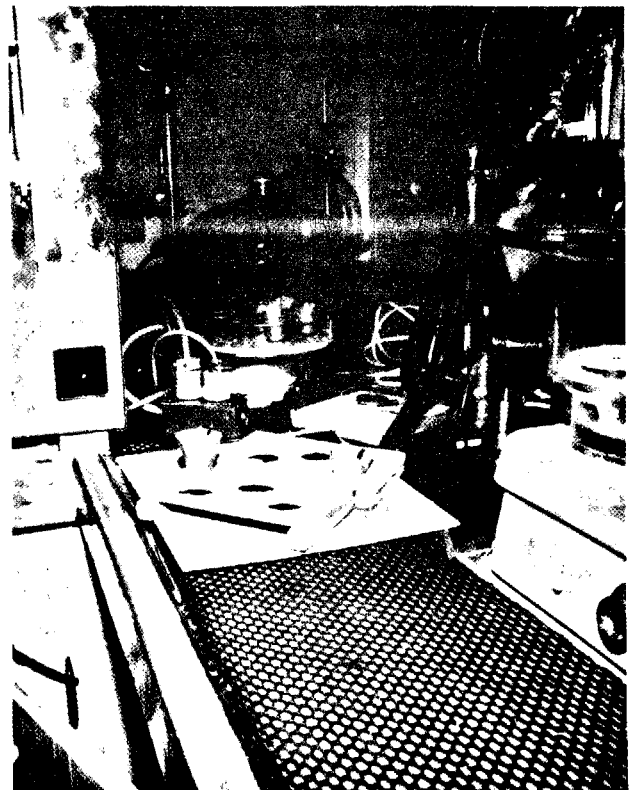


Figure 5. "OPR" (gold, platinum, rhodium) crucibles and fireproof holders.

crucibles are not new, as was the case in 1972 (during the Gibraco cruise), some of them are slightly bent so that the bottom surface of the glass disk is not quite flat. A flat surface is, of course, important for the geometry of X-rays and some totals of analyses (without Na₂O) were found to be too low (down to 92%). We discovered the cause of this problem by comparing the totals with the crucible numbers. Thus, the glass disks were ground to a flat surface, using a lap with 400 and 1000 grit (Figure 6). There was no evidence of contamination by grinding the glass disks. Comparison of results before and after grinding showed that grinding them increased all element concentrations, giving totals without Na₂O for most samples between 96.4 and 97.5.

The X-ray fluorescence unit operated without any problem throughout the cruise. Calibration curves were made onboard. The following elements were analyzed (as oxides): SiO₂, Al₂O₃, MgO, CaO, K₂O, TiO₂, and P₂O₅ (for some samples). The variation of count rate from one day to another was less than 2%. To take into account these possible variations from one day to another and not be obliged to make calibration curves every day for all the elements, we plotted the ratio of the count rate of a standard to the most concentrated standard for the elements involved (Figure 7). This way, measuring the most concentrated standard was sufficient to take into account the daily variations of the instrument, the calibration curve being tested by at least two other standards. Calculations were made with a Hewlett-Packard HP-45 hand computer to deduce concentration from the count rate, taking into account the count rate of the most concentrated standard, the background, and the slope and intercept of the straight line calibration.

PRECISION OF MEASUREMENTS

To test the precision of the method onboard, a tholeiitic basalt sample (provided by F. Aumento) was analyzed by the routine procedure. This sample was previously analyzed by four laboratories; the mean value of these determinations and 2 standard deviations are indicated in Table 1. The values determined onboard ship (Table 1) are all inside two standard deviations.

To test the reproducibility of the method as a function of time, measurements made on different days for one element around 15% (CaO), another around 1% (TiO₂), and a third around 0.2% (K₂O) are given in Table 2. For the less concentrated element (K₂O) the count rate is on the order of 4000 impls/20 sec; counting 20 sec, for this order of magnitude of 0.2% in K₂O the absolute precision is within ± 0.02 .

From this information it can be concluded that the precision of the method used onboard should be equivalent to the precision of the method used on shore.

COMMENTS ON SHIPBOARD MAJOR ELEMENTS DETERMINATIONS

The comparison between shipboard measurements and shore-based laboratories results now available is given in Table 3. It is not possible to calculate a σ value, because these results are for adjacent samples and are not repeated analyses of the same samples. Thus, in order to compare shipboard and land-based results, we

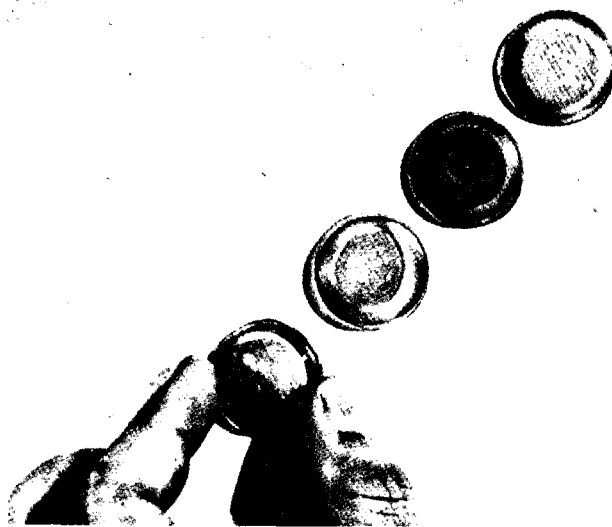


Figure 6. Glass discs: the different stages of grinding show the slope of unmold glass discs: the bottom of some crucibles were bent.

have chosen the mean difference between shipboard and AUF results for each element. The comparison shows that the mean difference for a given element is close to the mean difference observed between the results for two land-based laboratories (MUN or ML and AUF) and close to the 2σ values calculated from four laboratory determinations of the same sample. Even if it is not statistically correct to compare a mean difference to 2σ values, this evaluation of shipboard measurements confirms that the precision of shipboard determinations is of the same order of magnitude as that of land-based laboratories. The observed differences are not all random compared to AUF results—SiO₂ and Al₂O₃ generally are smaller and Fe₂O₃ larger. Note that the mean difference for K₂O is less than 0.01.

Because measurements made onboard were intended mainly to serve as a guide for sampling, precision is an important factor in the shipboard method; the results obtained during Leg 37 show that the necessary precision was achieved and that the number of analyzed samples (129) allows the samples to be classified into chemical units; the chemical units defined onboard have been confirmed by further major element analyses and transition trace element study.

From my experience, I give the following recommendations to IPOD for both equipment and personnel in order to obtain the best results from an X-ray lab onboard a drilling vessel:

1) Equipment: the laboratory should, in addition to equipment used during Leg 37, include X-ray diffraction equipment that can operate on a moving ship: it would permit determination of minerals that cannot be determined or are poorly determined in thin section.

2) Personnel: a geochemist familiar with XRF, one technician full time, one technician half time.

With this equipment and personnel the following could be done: (1) Eight samples could be analyzed per day for major elements, for instance six as representative of major units and two as special samples. (2) Some trace elements such as Sr or Ni which are easily

measured using pellets could be determined. (3) Some mineral determination could be made.

These additions compared to Leg 37 would significantly increase the scientific capabilities of a leg and would provide guidance for sampling for more detailed land-based studies.

PRELIMINARY RESULTS OBTAINED ONBOARD

Preliminary data obtained onboard including volatiles measured by Craig Dootson along with Na_2O , MnO , and P_2O_5 determined on shore are presented hole by hole. The data for four standards selected onboard are presented in Wright (this volume).

Hole 332A

Results are presented in Table 8 of Chapter 2 (this volume). All measured elements were plotted as a function of depth, and it was shown onboard that it was possible to distinguish several major chemical units; to clarify the figure, only three elements have been plotted in Figure 8 (MgO , Al_2O_3 , and TiO_2). The major units are: (a) 100-120 meters, Cores 6 and 7; (b) 120-150 meters, Cores 8-10; (c) 150-170 meters, Cores 11 and 12; (d) 170-250 meters, Cores 13-21; (e) 250-320 meters, Cores 22-28; (f) 320-430 meters, Cores 29-30; (g) 430-440 meters, Core 40.

In some units some analyzed samples do not fit in the major unit. This is the case for instance in Units e and f: Sample 28-1 (#3) mainly differs from Unit e by Fe_2O_3 , CaO , Al_2O_3 , and TiO_2 , but fits well in the following chemical Unit f.

The mean compositions of chemical units for Hole 332A are presented in Table 4. In most of the units, TiO_2 is constant or increases slightly from the bottom to the top of the layer: K_2O generally increases from the bottom to the top from 0.2% to 0.4%; in Units c and d, MgO decreases from the bottom to the top of the layer. Compared to the other units, the high alumina basalts of Unit a and the olivine-bearing basalts of Unit g are markedly depleted in TiO_2 .

Most of the boundaries between the different units correlate with possible long periods of noneruption as indicated by the presence of baked and lithified sediment, abundant calcite veins, or breccia.

Comparing these chemical units with the lithological units, it is possible to relate them as follows:

Chemical Unit	Core	Lithologic Unit	Basalt Type
a	6-7	I	Plagioclase phyric basalt
b	down to Core 10	upper part II	
c	11-12	II	Sparsely phyric basalt
d	13-21	end of II and IIA	
e	22-28	IIIB and IV	Plagioclase phyric basalt
f	29-39	V	Plagioclase phyric basalt
g	40	VI	Highly phyric basalt (plagioclase augite olivine)

There is a good correlation between the chemical and lithological Units a and I corresponding to the plagioclase phyric basalts, Units f and VI which are also a plagioclase phyric unit but with a lesser concentration of plagioclase than in Unit I, and Units g and VI, the highly plagioclase-augite-olivine phyric basalt unit.

A discrepancy exists between the b, c, d, e chemical units and the III, IV, and V lithological units corresponding to sparsely phyric or aphyric units. Assuming the validity of the chemical units, this discrepancy probably reflects the difficulty of establishing precise lithologic boundaries within sparsely phyric or aphyric units. Nevertheless, lithological Unit III is divided into two parts, IIIA and IIIB, separated by a sequence of lithified sediment; this separation corresponds to the transition d-e chemical units.

In Figure 8, magnetic polarities are indicated by the letters N, R, or Sh which mean Normal, Reverse, or Shallow (data of Hall and Ryall, this volume) without distinguishing between Normal Shallow or Reverse Shallow. Rocks are normally magnetized down to Core 13 and then shallow or reversely magnetized to the base of the hole. As a comparison, the best match between chemical boundaries and magnetic boundaries is between Cores 12 and 13 which corresponds to the transition c-d units and N to shallow magnetics.

In conclusion, six major chemical units can be defined in Hole 332A; the boundaries between these units correspond to the lithological boundaries except in the aphyric or sparsely phyric units. For the porphyritic basalts the chemical compositions closely reflect the mineralogical compositions, e.g., the plagioclase phyric basalts of Units I and V have $\text{Al}_2\text{O}_3 > 20\%$ and $\sim 17\%$, respectively, and the plagioclase-augite-olivine phyric basalt of Unit g has a higher concentration of MgO (9%-11%) than other analyzed basalts (up to 8%). The differences between other units are not so large.

Hole 332B

The chemical analyses of Hole 332B (52 samples) are presented in Table 8 of Chapter 2 (this volume) in the same way as for Hole 332A. Concentrations of three elements (MgO , Al_2O_3 , and TiO_2) are plotted as a function of depth in Figure 9. It is possible to distinguish 11 chemical units on the basis of these chemical data, as follows.

	Depth (m)	Cores
a	160-210	2-3 (upper part)
b	210-260	3-4 (lower part)
c	280-300	6
d	330-410	8-15
e	410-470	16-22
f	470-500	23-25
g	500-585	26-34
h	585-595	35
i	595-615	36-37
j	615-635	37-39
k	635-720	39-48

In each of these units, some anomalous samples are present. This is the case, for instance, with the samples of Core 10 and Core 33, Section 2, which have anomalously high CaO and of some high- MgO olivine basalts occurring as thin layers in these units.

Table 5 gives the mean composition of the chemical units; unit k is not given because of variability in this unit.

To complete this description, olivine basalts occurring in different units, mostly in the lower part of the hole, must be added: they are characterized by high

MgO value (>9%, up to 13%), by low TiO₂ values (~0.6%), and generally low K₂O values (~0.1%).

Some boundaries between these chemical units are marked by the presence of lithified sediment or breccia or calcareous veins, but this relationship is not as consistent as for Hole 332A.

The correlation between chemical and lithological units is as follows.

Chemical Unit	Core	Lithological Unit	Basalt Type
a	1-5 to 3-4	I	Plagioclase phyric
b	3-4 to 4	upper part of II	Aphyric
c	6	lower part of II	Sparsely plagioclase phyric
d	8 to 15	III	Plagioclase phyric
e	16 to 22	IV	Olivine phyric to highly olivine phyric
f	22 to 25	V	Plagioclase-olivine glomerophyric
g	25 to 34	VI	Some olivine and plagioclase phenocrysts
h	35-1 to 35-3	VII	Plagioclase phyric
i	35-3 to 37-2	VIII	Plagioclase phyric
j	37-2 to 29	IX	Olivine basalts
k	39 to 48	X and XI	Plagioclase-phyric basalts some olivine basalts

There is a good correlation between chemical units and lithological units. Lithological Unit II is divided into b and c chemical units mainly on the basis of Fe₂O₃ values: as only two samples define Unit b, mean values in Table 5 are given for Units b and c together.

Down to Core 39, each chemical unit corresponds to a lithological unit. From Core 39 down to the last core there are two lithological units (X and XI), but only one chemical unit is defined because the values are scattered and the only sample analyzed in Unit XI is not markedly different from Unit X.

The chemical compositions agree well with the lithological description: a good example is given by Unit e, defined as an olivine settling unit (IV); in this unit, magnesium increases markedly from the top to the bottom of the unit, from 11% to 15%, as Al₂O₃ decreases from 14.7% to 13.4%. A remarkable point is the concentration of MgO at 24% for a picrite of Core 35.

Magnetic polarity boundaries based on N, R, Sh fit well with chemical boundaries for a-b, b-c, c-d, d-e, and e-f boundaries. In the lower units, which are magnetically complicated but indicated as "mainly shallow," it should be noted that olivine basalts encountered in these series are reversely magnetized.

Correlation of Holes 332A and 332B

Without doubt, Holes 332B (Unit d) and 332A (Unit f) correlate well, both by comparing Figures 8 and 9 or by mean values shown in Tables 4 and 5; these layers are useful as a reference for further comparison of Holes 332A with 332B. The thickness from the first basalt recovered down to the bottom of 332A (Unit f) and of 332B (Unit d) are 325 and 250 meters, respectively.

The corresponding sequences mentioned above are 100 meters (Hole 332A) and 85 meters (Hole 332B)

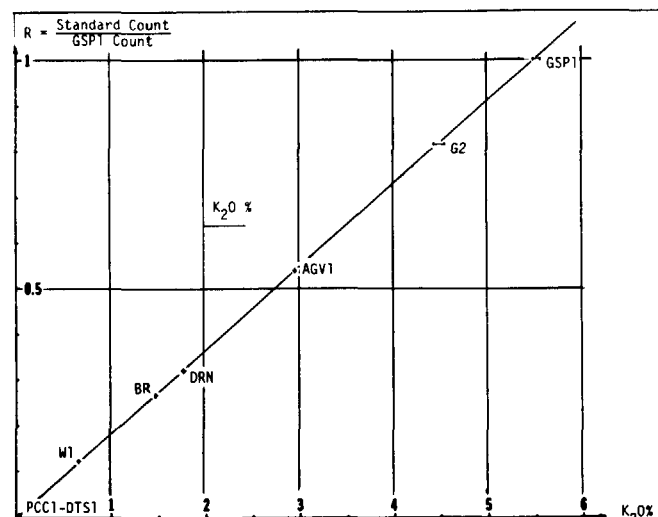
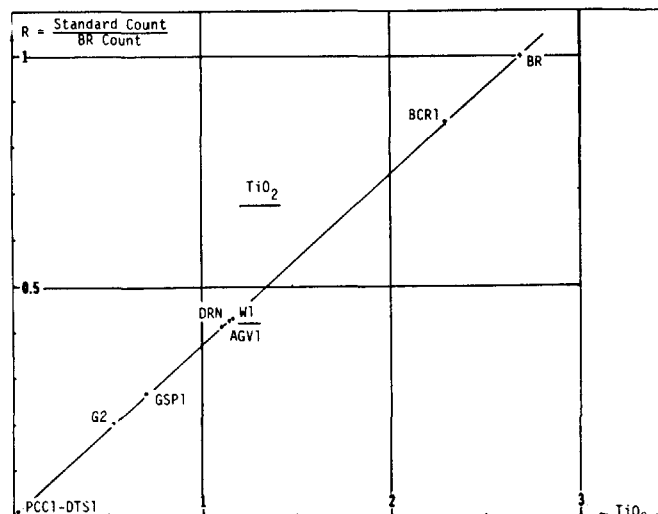
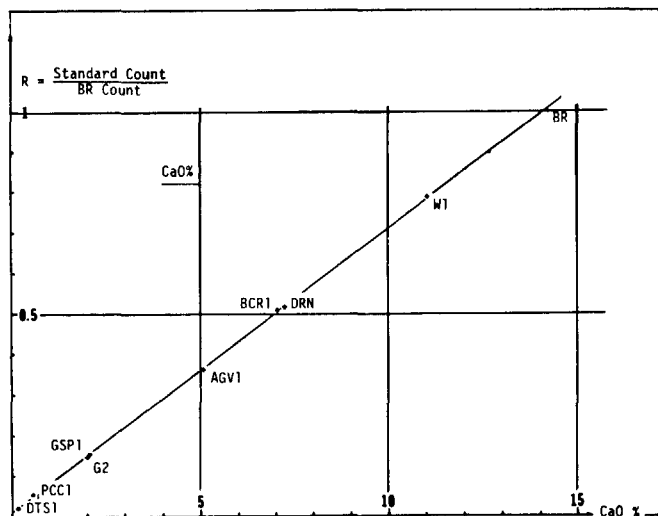


Figure 7. Calibration curves obtained onboard; the intensity is normalized to 1 for the most concentrated standard.

thick, respectively; 332A (Unit e) and 332B (Unit b or c) are also corresponding sequences in Holes 332A and

TABLE 1
Comparison of Onboard Determinations with Mean Values of
Analyses of Four Different Laboratories

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	K ₂ O	TiO ₂	P ₂ O ₅
Mean values (4 labs)	50.0	14.55	10.38	9.1	11.34	0.24	1.16	0.12
	±0.5	±0.20	±0.2	±0.2	±0.2	±0.04	±0.06	±0.5
Onboard determinations	49.7	14.5	10.4	9.0	11.3	0.26	1.18	0.15

TABLE 2
Stability of the XRF Unit Onboard:
Results of Measurements Made Different Days

	CaO		TiO ₂		K ₂ O	
	1st Value	2nd Value	1st Value	2nd Value	1st Value	2nd Value
7-2, 5-10	11.8	11.8	1.19	1.19	0.41	0.42
40-3, 90-93	13.95	14.07	0.53	0.53	0.11	0.11
12-2, 60-65	13.57	13.43	0.92	0.92	0.20	0.19
40-2 (#8)	11.86	11.97	1.22	1.23	0.3	0.3
22-1 (#16)	12.3	12.29	1.24	1.24	0.41	0.41
31-1 (#3)	12.81	12.79	1.03	1.02	0.36	0.37
20-2 (#7)	12.65	12.65	1.05	1.06	0.20	0.20

332B (close chemistry and similar magnetics). The corresponding sequences in Hole 332B are thinner than those of Hole 332A.

Looking at the top of these holes, the plagioclase pyritic basalts have somewhat different compositions (particularly in MgO content) and different magnetic polarities (N in 332A and R in 332B).

The proposed correlations are summarized in Figure 10.

Hole 333A

Table 10 of Chapter 3 (this volume) presents the 14 shipboard chemical analyses available for this hole. No

TABLE 3
Mean Differences (%) Between Shipboard Determinations and AUF Determinations
Compared with Mean Differences Between Two Land-based Laboratories and
2σ Calculated from Determinations of the Same Sample by Four Laboratories

	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO ^a	MgO	CaO	K ₂ O
Hole 332A ^b 24 samples ^c	0.39	0.10	0.51	0.19	0.01	0.24	0.17	0.01
Hole 332B 24 samples	0.61	0.046	0.44	0.29	0.006	0.14	0.14	0.011
Site 334 5 samples MUN or ML with AUF	0.45	0.02	0.39	0.42	<0.01	0.37	0.30	<0.01
8 samples 2σ from four determinations of same sample (shipboard report)	0.60	0.055	0.50	0.28	0.007	0.28	0.19	0.04
	0.50	0.06	0.20	0.20		0.20	0.20	0.04

^aXRF land determination.

^bNot taking into account 5 samples with totals below 98%

^cTaking into account all 24 samples.

TABLE 4
Mean Values for the Different Chemical Units, Hole 332A

	a	b	c	d	e	f	g
SiO ₂	47.31	49.72	49.22	49.62	48.98	49.09	47.61
Al ₂ O ₃	22.03	14.08	14.57	14.48	14.56	15.65	15.73
Fe ₂ O ₃	5.43	11.00	9.50	10.12	10.89	9.88	7.00
MgO	5.61	6.79	8.14	7.57	7.28	7.14	9.97
CaO	15.67	11.86	12.97	12.38	12.20	12.70	14.49
TiO ₂	0.38	1.14	0.91	1.06	1.19	1.06	0.54
K ₂ O	0.10	0.36	0.17	0.25	0.30	0.24	0.15

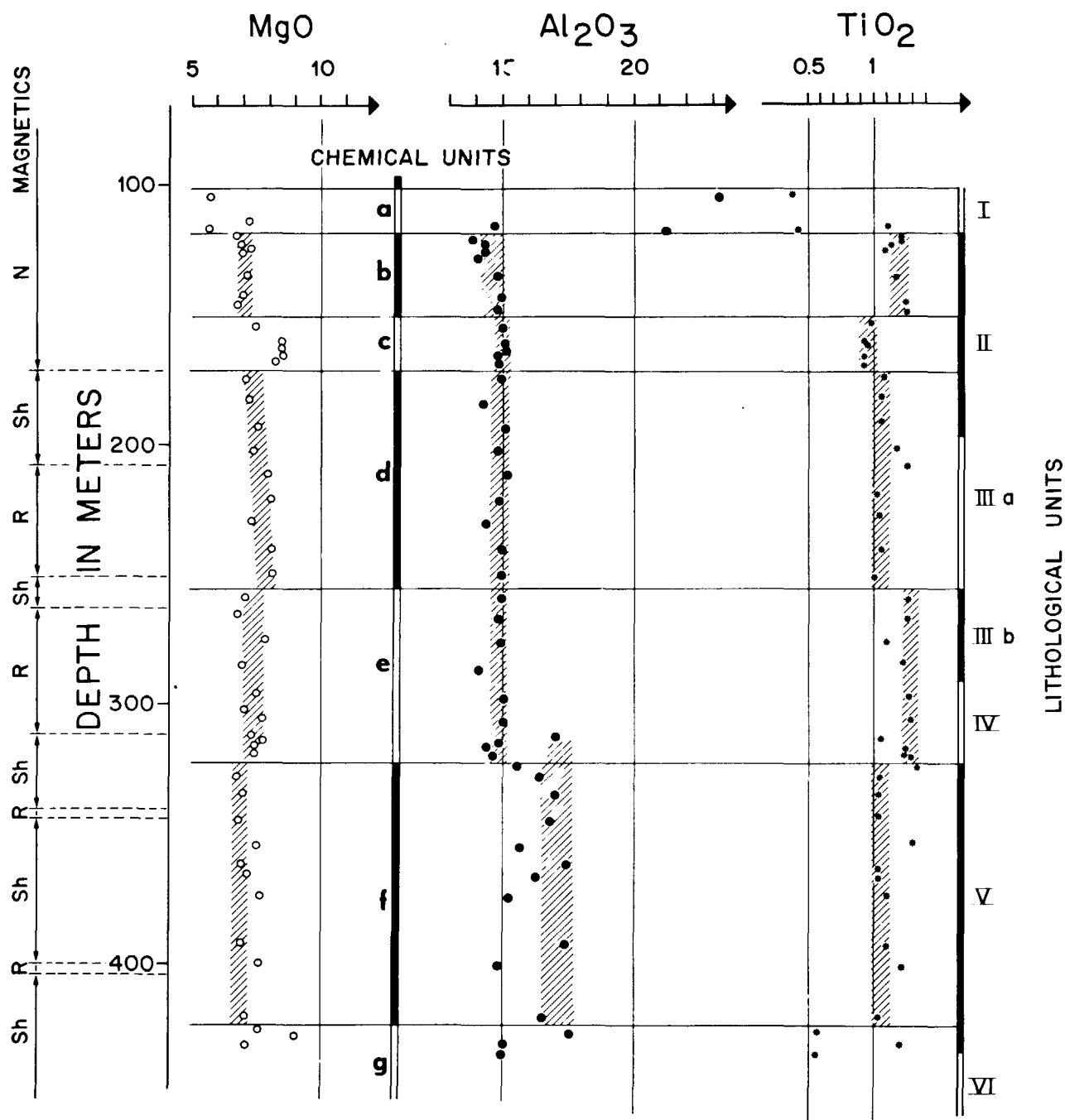


Figure 8. MgO , Al_2O_3 and TiO_2 plotted versus depth and chemical units, Hole 332A.

figure of elements concentrations as a function of depth is presented as is the case of Holes 332A and 332B because no trends could be determined. This may be the result of an insufficient number of analyzed samples, but could also be due to the poor recovery and the variety of rocks encountered. Nevertheless, for each sample, the chemical analysis obtained is consistent with the lithologic description as, for instance, Sample 1-3, (#5B) olivine basalt, 3-1 (#7A) plagioclase pyric basalt ($MgO = 10\%$ and $Al_2O_3 = 17\%$, respectively) or 6-1 (#2) sparsely plagioclase-pyroxene pyric basalt and 6-2 (#6) plagioclase and olivine pyric, with Al_2O_3 14.8% and 16.35%, MgO 8.1% and 8.9%, respectively.

Chemical results obtained confirm the complexity of this hole as indicated by the poor recovery and the lithological description. This complexity is due to the abundance of breccia at this site which may in part reflect accumulation of rubble along a fault scarp.

Site 334

Chemical analyses of Site 334 samples are presented in Table of Chapter 4 (this volume). In the upper basaltic layer, the results clearly show two basaltic units, the boundary occurring in Core 16, Section 4. A break is observed for most elements (Figure 11). These two layers are defined as sparsely pyric basalts with

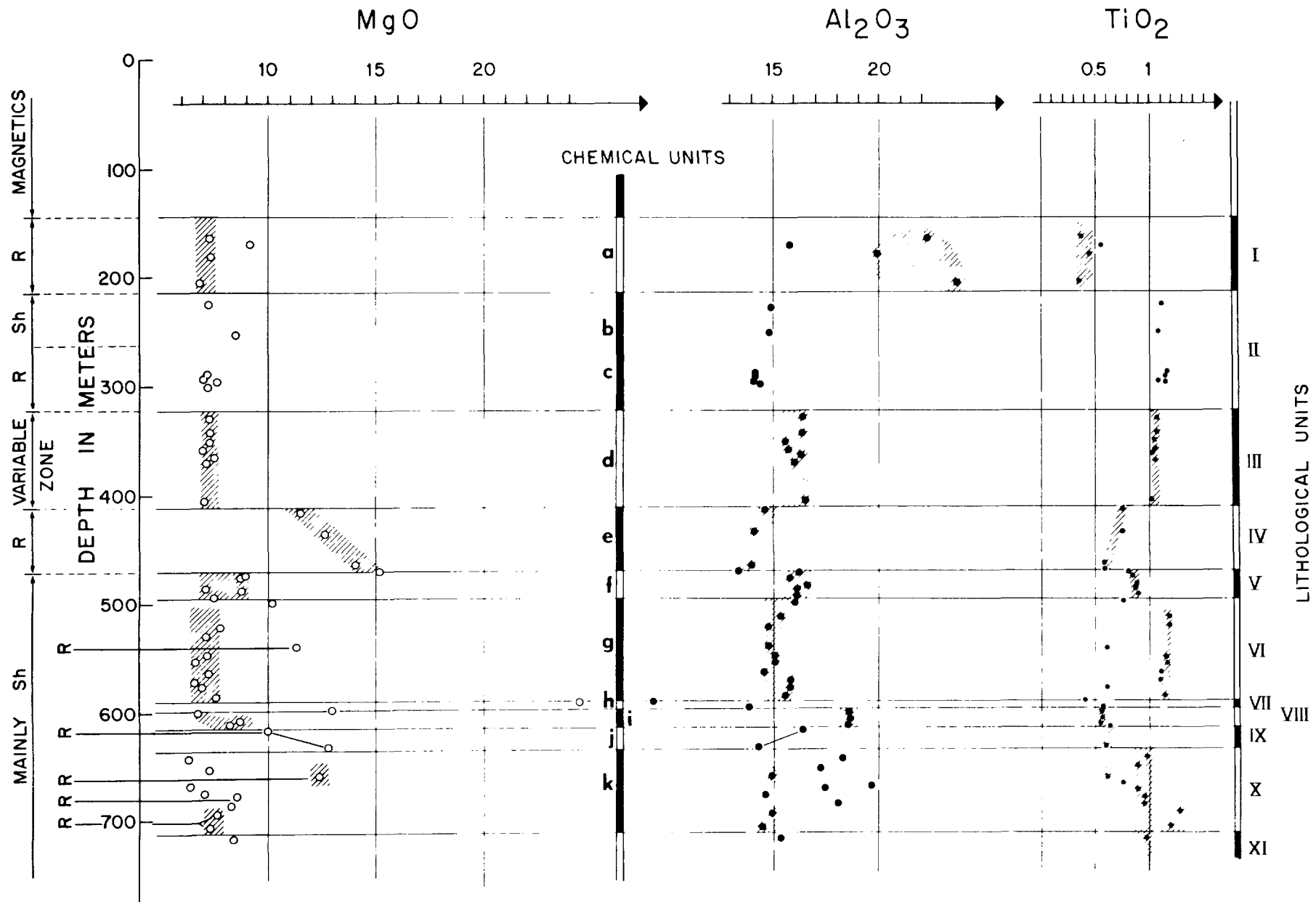


Figure 9. MgO, Al₂O₃ and TiO₂ plotted versus depth and chemical units, Hole 332B.

TABLE 5
Mean Values for the Different Chemical Units, Hole 332B

	a	b,c	d	e	f	g	h	i	j
SiO ₂	47.81	49.16	48.76	46.10	47.32	48.00	43.63	45.47	46.29
Al ₂ O ₃	21.47	14.16	15.84	13.65	15.80	14.71	11.17	17.74	14.89
Fe ₂ O ₃	5.81	10.84	9.51	10.32	10.53	10.44	10.32	7.78	10.15
MgO	7.00	7.30	7.06	12.94	8.00	6.93	18.07	7.58	11.10
CaO	15.05	12.02	13.13	10.94	11.89	12.15	9.33	13.52	11.46
TiO ₂	0.37	1.11	1.03	0.65	0.84	1.11	0.47	0.53	0.60
K ₂ O	0.07	0.32	0.26	0.16	0.16	0.32	0.17	0.11	0.07

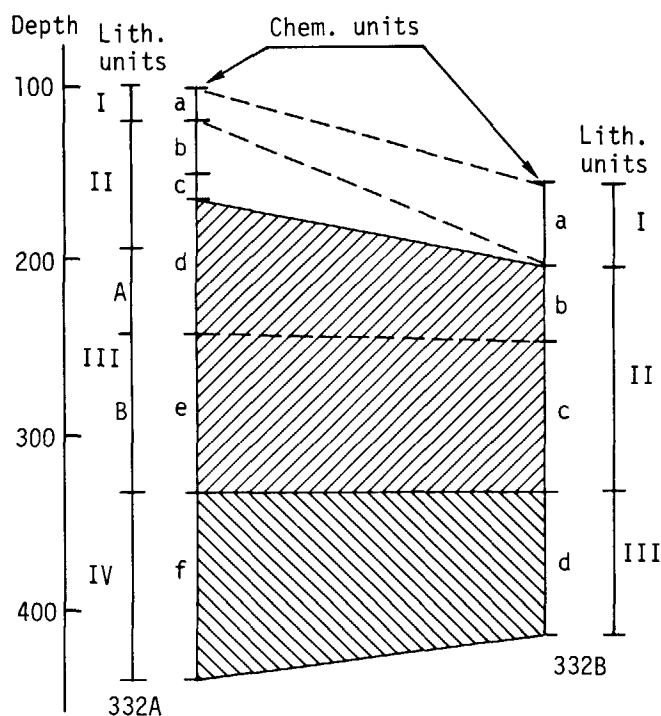


Figure 10. Stratigraphic correlation between Holes 332A and 332B from chemical data.

5%-10% of phenocrysts (plagioclase, augite, and olivine) and aphyric, olivine-bearing basalts. MgO increases from 7.6% at the top of the first layer to 9.1% at the bottom of the second layer. The mean values for the different chemical units are listed in Table 6.

In the lower mafic to ultramafic layer the composition of gabbros 23-1, 127-129 cm and 24-3, 112-114 cm reflects the composition of common gabbro and olivine gabbro. These samples are very fresh as indicated by their volatile content (0.69% in Sample 23-1, 127-129 cm). The serpentinized peridotites are plagioclase-bearing peridotites as reflected by their Al₂O₃ and CaO content (5.14% and 3.39%, respectively, in Sample 26-1, 20-22 cm).

Site 335

Chemical results obtained do not show major differences within this site (Table 2 of Chapter 5 [this volume]). Mean values of the following elements are

SiO₂: 46.88; Al₂O₃: 15.35; Fe₂O₃: 10.18; K₂O: 0.3; TiO₂: 1.16. MgO decreases from 8.3 to 6.4% from Core 5, Section 3 to Core 13, Section 3, with a value of 8.8% in the lowest analyzed sample 16-1 (# 3). High CaO and CO₂ values are present in 11-1 (#1) and 13-3 (#1B) corresponding to the presence of carbonate veins.

CONCLUSIONS

The X-ray fluorescence unit operated without any problem throughout the cruise; the number of analyzed samples (129) and the precision of determination compared to on-shore data now available demonstrate the feasibility of shipboard chemical analysis.

Chemical analyses made onboard delineate chemical units for Holes 332A and 332B and Site 334; these chemical units are in agreement with lithologic descriptions. Hole 333A chemical data are scattered and perhaps reflect accumulation of rubble along a fault scarp. Site 335 data seem to be homogeneous.

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TABLE 6
Mean Values for the Different Chemical Units, Site 334

	a	b
SiO ₂	49.70	49.42
Al ₂ O ₃	15.52	14.69
Fe ₂ O ₃	10.07	9.99
MgO	7.65	8.57
CaO	12.44	13.08
TiO ₂	0.86	0.73
K ₂ O	0.28	0.17

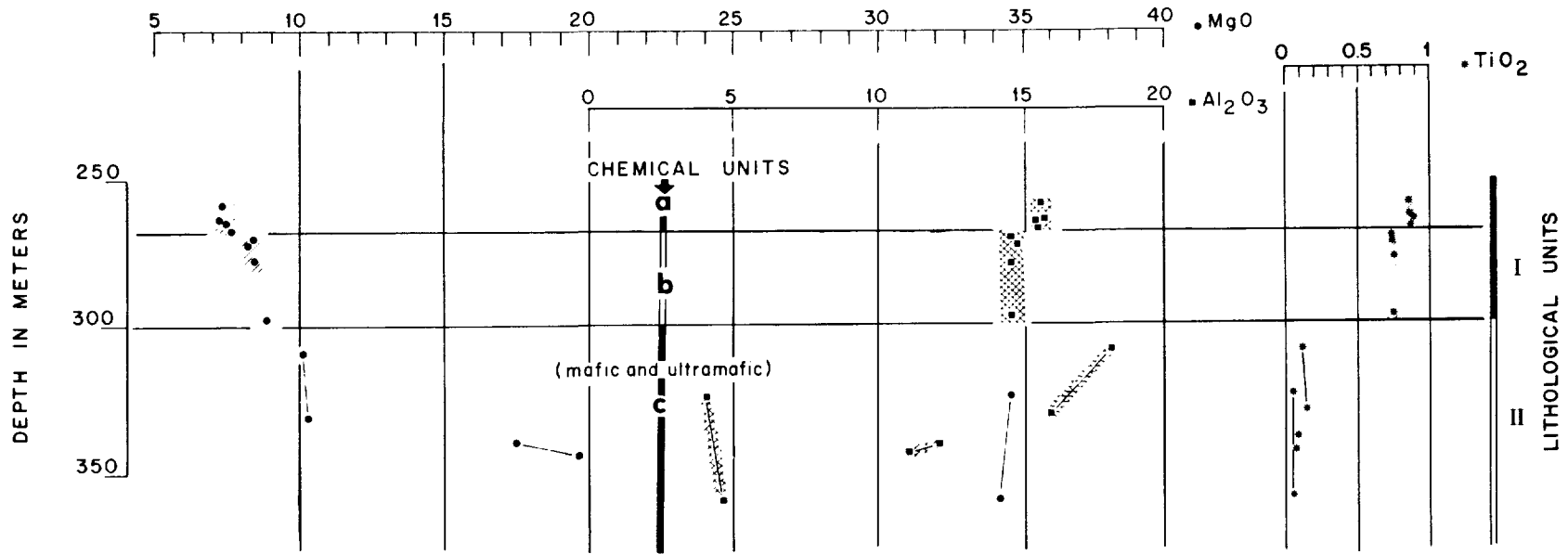


Figure 11. *MgO*, *Al*₂*O*₃ and *TiO*₂ plotted versus depth and chemical units, Site 334.