

Reports

Soudan Formation: Organic Extracts of Early Precambrian Rocks

Abstract. Biological-type alkanes are present in rocks of the Soudan formation that are more than 2.7×10^9 years old, but evidence of life in Soudan times is marginal. The distributional patterns of the alkanes from various regions in the Soudan indicate an indigenous origin of these compounds. Isotopic analyses do not confirm the compositional analyses.

Molecular precursors of life were presumably formed by irradiation of the primordial atmosphere (1). It is believed that biochemical evolution over a long period gave rise to photosynthesizing plants, and that oxygen released by plants during carbon fixation transformed the primordial atmosphere into an oxidizing atmosphere, producing marked changes in the quantity and quality of solar radiation that was received and retained by the earth. The forms of life that evolved during the transition that was presumably caused by the biological evolution would have adapted to and induced additional changes in their environment. Thus, the concurrent evolutions of life and of the earth may have been interdependent and synergistic, and paleobiological investigations may reveal evidence of the nature of these evolutions.

Until 10 years ago it was widely believed that life on earth originated near the beginning of the Cambrian time, approximately 550 million years ago. Barghoorn and Tyler (2), however, supplied much information about the varied forms of fossils in the 2-billion-year-old Gunflint chert, proving that complex, highly distinctive organisms existed long before the Cambrian period. Other investigations suggest a continuum of life from early Precambrian to the present (3).

Cloud (4) described the petrology of the source in the Soudan formation of the rocks used in this and other investigations. Analyses of alkanes from the formation (5) were presented as evidence of a biological origin of the

Soudan alkanes. This report supports the earlier findings and supplies additional evidence that pristane, phytane, and *n*-paraffins are indigenous to the Soudan formation.

Contamination is a major problem in paleobiochemical investigations. No direct method is available for determining the ages of organic compounds that are millions or billions of years old, but it is generally assumed that biological materials in sediments have the same ages as the sediments. This assumption is probably valid for most particulate organic substances, but it may not be valid for the molecular remnants of organisms. The principal components of former life are present in trace quantities in ancient sediments. Handling a rock may greatly increase its complement of certain biological compounds; however, such is not usually true of alkanes. Alkanes are minor constituents of organisms (6); because they are more inert than most biological molecules, the carbon cycle apparently concentrates alkanes in sediments, and the mixing and redistribution of sedimental alkanes make their distribution unlike their distribution in Recent sediments and organisms (6, 7). Thus it is usually possible to distinguish between alkanes from living organisms and from ancient sediments, but a sedimentary rock may also be contaminated in its natural environment.

Petroleum deposits are apparently accumulations of hydrocarbons and other relatively nonpolar organic materials that have migrated from and through rocks and collected in porous beds. (7). Petroleum attests to the pos-

sibility that sediments may be contaminated, and the widespread use of petroleum products increases the likelihood that alkanes may be added to minerals inadvertently.

The contamination problem was carefully considered when the Soudan rocks were analyzed. Two samples of the rocks were analyzed, and a 2-month interval between the two analyses reduced the chances either that cross-contamination could occur or that common solvent and reagent blanks would be added to both samples. Complete laboratory blanks were run, using borosilicate beads in place of the Soudan rocks; blanks contained negligible quantities of alkanes.

Sample 1 was a single piece weighing 1880 g from a carbon-rich silicate layer. Sample 2 consisted of two carbonaceous lenses weighing 490 and 750 g. Both samples were from the 21st level of the Soudan mine, Soudan, St. Louis County, Minnesota (8). All solvents and utensils have been described (9). The samples were extracted in stages. The whole rocks were extracted, and then they were crushed to pieces (largest dimension, 2 mm) and reextracted. The crushed rocks were finally immersed for 1 day in 800 ml of 48-percent hydrofluoric acid, and the HF slurry was extracted. Extractions of whole and crushed rocks were made in a 150-watt ultrasonic extractor at an operating frequency of 20 kcy/sec. The HF slurries of the crushed rocks were extracted in a 2-liter glass separatory funnel fitted with a Teflon stopcock. Sample 1, whole and crushed, was extracted four times in each form; sample 2, whole and crushed, was extracted ten times in each form. A mixture of benzene and methanol (4:1 by volume) was used for these extractions, while the HF slurries were extracted with benzene. For each extraction in the ultrasonic extractor the sample was covered with solvent; individual extractions lasted for 10 minutes (sample 1) or 15 minutes (sample 2). The HF slurries were poured into the separatory funnels and a total of 500 ml of distilled water was used to rinse the residue of each into the funnel. Slurries obtained by the acid treatments of samples 1 and 2 were separately extracted by shaking each slurry for 3 minutes with three successive 300-ml portions of benzene.

Extracts of samples 1 and 2 were divided into fractions. Elemental sulfur

Table 1. Alkane contents of successive organic extracts of sample 1, intact, crushed, and crushed and slurried with hydrofluoric acid. CRS, carbon-rich silicate.

No.	Fraction		Alkanes (%)
	Extracts in	Weight (mg)	
1	<i>Intact CRS (4 extracts)</i>		14
	All 4	30	
2	<i>Crushed CRS (4 extracts)</i>		60
	All 4	11	
3	<i>CRS, crushed and slurried (3 extracts)</i>		10
	All 3	11	

was removed from the fractions (10), and then the fractions were chromatographed on silica-gel columns (9). Special emphasis was placed on analyses of the *n*-heptane eluates from silica gel; these eluates are composed predominantly of alkanes (9), which are probably the most durable molecular remnants of former life (3, 6, 7). Results of the analyses for alkanes appear in Tables 1 and 2 for samples 1 and 2, respectively. My gas-liquid chromatograms were obtained (11) by use of two different Barber-Coleman, Apiezon L-coated, 0.025 by 3000-cm, stainless-steel, capillary columns. Column 1 was programmed to rise 5°C per minute; column 2, 2°C per minute.

Infrared and ultraviolet spectra of silica gel-chromatographic fractions of sample 1 are comparable with spectra of equivalent chromatographic fractions of other extracts of ancient sediments (2, 9). Gas-liquid chromatograms of all the Soudan alkane fractions have well-defined peaks, with retention temperatures equal to those of pristane, phytane, and certain *n*-paraffins. Mass spectra of the alkanes from fractions 1 and 2 of sample 1 contain distinctive "parent ions" of *n*-paraffins (9) and a large peak at mass 183 (12). The mass-spectra and gas liquid-chromatographic analyses for *n*-paraffins, pristane, and phytane are in quantitative agreement. Additions of reference pristane and phytane to fractions 1 and 2 of sample 1 increased the peak heights without either broadening or displacing the peaks believed to be produced by pristane and phytane. Gas-liquid chromatograms of these fractions, with the pristane and phytane added, were obtained on columns 1 and 2.

Gas-liquid chromatograms of the alkanes from the three fractions of extract of the first Soudan sample (Figs. 1-3) all have large base-line humps that are produced chiefly by unre-

solved peaks of branched-chain paraffins and cycloalkanes—the major constituents of the alkanes; in shape the humps generally resemble a Gaussian distribution curve, and the mean molecular weights of the alkanes in fractions 1-3 are approximately defined by the maxima in the humps. The positions of the *n*-paraffin peaks are recorded along the bottom of each chromatogram, and pristane and phytane form the large peaks that appear immediately to the left of *n*-heptadecane (*n*-C₁₇) and *n*-octadecane (*n*-C₁₈), respectively. An alkane (referred to for convenience as the unknown alkane) forms the largest peak in the interval between the *n*-hexadecane (*n*-C₁₆) and pristane peaks. *n*-Paraffins, pristane, and phytane commonly occur in alkanes from biological sources (3, 5, 6, 12). Chlorophyll has been suggested to be a precursor of pristane and phytane (12).

The chromatogram of the alkanes extracted from intact sample 1 appears in Fig. 1. The *n*-paraffin peaks are easily identified throughout the C₁₅ to C₂₇ range, and the *n*-C₁₆, *n*-C₁₇, and *n*-C₁₈ peaks are appreciably larger than the unknown-alkane, pristane, and phytane peaks; a maximum in the base-line humps occurs near *n*-nonadecane (*n*-C₁₉).

The chromatogram of the alkanes extracted from sample 1 after the intact rock had been extracted and then crushed appears in Fig. 2; pristane and phytane produce the two largest peaks and the unknown-alkane peak is larger than the *n*-C₁₆ peak; it is clear that the alkanes extracted from the crushed rock are composed predominantly of nonlinear hydrocarbons, and that the peaks of *n*-paraffins that contain more than 21 carbon atoms cannot be easily identified. Amplitude of the base-line hump is greater in Fig. 2 than in Figs. 1 or 3, and the maximum in Fig. 2 appears near *n*-C₁₈. The alkanes in fraction 2 apparently have a lower and sharper boiling-point range than those in fractions 1 and 3.

A chromatogram of the alkanes released from the silicate phases of sample 1 by treatment of the previously extracted, crushed rock with HF is shown in Fig. 3; all the *n*-paraffin peaks are easily identifiable and the maximum in the baseline humps appears between *n*-eicosane (*n*-C₂₀) and *n*-heneicosane (*n*-C₂₁). The ratios of the *n*-C₁₆: unknown-alkane, *n*-C₁₇: pristane, and *n*-C₁₈: phytane peak

Table 2. Alkane contents of successive organic extracts of sample 2, intact, crushed, and crushed and slurried with hydrofluoric acid; alkanes were isolated by silica-gel chromatography. CL, carbonaceous lens.

No.	Fraction		Alkanes (%)
	Extracts in	Weight (mg)	
<i>Intact CL (10 extracts)</i>			
1	1st	24	10
2	2nd, 3rd	1	63
3	4th, 5th, 6th	2	30
4	7th, 8th, 9th, 10th	4	9
<i>Crushed CL (10 extracts)</i>			
5	1st, 2nd, 3rd, 4th	12	28
6	5th, 6th, 7th, 8th, 9th	3	35
<i>CL, crushed and slurried (3 extracts)</i>			
8	1st, 2nd, 3rd	7	6

heights in Fig. 3 are larger than the same ratios in Fig. 2 and smaller than those in Fig. 1.

The carbonaceous lenses that constituted sample 2 were analyzed in a manner that permitted detailed assessment of the distributions of the alkanes. Eight extract fractions were obtained of sample 2: fractions 1-4 were successive organic extracts of intact rock and fractions 5-7 were successive organic extracts of the crushed rock, while fraction 8 contained the organic materials that were released by treatment of the crushed rock with HF. Fraction 7, the tenth extract of the crushed rock, contained no detectable alkanes. Table 1 shows that the first extracts of the whole and crushed rock contained nearly 80 percent of the organic materials that were extracted from the intact and crushed sample; the data indicate an appreciable degree of isolation between the substances that were successively extracted from whole and crushed rock.

Gas-liquid chromatograms of the alkanes from fractions 1-6 and 8 appear in Figs. 4-10. Comparisons of Figs. 1 and 4-7, Figs. 2, 8, and 9, and Figs. 3 and 10 clearly indicate marked similarities in the distributions of the alkanes within samples 1 and 2. Admittedly Figs. 3 and 10 do differ, but the compositional variations in the alkanes from the whole, crushed, and acid-slurried rocks of samples 1 and 2 follow the same general patterns. In both samples, the alkanes that were bound in the silicate phases appear to be intermediate in composition to the alkanes obtained from intact and

crushed rocks, and the differences between the silicate-bound alkanes of samples 1 and 2 may reflect the degree of isolation of these alkanes; the silicate phases were better defined and more abundant in sample 1 than in sample 2. The resemblances between the alkanes in fractions 6 and 8 suggest that the silicate-bound alkanes were not isolated completely from other alkanes in sample 2.

Pristane, phytane, and *n*-paraffins are widely believed to indicate a biological origin (3, 5, 6, 12); if they are indigenous to the Soudan formation, their presence in the samples may indicate that life existed 2.7 billion years ago. The principal question regarding the source of the Soudan alkanes is whether these hydrocarbons were deposited with or subsequently acquired by the sediments. Minerals (4) and carbonaceous materials in the Soudan rocks clearly indicate that the sediments were subjected to elevated temperatures. Amorphous carbon in the Soudan samples resembles pyrolysis products of organic materials, and the low concentrations of extractable organic compounds in these carbon-rich rocks suggest that some volatile organic substances were expelled.

Analyses shown in Figs. 1–10 indicate that three differently constituted fractions of alkanes are effectively isolated within various matrices of samples 1 and 2. Observations made during the extraction of sample 1 suggest that the alkanes extracted from intact rock were associated mainly with the amorphous carbon. The carbon-rich silicate rock split during extraction, the splitting occurring at major stratifications where the amorphous carbon was concentrated, exposing the carbon to solvents. Alkanes extracted from the crushed rocks were probably distributed chiefly within the small pores. In intact rock, capillary pressure would have excluded solvents from major segments of these pores, but crushing reduced the lengths of pores and made some pores accessible to solvents. Extracts of the acid slurries probably consisted largely of materials that were occluded in silicate phases.

My observations and analyses may be explained by this hypothetical history of the Soudan formation:

1) Minerals and organic materials were deposited together, and consolidation and lithification occurred within several million years of Soudan time.

2) Organic materials occluded in

the silicate phases were partially isolated from similar materials distributed throughout the Soudan rocks.

3) The rocks subsequently reached temperatures between 200° and 400°C and some organic constituents were either pyrolyzed or volatilized.

4) Products of pyrolysis and volatile compounds were expelled from the heated rocks, and viscous and unstable organic substances were extruded along stratifications and into vugs; degradation of these substances produced amorphous carbon. Volatile and stable organic compounds were expelled along lines of stratification and through pores.

5) Stable, polar, and volatile organic substances other than hydrocarbons escaped mainly along the stratifications coated with nonpolar carbon and were concentrated with the *n*-paraffins of high molecular weight, which were preferentially adsorbed on this carbon. Thus, my extracts of intact rocks contained relatively small amounts of alkanes, which were en-

riched in *n*-paraffins of high molecular weight.

6) The small pores in Soudan rocks functioned as molecular sieves; escape through them was primarily limited to *n*-paraffins of low molecular weight and nonlinear alkanes (13). Thus the extracts of crushed rocks contained high concentrations of alkanes that were composed principally of nonlinear hydrocarbons.

7) The silicate-bound and other organic materials were probably degraded to the same extent by the high temperature, but the silicate bound phases most likely did not participate as freely in the redistribution processes as other organic substances.

If these presumptions—an indigenous origin and a common chemical history for the alkanes—are valid, the compositional differences in the alkanes isolated from the various extracts should be attributable to physical processes, and, since the silicate-bound alkanes probably could not participate freely in these processes, alkane com-

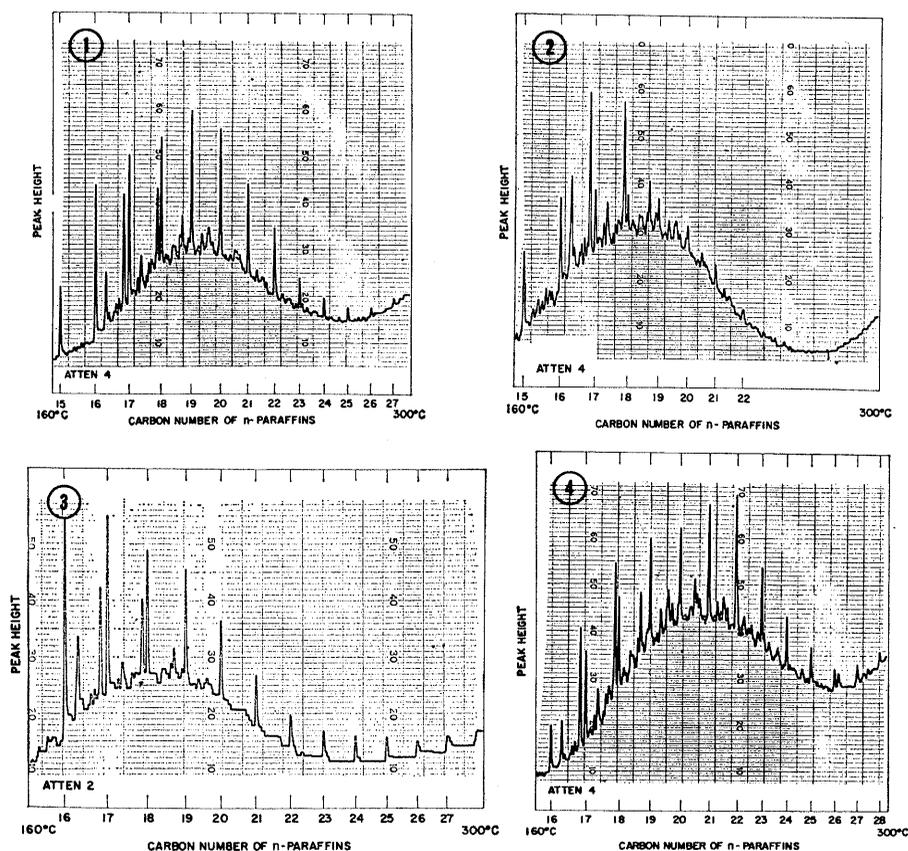


Fig. 1. Gas-liquid chromatogram of alkanes extracted from the intact rock of the first Soudan sample (sample 1). [GLC column 1] Fig. 2. Gas-liquid chromatogram of alkanes extracted from the pulverized rock of sample 1. [GLC column 1] Fig. 3. Gas-liquid chromatogram of alkanes extracted from the pulverized rock of sample 1 after the silicates in the rock had been dissolved in hydrofluoric acid. [GLC column 1] Fig. 4. Gas-liquid chromatogram of alkanes in the first extract (fraction 1) of the intact rocks of the second Soudan sample (sample 2). [GLC column 2]

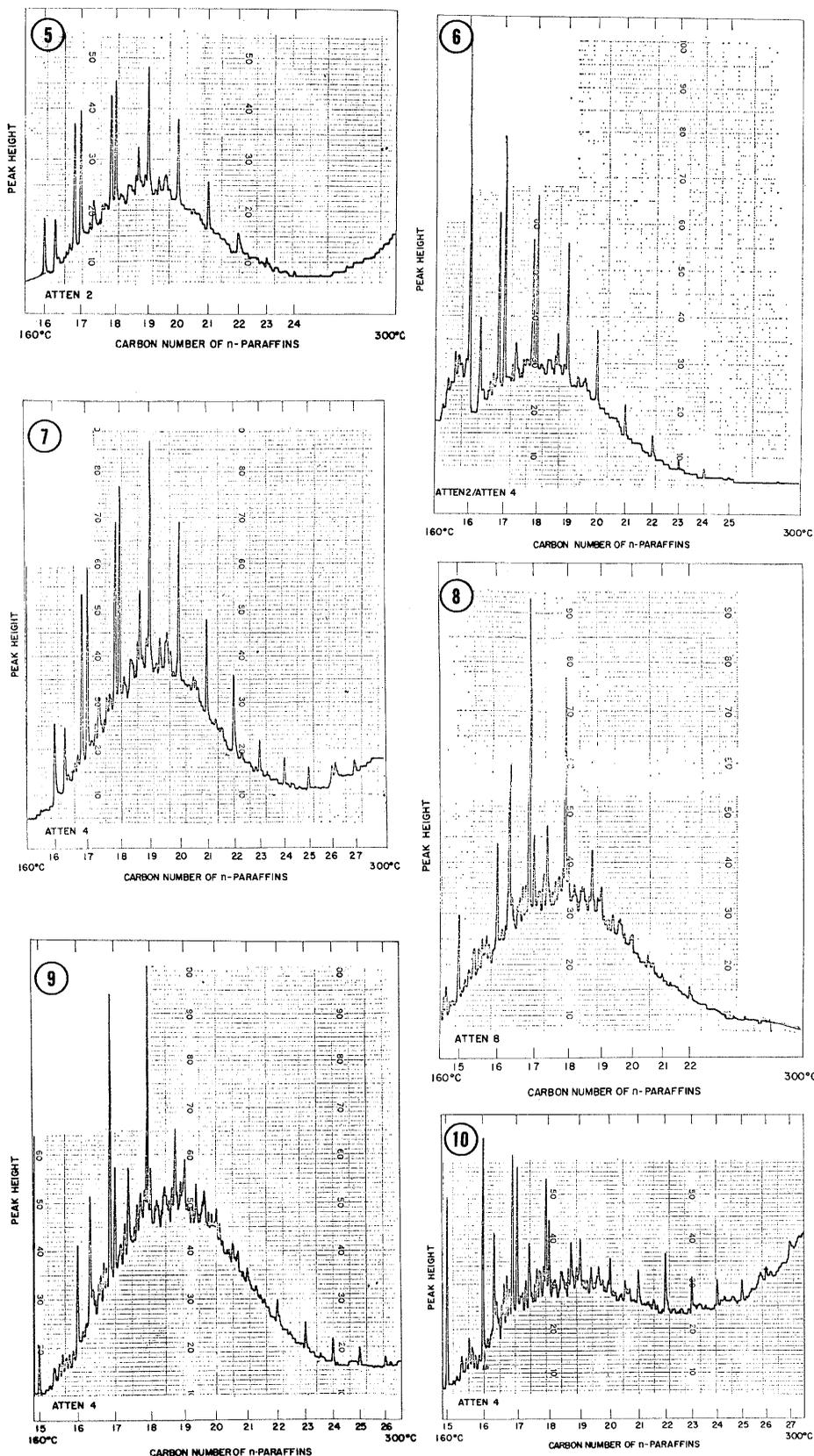


Fig. 5. Gas-liquid chromatogram of alkanes in fraction 2, the second extract fraction of the intact rocks, of sample 2. [GLC column 2] Fig. 6. Gas-liquid chromatogram of alkanes in fraction 3, the third extract fraction of the intact rocks, of sample 2. [GLC column 2] Fig. 7. Gas-liquid chromatogram of alkanes in fraction 4, the fourth extract fraction of the intact rocks, of sample 2. [GLC column 2] Fig. 8. Gas-liquid chromatogram of alkanes in fraction 5, the first extract fraction, of the pulverized rocks of sample 2. [GLC column 2] Fig. 9. Gas-liquid chromatogram of alkanes in fraction 6, the second extract fraction, of the pulverized rocks of sample 3. [GLC column 2] Fig. 10. Gas-liquid chromatogram of alkanes in fraction 8, the extract of the pulverized rocks of sample 2 after the silicates in the rocks had been dissolved in hydrofluoric acid. [GLC column 2]

positions should be intermediate between those of alkanes extracted from intact and from crushed samples. Analyses discussed here and elsewhere (5) seemingly confirm the intermediate composition of the silicate-bound alkanes. When rock was crushed before extraction, there were no significant compositional differences between alkanes that were extracted either initially or after treatment of the crushed rock with HF (5).

If alkanes had moved into the Soudan formation or had contaminated the samples, the most inaccessible part of the samples, the part occluded in the silicates, would most likely have contained the hydrocarbons of lowest molecular weight. It seems unlikely that extraneous alkanes and organics other than hydrocarbons could have become as widely and inhomogeneously distributed as those in samples 1 and 2.

Hoering (14) determined that the δC^{13} values of soluble and insoluble organic matter in most rocks are comparable. However, solid carbon in the Soudan rocks yielded a δC^{13} of -34.54 per mille, while values of -25.06 to -25.99 per mille were measured on four extracts. Hoering observed similar disparities in the isotopic compositions of extracts and of insoluble carbon in some, but not all, early-Precambrian rocks.

Disparities in the carbon-isotopic measurements of the Soudan indicate either different origins or different reactivities of carbonaceous materials. The δC^{13} values of crude oils from different wells may vary as much as 12 per mille (14); thus migration of extractable organic materials into the Soudan formation may explain the anomalous δC^{13} results. This explanation, however, would conflict with my interpretations of the analyses of the Soudan extracts.

Chemical transformations can be isotopically selective (15), and apparent discrepancies in interpretations of the isotopic and compositional results obtained on carbonaceous materials from the Soudan sediments may be alleviated by assuming that alterations caused by high temperatures produced the disparities in the δC^{13} values. C^{13} and C^{12} are concentrated in the oxidized and reduced compounds, respectively, which are produced by associated reactions at equilibrium (16). If thermal degradation of biological remnants in the Soudan formation formed amorphous carbon and extractable organic materials, the amorphous carbon

may be isotopically lighter than the organic extracts, which contain oxygenated compounds. The mean $\delta^{13}\text{C}$ value for carbonaceous substances in sediments is approximately -29 per mille, a value that is intermediate between the Soudan values.

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Igneous Rocks of the Indian Ocean Floor

Abstract. Four dredge hauls from near the crest and from the eastern flank of the seismically active Mid-Indian Ocean Ridge at 23° to 24°S , at depths of 3700 to 4300 meters, produced only low-potassium tholeiitic basalt similar in chemical and mineralogic composition to basalts characteristic of ridges and rises in the Atlantic and Pacific oceans. A fifth haul, from a depth of 4000 meters on the lower flank of a seamount on the ocean side of the Indonesian Trench, recovered tholeiitic basalt with higher concentrations of K and Ti and slightly lower amounts of Si and Ca than the typical oceanic tholeiite of the ridge. The last sample is vesicular, suggesting depression of the area since the basalt was emplaced. Many of the rocks dredged are variously decomposed and hydrated, but there is no evidence of important chemical modification toward conversion of the lava flows to spilitite during extrusion or solidification.

The complex ridgelike features and extensive intervening basins of the Indian Ocean were first clearly indicated by surveys of the Dana and John Murray expeditions, and by the gravity-measuring traverses of Vening Meinesz (1). Recent oceanographic investigations undertaken by the International Indian Ocean Expedition, 1960-1965, have further clarified several major geological features, especially its fascinating patterns of both elongated and bifurcated faulted ridges, the large "plateaus" and irregular deeps, and the texture of the deep-sea floor (2). Figure 1 is a simplified map of the Indian Ocean showing one version of the gross patterns of the ridge complex shallower than 4000 m, and the locations of islands and dredge samples discussed in this report.

Early petrologic studies in the Indian Ocean were confined to the volcanic islands which lie along and flank the Indian Ocean ridges (3). These islands include Mauritius, Rodriguez, Réunion, New Amsterdam, St. Paul, Crozet, and the Kerguelen Archipelago (Fig. 1). Each consists of one or more variously coalescing, sometimes fragmented, volcanic cones. Continuing studies, ranging over more than a century, have shown that the dominant volcanic rocks capping these islands and the higher submarine volcanoes are alkali basalts, which are commonly accompanied by subordinate, derivative members of the alkali series, especially andesine- and oligoclase-andesites, trachytes, and more rarely phonolites and rhyolites (4). Olivine-rich (picritic) basalts are

found on some of the islands; tholeiitic basalts with low concentrations of potassium, high concentrations of calcium and silica, and high ratios of calcium to magnesium are extruded, notably on Mauritius and Réunion islands (5). Small inclusions of magnesium-rich peridotite and pyroxene amphibolite occur in the alkali basalts and in associated beds of ash, but these inclusions represent less than 0.1 percent of the exposed rock. The alkali-rich basalts comprise over 90 percent of the igneous rocks of most islands.

These relative abundances of rock types from the exposed parts of the volcanic cones along the Indian Ocean ridge systems are similar to those from volcanic islands of the Atlantic and Pacific oceans, where alkali basalts and subordinate members of the alkali series also predominate. In the Atlantic and Pacific, olivine-rich basalts are common but volumetrically subordinate. Tholeiitic basalts occur only rarely, except in the several major oceanic rift-island systems such as Hawaii and Iceland, where they are abundant (6).

The alkali basalts which are typical and predominant constituents of the higher oceanic volcanoes are characterized, among the basalts, by relatively high concentrations of K, Na, Ti, and P and a high ratio of Fe^{3+} to Fe^{2+} . They also contain distinctly higher concentrations of Ba, La, Nb, Pb, Rb, Th, U, and Zr, and higher ratios of K to Rb, Sr^{87} to Sr^{86} , and Pb^{206} to Pb^{204} than are found in picritic and tholeiitic basalts of the oceans. Detailed discussions of the chemical characteristics of oceanic basalts, including abundances of minor and trace elements and of the isotopes of strontium and lead, are being published separately (7).

The dominance of alkali-rich basalts on readily accessible oceanic islands caused early petrologists to suggest that these rocks were characteristic of the entire oceanic crust (8). It was in the light of this petrologic concept that Wiseman (9) examined and first described dredge hauls from the more deeply submerged parts of the ridge complex in the northwestern Indian Ocean. The location of Wiseman's samples is shown in Fig. 1 (position JM). Wiseman recognized that the ridge rocks were not typical alkali basalts like those found on the islands of the Indian Ocean. He noted that the dredged basalts were relatively uni-