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GEOCHEMICAL DATA FOR JURASSIC DIABASE ASSOCIATED WITH EARLY
MESOZOIC BASINS IN THE EASTERN UNITED STATES: GEOLOGIC
SETTING, OVERVIEW, AND CHEMICAL METHODS USED

By

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I. GEOLOGIC SETTING AND OVERVIEW

Introduction

At least four major quartz-normative and a minimum of two olivine-normative magma types have been defined among the continental tholeiites from Massachusetts to North Carolina. Analyses of basaltic lava flows and chilled margins of intrusive diabase sheets and dikes associated with rift-related early Mesozoic sedimentary basins are the basis for the definitions of magma types. The study of these magma types has been evolving over the past two decades through the work of many petrologists (Weigand and Ragland, 1970; Smith and others, 1975; Ragland and Whittington, 1983; Puffer and Philpotts, 1988; Tollo and Gottfried, 1989). In South Carolina and Georgia, south of the area covered by this suite of reports, other olivine-normative magma types have been defined in early Mesozoic dikes (Georgia: Gottfried and Arth, 1985; South Carolina: Warner and others, 1985).

Diabase dikes that strike north, northeast, and northwest cut strata of the early Mesozoic basins and intrude the adjacent Piedmont crystalline and folded Paleozoic terranes. Regardless of strike, all principal magma types are represented, although quartz-normative dikes are more common northward from central Virginia, and olivine-normative dikes are more abundant to the south. Similarly, olivine-normative diabase sheets are exposed in North Carolina, whereas quartz-normative sheets are widespread from Connecticut to central Virginia.

Table 1 summarizes the ranges of selected major elements and some trace element average ratios that have been used to help define the principal magma types. As no single ratio or range in elements is totally effective in defining magma types, major element and several element ratios should be used together (see Table 1). As not all magma types present are documented in this report, we have used the following magma type terminology: HTQ = High Ti, quartz normative; HFQ = High Fe, quartz normative; LTQ = Low Ti, quartz normative; ON = Olivine normative.

From 1984 to 1990, the U.S. Geological Survey carried out a field geochemical and petrologic assessment of early Jurassic diabase intrusives associated with the exposed early Mesozoic basins. Major, trace and platinum group elements were analyzed for more than 960 samples as part of the Survey's Strategic and Critical Minerals Program. Although the results of some of the studies based on these analyses have been published (Froelich and Gottfried, 1988; Gottfried and Froelich, 1988; Gottfried and others, 1990), the complete geochemical data base has never before been provided. Included in this study are powders of diabase samples from local studies by both USGS and non-USGS workers, documented elsewhere, that were analyzed for selected elements by the USGS in the course of these studies. Samples generally labeled FG- in the tables, but not labeled on the map, were collected by Froelich and Gottfried of the USGS in the course of this regional study. Samples collected by others are identified, described, and referenced in the brief text accompanying the map (scale 1:125,000) of each of the eight basin areas documented in this study. A very brief discussion of the local PGE distribution in the diabase is also included with these texts.

Of the principal magma types recognized in the Mesozoic tholeiitic province of the eastern United States, the high Ti quartz-normative (HTQ) variety and some of the olivine normative (ON) types have similar original abundances and ratios of Pd and Pt; about 10 parts per billion (ppb) each in chilled margin samples throughout the province. The other quartz-normative

varieties, especially those that formed sheets (LTQ and HFQ), have Pd and Pt abundances commonly less than 5 ppm in the chilled margins and nowhere show significant enrichment. Both ON and HTQ cumulate zones enriched in olivine or orthopyroxene show slight enrichment of Pt (30 to 50 ppb) and depletion of Pd (less than 5 ppm). In contrast, late stage ferrogabbro, ferrodiorite, and granophyre differentiates are significantly enriched in Pd (100-500 ppb) in at least four of the HTQ sheets and one ON sheet. As the enriched zones are relatively thin (tens of meters), it is likely that many such zones in other thick HTQ sheets were not sampled in the course of this reconnaissance study.

Table 1. Classification of magma types of early Mesozoic diabases and basalts based on characteristic major element abundances and selected trace-element ratios (modified from Tollo and Gottfried, 1989, and published and unpublished U.S. Geological Survey data; quartz-normative data are based on more than 200 analyses).

Magma type	Major elements Weight percent, (Average basalts and diabases)				Trace element ratios (Average basalts)			
	SiO_2	MgO	TiO_2	Fe_2O_3T	100 Nb/Ti	Th/Hf	Hf/Ta	La/Yb(n)
Quartz normative								
HTQ (High Ti)	50-53	7-8	1.0-1.2	<13	0.10	0.85	4.7	3.2
LTQ (Low Ti)	49.5-52	7.5-8.5	.7-.85	<13	.07	.90	6.0	1.7
HFQ (High Fe)	51-54	5.2-6	1.0-1.2	12.5-15	.07	.94	6.6	2.1
HFTQ (High Fe-Ti)	51-54	4.5-6.0	1.3-1.5	>15	.05	.76	8.0	1.4
Olivine normative	(Average dikes and sheets)				(Average dikes and sheets)			
ONA	47-49	8-15	.25-.45	<13	.12	1.2	5.0	1.8
ONB	47-50	7-24	.35-.8	<13	.07	.35	7.0	1.35

Fe_2O_3T = total iron ($FeO+Fe_2O_3$). La/Yb(n) = ratio normalized to chondrites. < = less than; > = more than.

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II. CHEMICAL METHODS USED IN THE ANALYSIS OF DIABASE ASSOCIATED WITH EARLY MESOZOIC BASINS, EASTERN UNITED STATES

Introduction

Chemical analyses listed in this report were done in the Reston, Virginia and Denver, Colorado laboratories of the U.S. Geological Survey over a period of eight years (1984 to 1991). All of the chemical procedures that were used are listed below. Because methodology and data requirements varied over time, many elements were determined by more than one analytical procedure. For some cases, ranges of laboratory numbers, corresponding to the numbers in the data tables, are listed next to the name of a method description, indicating which samples were analyzed by which methods. However, data for some elements are primarily taken from a "preferred" method, with missing values filled in with data from secondary methods. In these cases, it is impractical to specify which samples were analyzed by which methods. All intermethod biases are believed to be less than 10%.

For elements where ranges of laboratory numbers are not specified, the orders of preference for analytical methods are as follows (abbreviations: flame atomic absorption spectrometry (FAA), graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma atomic emission spectrometry (ICP-AES), instrumental neutron activation analysis (INAA), wavelength dispersive X-ray fluorescence spectrometry (WDXRF), and energy dispersive X-ray fluorescence spectrometry (EDXRF)):

Na_2O: 1) INAA, 2) "Rapid rock" (see description below) and WDXRF, 3) ICP-AES
CaO: 1) "Rapid rock" and WDXRF, 2) INAA
Cr: 1) INAA, 2) GFAA, 3) ICP-AES
Fe_2O_3 (total): 1) INAA, 2) "Rapid rock" and WDXRF, 3) ICP-AES
Co: 1) INAA, 2) FAA, 3) ICP-AES
Ni: 1) ICP-AES, 2) FAA, 3) EDXRF
Cu: 1) EDXRF, 2) FAA
Zn: 1) EDXRF, 2) INAA, 3) ICP-AES
Rb: 1) EDXRF, 2) INAA
Sr: 1) EDXRF, 2) ICP-AES
Y: 1) EDXRF, 2) ICP-AES
Ba: 1) EDXRF, 2) INAA, 3) ICP-AES

Data Availability

Digital versions of the data tabulated in this report are available on double-sided high-density (1.2 MB) $5\frac{1}{4}$" floppy diskettes (Grossman *et al.*, 1991a) and double-sided high-density (1.44 MB) $3\frac{1}{2}$" diskettes (Grossman *et al.*, 1991b). These data are in a form compatible with Lotus 1-2-3 Release 2 or its equivalent on IBM-PC-type computers.

Sampling.

Representative samples of fresh core, quarry, or bedrock outcrops of at least 2 kg were collected, labeled, megascopically identified, and located on 1:24,000 topographic quadrangles by latitude and longitude. Where possible, weathered rinds, veins, and obviously altered material were removed and discarded. In the laboratory, the rock samples were re-examined to assure that all undesireable portions were removed; if any extraneous material was present, it was cut away with a diamond saw and discarded. In general, 2-3 cm-wide, square thin-section blanks were cut from representative specimens.

Grinding.

Large rock fragments (>5 cm) are crushed to ~8 mesh (2 to 3 mm) in a jaw crusher with adjustable, hardened Mn-steel plates. The entire sample is run through a "vertical" grinder with alumina plates, producing a powder of ~100 mesh (i.e., most particles are <150 μm). Grinding equipment is cleaned before each sample by processing quartz-sand.

Splitting.

To prepare aliquants for use in different laboratories, the rock powder is thoroughly mixed, poured onto glassine paper, and divided into four piles. Small portions are taken from throughout each mound with a spatula, and placed in a new bottle. For large samples, cone splitters or riffle splitters are used to obtain aliquants.

Analysis of Major Element Oxides

"Rapid Rock" method (Lab numbers < W-238427)

Following the method of Shapiro *et al.* (1975), 200 mg of samples are fused with a lithium meta-borate/tetra-borate flux. The fused sample is dissolved in dilute HNO_3. Silicon, Al, P, and Ti are determined colorimetrically. Total Fe, Ca, K, Mg, Mn, and Na are determined by FAA. Fe_2O_3 is calculated by difference from total Fe and FeO (measured by the method listed below). All data are reported as oxides.

Wavelength-dispersive XRF (Lab numbers $\geq$ W-238427)

Major element oxides are determined in rocks and minerals by WDXRF (Taggart *et al.*, 1987). 800-mg samples are weighed and then ignited in a Pt-Au crucible at 900 to 925°C for 45 minutes. After cooling, the samples are reweighed to determine the total loss on ignition (LOI). The ignited samples are fused with 8 g of lithium tetraborate by heating at 1120°C for 40 minutes, poured into molds, and the resultant glass disc is irradiated by X-rays generated by a Rh-target tube operating at 35-kV and a current of 60 mA. Characteristic X-rays emitted by each element in the sample are counted, corrected for matrix effects using the deJongh (1973) model, and concentrations are determined using previously prepared calibration standards. Concentration data are then recalculated to account for any mass change on ignition.

FeO (Peck, 1964).

A 500-mg sample is decomposed using HF and H_2SO_4. This solution is treated with boric, sulfuric, and phosphoric acids. Fe (II) is determined by a colorimetric or a potentiometric titration with potassium dichromate. Sodium diphenylamine sulfonate is used as the endpoint indicator in the colorimetric titration.

CO_2 (Engleman *et al.*, 1985).

A 500-mg sample is digested with $HClO_4$. CO_2 is evolved and carried into a coulometric cell. The CO_2 is converted into a strong acid by ethanolamine, and is titrated coulometrically.

H_2O^- (Shapiro, 1975).

A 1-g sample is weighed and dried at 110°C for a minimum of 1 hr. After cooling in a dessicator, the sample is weighed again and H_2O^- is calculated by difference.

H_2O^+.

Method of Shapiro (1975) A 1-gram sample is fused with 2 grams of anhydrous sodium tungstate. Water in the sample is driven off and collected on a preweighed filter paper. Total water is determined by reweighing the filter paper. H_2O^+ is calculated as the difference of total H_2O and previously determined H_2O^-.

Method of Jackson et al.(1987) (Lab numbers W241388-828, W243816-838). A 50-mg sample is mixed with 150 mg of lead oxide/lead chromate flux. The sample is heated to 950°C. The evolved water is determined coulometrically by Karl-Fischer titration. This gives the total H_2O in the sample. H_2O^+ is determined from the difference between total H_2O and H_2O^-.

Analysis of Sulfur

A 200-mg sample is weighed. Vanadium pentoxide is added as a combustion aid. The sample is combusted in a sulfur analyzer and the sulfur dioxide evolved is measured by an IR detector (Kirschenbaum, 1983).

Analysis of Chlorine

A 200-mg sample is decomposed using HF, H_2SO_4, and $KMnO_4$ in a specially designed, sealed teflon container. Chlorine is captured in a KOH/Na_2SO_3 solution in a center compartment of the container. Chlorine is determined as chloride by the selective ion electrode (SIE) method of Aruscavage and Campbell (1983).

Analysis of Fluorine

Method of Kirschenbaum (1988).

A 100-mg sample is fused with a Na_2CO_3/ZnO flux. The fusion cake is leached with H_2O. HCl is added to expel CO_2. An aliquot of the sample solution is buffered with a sodium citrate/KNO_3 solution. This solution is analyzed for fluorine, as fluoride, by the selective ion electrode (SIE) method of Kirschenbaum (1988).

Method of Hopkins (1977) (Lab numbers W241388–828, W243816–838).

A 50-mg sample is fused with a $Na_2CO_3/K_2CO_3/KNO_3$ flux. The fusion cake dissolved in 1M citric acid. The sample solution is buffered with sodium citrate. This solution is analyzed for fluorine, as fluoride, by a standard addition method using the SIE method of Hopkins (1977).

Analysis of Platinum Group Elements

Fire Assay/GFAA method.

A 15-gram sample and 2 mg of gold wire are fused with a $PbO/Na_2O_3/Borax$-glass/flour flux for 45 min. at 1000°C. The lead button is separated from the fusion slag. Bone-ash cupels are preheated at 1000°C, and the lead buttons are added. After the buttons have melted the temperature is reduced to 850°C, and air is passed over the cupels. The lead is oxidized to PbO, and is absorbed into the bone ash, leaving a gold bead which is dissolved in aqua regia and evaporated to dryness. The residue is taken up in 6M HCl, and analyzed for Pd, Pt and Rh by GFAA (Aruscavage *et al.*, 1984).

The accuracy and precision of Pd and Pt analyses by this method was monitored by including replicate samples of USGS standard rocks DNC-1, W-2, and BHVO-1 (see Gottfried and Froelich, 1988, for a full discussion).

Nickel sulfide/ICP-MS method (Lab numbers W241388–828, W243816–838).

A 10-g sample is fused with a $Ni/S/SiO_2/Na_3CO_2/borax$-glass flux for 2 hours at 1050°C. The resulting NiS button is removed, broken into pieces, and placed into a test tube. 1 mL of 20% $SnCl_2$, 0.1 mL of 1% tellurium solution, and 50-70 mL of concentrated HCl are added to the tube to dissolve the NiS button. The solution is filtered and the residue is collected on polycarbonate filter paper. The filter paper is dissolved in HCl and HNO_3, diluted to 10 mL with 1% HCl and the solution is analyzed for Pd, Pt, Rh, Ru and Ir by inductively coupled plasma mass spectrometry (ICP-MS).

Analysis of Ni, Cu, Zn, Rb, Sr, Y, Zr and Ba by Energy-Dispersive XRF

Approximately 1.0 g of 100-mesh, powdered sample is pressed into a Mylar cup. Samples are analyzed for Ni, Cu, Zn, Rb, Sr, Y, Zr and Ba using a Kevex 700 EDXRF spectrometer with a Kevex 7000 analyzer (Johnson, 1984; Johnson and King, 1987). The secondary targets used to fluoresce each element were:

    Ni, Cu and Zn : Germanium
    Rb, Sr, Y and Zr : Silver
    Ba : Gadolinium

Corrections are made for background interferences, escape peaks, and spectral overlaps. Sources of error inherent to EDXRF analysis are corrected using the Compton ratio method. Trace element concentrations in the samples are calculated from calibration graphs of the intensity ratio vs. concentration for a series of standard reference materials found in Abbey (1983).

Multielement Neutron Activation Analysis

Sample aliquants of ~0.5 g each are irradiated for 6-8 hours at a flux of ~2×10^{12} n-cm^{-2}-s^{-1} in the "TRIGA" reactor at the U.S. Geological Survey, Denver, CO. Standards for most elements are aliquants of a powdered natural obsidian spiked with primary

solutions, taken to dryness and homogenized. Standards for Ca, Ti and Au are powdered, $CaCO_3$, TiO_2, and homogeneous low-Au quartz, respectively. At least one replicate sample plus USGS standard rocks DNC-1 and W-2 were irradiated together with the samples and standards.

Samples are counted three times on co-axial Ge and/or Ge(Li) detectors with resolutions ranging from 1.78 to 1.86 KeV measured at 1.33 MeV using the following scheme: 1-hour counts after 6-8 days of decay, 2-hour counts after 14-17 days of decay, and 2-4 hour counts ~50 days after irradiation. In addition, one count is done on an intrinsic Ge, low-energy photon detector (for 1 hour, 8-10 days after irradiation).

Gamma-ray spectra are analyzed for Na, Ca, Sc, Cr, Fe, Co, Zn, As, Rb, Zr, Sb, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Yb, Lu, Hf, Ta, Au, Th and U. Computer processing is done with SPECTRA and associated programs on a VAX 11/780 computer (Grossman and Baedecker, 1987; Baedecker and McKown, 1987; Baedecker and Grossman, 1989). Corrections are made for spectral interferences as well as for interferences on Zr, Ba, La, Ce and Nd from the products of ^{235}U fission in the reactor. Data for Na, Ca and Fe are reported as oxides.

Multielement ICP Analysis
Denver method (Ba, Cr, Co, Ga, Ni, Sr, Y, and Zn).

Trace elements are measured using the ICP-AES method of Lichte *et al.* (1987) and Crock *et al.* (1983). The sample is decomposed using a mixture of HCl, HNO_3, $HClO_4$, and HF at low temperature. The digested sample is taken to dryness, the residue treated with aqua regia, and the solution brought to 10 grams with dilute nitric acid. The solution is nebulized into the ICP-AES plasma discharge.

Reston method (V, Fe, Na)

A 100-mg sample is decomposed with HNO_3, $HClO_4$, and HF and evaporated to dryness overnight. The residue is dissolved in 10 mL of 2N HCl. Analysis of V, Fe and Na is done directly on this solution by ICP-AES (Lichte *et al.*, 1987). Dilutions may be required for higher concentrations of major elements. If the samples are low (<10 ppm) in V the solutions are analyzed directly by GFAA (Aruscavage and Crock, 1987).

Analysis of Silver
Following the method of Aruscavage and Campbell (1979), a 200-mg sample is decomposed using HNO_3, $HClO_4$, and HF, and evaporated to dryness overnight. The residue is dissolved in 15 mL of 20% tartaric acid. The Ag is extracted into butyl acetate as the diphenylthiourea complex. The organic layer is analyzed for Ag by GFAA. Standard solutions are taken through the procedure along with the samples.

Analysis of Niobium
A 100-mg sample is decomposed with HNO_3, $HClO_4$, and HF and evaporated to dryness overnight. The residue is dissolved in 15 mL of 10N HCl. The solution is passed through an ion exchange column to remove the alkali metals. The chloride form of Nb is adsorbed onto the resin. The column is washed with 5N HF to remove Fe. A solution of 7N HNO_3 is poured through the column to quantitatively strip the Nb from the resin.

This fraction is collected and evaporated to dryness. The residue is dissolved in 2 mL of 2N HCl and analyzed by ICP-AES.

Analysis of Co, Cr, Ni and Cu by Atomic Absorption

A 100-mg sample is decomposed with HNO_3, $HClO_4$, and HF and evaporated to dryness overnight. The residue is dissolved in 10 mL of 2N HCl. Analysis of Co, Cu and Ni is done directly on this solution by FAA (Aruscavage and Crock, 1987). If the samples are low (<10 ppm) in Co, Cr and Cu the solutions are analyzed directly by GFAA (Aruscavage and Crock, 1987). Ni at <20 ppm is analyzed by GFAA using a magnesium nitrate matrix modifier.

Analysis of Boron by Emission Spectrography

10 mg of powdered sample is thoroughly mixed and ground with 40 mg of copper hydroxyfluoride (CuOHF) in an agate mortar. Boron is measured on an emission spectrograph using the method of Golightly et al. (1987), based on the volatilization of boron fluorides into a direct-current arc. The CuOHF inhibits the formation of refractory boron carbide on the graphite electrode. The concentration of boron, which may range from 0.2 to 600 ppm, is calculated from the intensity of the light emitted from the arc of the sample electrode relative to that of artificial and natural standards run under similar spectrographic conditions.

Explanation of Tables

The accompanying tables were generated by computer from a large data base. Several general rules governed the display of significant figures. (1) Data for major elements are all given in wt.% to two decimal places. (2) Data for trace elements are given to enough figures that the round-off error is ≤3% and averages ~1%. These rules rarely lead to the display of too few significant figures, but may result in extra trailing zeros when a laboratory reported fewer digits due to low precision.

$Fe_2O_3^*$ represents total iron calculated as Fe_2O_3. The totals of major elements (Σ) have not been corrected for excess oxygen when S, F and Cl are present. Values reported as upper limits were included in the analysis, but were below the detection limit (note that detection limits are matrix dependent by some methods, notably INAA). Dashes are shown for elements that were not analyzed.

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