Reports

Water and Carbon in Rusty Lunar Rock 66095

Abstract. *Lunar rock 66095 contains a hydrated iron oxide and has an unusual amount of water for a lunar rock (140 to 750 parts per million), 90 percent of which is released below 690°C. The* δ*D of water released at these low temperatures varies from* -75 *to* -140 *per mil relative to standard mean ocean water (SMOW). The small amount of water released between 690° and 1300°C has a* δ*D of about* -175 ± 25 *per mil SMOW. These* δ*D values are not unusual for terrestrial water. The* δ^{18}*O of water extracted from 110° to 400°C has a value of* $+5 \pm 1$ *per mil SMOW, similar to the value for lunar silicates from rock 66095 and different from the value of* -4 *to* -22 *per mil found for samples of terrestrial rust including samples of rusted meteoritic iron. The amount of carbon varies from 11 to 59 parts per million with a* δ^{13}*C from* -20 *to* -30 *per mil relative to Pee Dee belemnite. Only very small amounts of reduced species (such as hydrogen, carbon monoxide, and methane) were found, in contrast to the analyses of other lunar rocks. Although it is possible that most of the water in the iron oxide (goethite) may be terrestrial in origin or may have exchanged with terrestrial water during sample return and handling, evidence presented herein suggests that this did not happen and that some lunar water may have a* δ*D that is indistinguishable from that of terrestrial water.*

Lunar rock 66095 exhibits rusty-appearing streaks, which have been identified as goethite, a hydrated iron oxide. Agrell *et al.* (*1*) have noted the occurrence of small amounts of goethite in Apollo 14 breccias 14301 and 14307. However, in contrast to the Apollo samples, the goethite in sample 66095 is abundant enough to give the visual impression of rusty streaks in the rock.

Although the evidence appears strong that the rusty streaks were present when the sample boxes were opened at the Lunar Receiving Laboratory in Houston (*2*), we cannot be sure that the original rusty color was due to goethite. It is possible that the reddish-brown color was due to an unhydrated iron oxide, which then hydrated during sample processing. The samples were handled in glove boxes under a dry N$_2$ atmosphere, but the total exclusion of water vapor cannot be assured.

The question of the lunar origin of goethite is important, since it would imply the existence of larger amounts of water, at least on isolated parts of the moon, than has been shown from the analysis of lunar materials returned from earlier missions. Williams and Gibson (*3*) have discussed the stability of goethite and conclude that it could be produced as the result of normal low-temperature processes indigenous to the moon.

The possible origins for the water in the goethite are: (i) lunar; (ii) terrestrial—the goethite formed after sample return; or (iii) partly lunar and partly terrestrial—the goethite formed on the moon but exchanged part or all of its water after sample return. The research reported here is an attempt to decide the origin of the water in the goethite.

The experiments were conducted in four phases. The first group of experiments was designed to determine the δD [δD = D/H relative to standard mean ocean water (SMOW)] and δ^{13}C [δ^{13}C = ^{13}C/^{12}C relative to Pee Dee belemnite (PDB)] of volatiles in rock 66095 with reasonably high precision and to compare these values with corresponding values for materials extracted from other lunar samples. In the next group of experiments the primary aim was to measure the δ^{18}O (δ^{18}O = ^{18}O/^{16}O relative to SMOW) of the water extracted at low temperature (below ~ 400°C). In some of these experiments δD was also measured. In the third group of experiments we measured the δ^{18}O of water extracted from terrestrial rust from several sources to compare these values with the δ^{18}O of water extracted from rock 66095. In the last group of experiments, we measured the δ^{18}O of the oxygen in the goethite after dehydration in a vacuum at 400°C.

All of the samples were transported from Houston to Denver in sealed stainless steel containers (McKinney containers) and were opened just moments before being introduced into the vacuum system. All of the samples were in the form of approximately cubic fragments about 0.5 to 1 cm on a side. They were handled only with cleaned platinum forceps and were exposed to laboratory air for a maximum of 5 minutes before being evacuated. In the pyrolysis experiments, the samples were outgassed at 110°C for 24 hours in a vacuum before being dropped into a cooled platinum crucible which had been outgassed at 1400°C in a vacuum. The crucible, contained in a fused quartz vacuum envelope, was slowly heated by an external furnace or induction heater to a fixed temperature and maintained at this temperature for several hours, until gas evolution ceased. The condensable gases were continuously frozen in a trap cooled with liquid N$_2$. Noncondensable gas was pumped by means of a small mercury diffusion pump through a copper oxide furnace heated to between 600° and 700°C, and the products of the reaction were collected in another trap cooled with liquid N$_2$. Any noncombustible gas, mainly helium, was finally pumped into the mass spectrometer inlet system; after the mass 3 and 4 peak intensities had been measured, it was pumped away. Water and CO$_2$ (plus sulfur gases) in both traps were separated at Dry Ice temperature. The water was converted to H$_2$ and O$_2$ by reaction with uranium metal at 700°C, and the H$_2$ was pumped into the mass spectrometer where its δD and volume were determined. The CO$_2$ was separated from sulfur gases by distillation at the temperature of melting

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ethyl alcohol. Then the CO_2 was transferred to another mass spectrometer, where its $\delta^{13}C$ and volume were measured. The volumes of H_2 and CO_2 were calculated from ion-intensity measurements, as well as from vacuum gage readings in calibrated volumes. Temperatures below 970°C were obtained by means of an external furnace; radio-frequency induction heating was used to obtain higher temperatures.

Calibration of the mass spectrometers showed that it was possible to measure amounts of water (as H_2) as small as 2 ± 0.2 μg, and to determine δD to ± 20 per mil on such samples. With samples as large as 20 μg, δD was precise to ± 2 per mil. With CO_2 we could measure about 10 μg to a precision of ± 1 μg and $\delta^{13}C$ to a precision of ± 5 per mil on such small samples.

One sample was combusted at 975°C in 1 atm of purified O_2 in a specially constructed clean combustion system, free from carbon- and hydrogen-containing materials. Blanks showed less than 0.5 μg of water and less than 1 μg of CO_2 and SO_2. The results of the three analyses are given in Table 1.

Rock 66095 emitted larger amounts of water at markedly lower temperatures than previous lunar samples (*4, 5*). Almost no reduced gases (H_2, CO, CH_4) were observed, again in contrast to previous lunar samples. The amount and isotopic composition of carbon in this rock are not unusual by comparison with values for other lunar rocks.

Epstein and Taylor (*5*) have suggested that the $\delta^{18}O$ of water extracted from lunar rocks at temperatures below ~ 400°C should provide a test of whether the water was of lunar origin. Their argument was that, since lunar water could originate from the interaction of hydrogen, either as solar wind protons or derived by other processes, with the oxygen of lunar silicates, the water so produced should have a $\delta^{18}O$ similar to that in lunar rocks, namely, + 5.5 to + 7 per mil.

In order to analyze the δD and $\delta^{18}O$ of water from lunar rock 66095, a 6-g sample was sealed in our equipment described above. After evacuating for approximately 1 minute to a pressure of 10^{-3} mm-Hg, we collected water and CO_2 by cooling a trap connected to the apparatus with liquid N_2. After several hours of collecting with the sample at room temperature, the CO_2 evolved was transferred to the mass spectrometer and its amount and $\delta^{13}C$ and $\delta^{18}O$ were measured. The evolved water was allowed to react with carbon heated to 900°C in an apparatus similar to that described by Majzoub (*6*). The H_2 produced by the carbon-water reaction was allowed to diffuse through a palladium-silver thimble. The H_2 was then collected by adsorption on charcoal at liquid N_2 temperature and transferred to the mass spectrometer where its δD was measured. The CO produced by the reaction was converted quantitatively to CO_2 (plus carbon) by a high-voltage discharge. The amount of CO_2 was determined in a manometer, and the CO_2 was transferred to a mass spectrometer for $\delta^{18}O$ analysis. Volatile material was collected from the lunar sample at various temperatures. The results are given in Table 2.

Table 1. Isotopic analyses of water, hydrogen, and carbon from lunar rock 66095. The pyrolysis procedure is described in the text.

Temperature (°C)	Water (ppm)	δD	CH_4, CO		CO_2	
			C (ppm)	$\delta^{13}C$	C (ppm)	$\delta^{13}C$
		Rock 66095-62 (2.46 g)				
100°–270°	223	− 80	0.2		1.9	− 43
270°–690°	158	− 75	0.1		6.3	− 12
690°–935°	7.2	− 200			2.4	− 22
935°–1350°	1.8	− 170			0.2	
Total	390				11.1	
Weighted average		− 81				−20
		Rock 66095-31 (4.67 g)				
100°–270°	60	− 100			1.1	− 24
270°–690°	156	− 140	0.02		21.7	− 26
690°–970°	9.0	− 150			14.9	− 38
970°–1350°	4.5	− 160	4.5	− 19	16.7	− 33
Total	230				59	
Weighted average		− 130				− 30
		Rock 66095-31 (2.27 g)				
Combustion (+ 975°)	137	− 110			15.0	− 21

In order to compare the amounts of water and CO_2 extracted from the lunar sample and the δD and $\delta^{18}O$ values with the corresponding values for terrestrial material, a sample of terrestrial "Green Dragon" rust was treated in a similar fashion. This rust was obtained from a 1951 automobile which spent its first year on the south side of Chicago exposed to steel mill fumes. It then continued to rust for 10 years in Washington, D.C., and then for 11 years in Denver, Colorado. We believe that the Green Dragon rust is a representative sample of U.S. rust and should have a variable $\delta^{18}O$, reflecting the variability of the $\delta^{18}O$ of the water involved in the rusting process. The data in Table 2 show that the water from Green

Table 2. Isotopic analyses of water and carbon from lunar rock 66095 and several rust samples. The water was processed by carbon reduction as described in the text.

Temperature (°C)	Water			CO_2			CH_4, CO
	ppm	δD	$\delta^{18}O$	C (ppm)	$\delta^{18}O$	$\delta^{13}C$	C (ppm)
			Rock 66095-20 (6.19 g)				
25°	1,300	− 140	− 1 ± 1	3.6	+ 44	− 18	
25°–110°	360	− 160	+ 6 ± 2	7.1	+ 31	− 17	
110°–200°	500	− 140	+ 5 ± 2	2.9	+ 48	− 12	
200°–350°	240	− 85	+ 5 ± 2	20	+ 28	− 17	~ 2
350°–490°	~ 15	Sample lost		~ 1	+ 46		~ 1
490°–670°	20	− 183	+ 20 ± 10	2.6	+ 24	− 18	
110°–350°	740*		+ 5 ± 2				
			Green Dragon rust (0.58 g)				
25°	4,800	− 120	+ 7 ± 10	300			
25°–120°	17,800	− 100	− 1 ± 2	2,000			
120°–240°	65,000	− 150	− 5 ± 2	2,000			
240°–400°	50,000	− 157	− 2 ± 2	3,000			
400°–600°	~ 1,000			~ 500			
120°–400°	115,000*		− 4 ± 2				
			Rust from Cranbourne, Australia, meteorite (0.082 g)				
50°–300°		− 170	− 11 ± 1				
			Rust from Odessa, Texas, meteorite (0.058 g)				
70°–125°		− 90	− 5 ± 2				
125°–360°		− 160	− 1 ± 1				

* Accumulative water for the temperature range given.

Dragon rust is variable in composition, but more depleted in $\delta^{18}O$ than rock 66095, and that the water from rock 66095 does have a $\delta^{18}O$ similar to that of lunar silicates from rock 66095.

As an additional check on our $\delta^{18}O$ analysis, samples of rock 66095-20, Green Dragon rust, and rust collected from rusted meteoritic iron (Canyon Diablo, Arizona; Campo del Cielo, Argentina; Cranbourne, Australia; Odessa, Texas; and Wolf Creek, Western Australia) were outgassed at 50° to 100°C in a vacuum and then heated to approximately 400°C. All water evolved between these temperatures was collected and reacted with BrF_5 to give O_2 gas. The O_2 was reacted with hot graphite to give CO_2, which was then analyzed for $\delta^{18}O$ (*7*). The results are given in Table 3. In this case also the water extracted from rock 66095 gives a $\delta^{18}O$ close to that for the silicates from rock 66095 ($+6.3 \pm 0.1$ as determined by BrF_5 treatment of rock 66095), whereas the terrestrial rust of various origins has variable $\delta^{18}O$ but is depleted in $\delta^{18}O$ relative to rock 66095.

It has been suggested that the rust in rock 66095 was formed by the rapid reaction of lunar lawrencite, $FeCl_2$, with moist air shortly after sample return (*8*). Lawrencite has also been postulated to be the precursor of rust in certain meteorites that contain an anomalously high chloride content. Several of the samples of meteoritic rust that we analyzed came from meteorites with high chloride contents. For example, an analysis of material from Cranbourne similar to that analyzed by us gave 0.3 percent total chloride and 0.15 percent water-soluble chloride (*9*).

We carried out another group of experiments with rock 66095 and meteoritic rust to examine the ^{18}O content of the oxygen in the ferric oxide itself as well as in the oxygen of the adsorbed or hydration water. First, the sample of rock 66095 that had previously been heated to 400°C in a vacuum as described above was allowed to remain in the laboratory for 7 months in a container that was not tightly sealed. The sample was then outgassed in a vacuum at 75°C and then heated to 400°C in a vacuum. Water collected at the higher temperature was processed with BrF_5 and analyzed. It had a $\delta^{18}O$ of -7.3 per mil. The sample was then heated to 600°C, and H_2, purified by diffusion through palladium, was reacted with the iron oxide to give water. This water was also processed with BrF_5 and analyzed. It gave a $\delta^{18}O$ of $+1.1$ per mil. We believe that the $\delta^{18}O$ of the iron oxide was originally $+5$ per mil but that some exchange between the oxygen in the iron oxide and the oxygen in the adsorbed water had occurred, which raised the $\delta^{18}O$ of the adsorbed water and lowered the $\delta^{18}O$ of the iron oxide. A sample of Odessa meteoritic rust when treated in the same manner as rock 66095 gave a $\delta^{18}O$ of -1 per mil for oxygen in the adsorbed water and -4.7 per mil for the iron oxide oxygen.

Table 3. Analyses of the $\delta^{18}O$ (iron oxide oxygen) of water extracted from various materials by heating in a vacuum to 400°C.

Weight (g)	Temperature (°C)	Water	
		ppm	$\delta^{18}O$
	Rock 66095-20		
1.15	115°–280°	150	$+5 \pm 2$
	280°–400°	75	
	Green Dragon rust		
	85°–500°		-19 ± 1
	85°–350°		-17 ± 0.5
	Terrestrial rust from Canyon Diablo meteorite		
	60°–300°		-14 ± 1
	Terrestrial rust from Campo del Cielo, Argentina, meteorite		
	85°–350°		-16 ± 1
	Terrestrial rust from Cranbourne, Australia, meteorite		
10.0	50°–300°		-4.6 ± 0.1
0.05	50°–300°		-18 ± 1
0.05	50°–300°		-22 ± 1
0.05	50°–300°		-22 ± 1
0.05	50°–300°		-21 ± 1
	Terrestrial rust from Odessa, Texas, meteorite		
4.0	50°–300°		-8.7 ± 0.1
	Terrestrial rust from Wolf Creek, Western Australia, meteorite		
4.0	25°–120°		-22.8 ± 0.1
	120°–300°		-6.3 ± 0.1

Is any of the water emitted by these samples lunar? If we assume that lunar hydrogen (and water) is derived mainly from the solar wind and is depleted in deuterium, then we see little of such material! If lunar water had been present in the goethite, it may have exchanged with terrestrial water vapor during sample return and handling.

Another equally valid assumption is that the bulk of this water is truly lunar, and that some lunar water is not too different in isotopic composition from the terrestrial water. For many reasons, we favor this view. The $\delta^{18}O$ of the water extracted from rock 66095 at temperatures from 25° to 400°C resembles the $\delta^{18}O$ of lunar silicates from rock 66095 and is appreciably enriched in ^{18}O by comparison with terrestrial water vapor and with water extracted from samples of terrestrially formed rust.

Various investigators have reported some lunar samples that are highly depleted in deuterium and others that are enriched in deuterium, as compared with the hydrogen composition of terrestrial samples (*4, 5*). Any sample of hydrogen (or water) from lunar rocks that shows a deuterium abundance in the terrestrial range has been suspected of having undergone terrestrial contamination. Perhaps we should be more open-minded in trying to define lunar water.

The latest estimate by Brinkmann (*10*) of the escape rate of hydrogen from the upper atmosphere of the earth is 1.6×10^{9} atom cm^{-2} sec^{-1}. This would result in a total loss through geologic time of about 2 percent of the amount of hydrogen present as water on the earth. Even if no deuterium were lost by this escape, the δD of the "original" terrestrial water would have been only approximately -20 per mil. (The present δD range in terrestrial water is -200 to $+50$ per mil with 98 percent of all water on the earth having a value of zero.)

If during its formation the moon accumulated water as ice or combined as hydrated compounds in a manner similar to that of the primitive earth, the hydrogen isotopic composition of this water should have been about -20 per mil. Any subsequent loss of hydrogen from the moon, either as free hydrogen or as water, probably would not have resulted in a sizable isotopic fractionation of deuterium and protium (light hydrogen). The addition of hydrogen by solar wind would presumably have enriched the moon in protium, and samples of protium have been reported in lunar materials. Deuterium produced by spallation caused by cosmic-ray bombardment of lunar rocks would tend to enrich the moon in deuterium, and some enrichment in deuterium relative to that of terrestrial samples has also been observed in lunar rocks (*4, 5*). From the foregoing considerations, we would expect lunar water from sources deep in the moon to approximate terrestrial water in its deuterium abundance. Perhaps the water in rock 66095 is such a sample.

The data of Krähenbühl *et al.* (*11*) and Nunes and Tatsumoto (*12*) show that rock 66095 is highly enriched in volatile materials by comparison with other lunar rocks. These authors suggest that this enrichment in volatiles is caused by volcanic or fumarolic activity. Water released by such activity may be juvenile lunar water. It should resemble terrestrial water in its δD value and should have a $\delta^{18}O$ of $+5$ to $+8$ per mil, as we have found for the water extracted from rock 66095.

Note added in proof: Epstein and Taylor have reported (*13*) that they did not detect $\delta^{18}O$ in their aliquot of sample 66095, and they conclude that the water in this lunar rock is terrestrial. We do not know the reason for the apparent disagreement between their results and ours. Since we used two different techniques to extract the water for analysis with similar results, we do not believe that our experimental techniques can be the problem. Epstein and Taylor analyzed samples that had been stored for a year longer than ours. Perhaps exchange with terrestrial water occurred during this period. The positions of our respective samples in the original lunar boulder from which the astronauts broke off sample 66095 may be important. In spite of the excellence of the data of Epstein and Taylor, we believe that our analysis and conclusions must remain unchanged.

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Scatological Origin of Microvertebrate Fossil Accumulations

Abstract. *Small-mammal bone found in Recent carnivore droppings (scat) is identical in appearance with that in many Mesozoic and Tertiary microvertebrate fossil collections. Such fossil specimens passed into or through the digestive tracts of carnivores before being left as scat that was later reworked into sedimentary rocks. The term "coprocoenosis" is proposed for such an assemblage. Caution is urged in drawing conclusions about the composition of paleocommunities because carnivores can catch prey representing several different communities and leave a mixed assemblage in a particular depositional environment.*

A typical Mesozoic or Tertiary microvertebrate collection consists of isolated mammalian and reptilian teeth, upper and lower jawbones, and other fragmentary skeletal remains. Most of the fragmentary limb bones have sharp breaks on their edges, some of which may have occurred during the collection process (*1*), but most of which, judged by the abundance of such fragments, must have occurred immediately prior to deposition (*2*).

Remains are found in sands and clays, and in spite of their fragmentary nature the small bones show little evidence of abrasion. Most interpretations of these accumulations assume that the animals lived and died near a stream or lake and that their bones were disaggregated by running water once the soft tissues had decomposed (*3*). That would explain the presence of whole limb bones (which occur rarely), but it is difficult to envision a stream that would fragment or shatter bones into small pieces and yet leave the bones free of evidence of abrasion. It is also difficult to explain how bones could have been fragmented in lake deposits, far from any source of hydraulic energy. Trampling of the bones by large tetrapods might be postulated if the ground surface were hard, but it is difficult to see how bones 1 or 2 cm long could be broken into pieces when they are pushed into soft muds.

I suggest that most or all microvertebrate fossil accumulations first passed into or through the digestive tracts of carnivores (mainly mammalian, but including predacious fish, reptiles, and birds) and were deposited as fecal droppings (scat) in or near a stream, lake, or other basin, where they were subsequently covered by sediment. I propose the term "coprocoenosis" for such an accumulation (*4*).

Using Pearson's (*5*) technique, I extracted bone fragments from bobcat, coyote, and badger scat that was collected in southeast Wyoming and northeast Colorado. Bones so obtained are totally clear of soft tissues and show little evidence (other than breakage) of what they have undergone (Fig. 1c). Because bone will break down readily in an acid environment, its residence time in the carnivore stomach must be relatively short. Nor do carnivores thoroughly masticate the small mammals they consume. One scat sample (Fig. 1a) contained a still articulated, although broken, radius and ulna of the plains pocket gopher *Thomomys bottae*. Another contained the perfectly preserved ribs of the tiny deer mouse *Peromyscus*. Bones of large mammals such as deer, on the other hand, are fragmented and heavily chewed. The chewed edges of such pieces give them a water-worn appearance, and if found in a sedimentary deposit such material might mislead a worker to assume that hydraulic action had been involved in its development.

The similarity between bony remains in disaggregated scat and microvertebrate fossil collections is striking (Fig. 1d). Not only is there a similar size range, but certain bones show breaks at identical loci.

There are records of identifiable mammal bones found in coprolites (fossil scat, Fig. 1b), although such occurrences are not common. Lundelius (*6*), however, discovered coprolites and bone fragments in Australian caves where they were left by marsupial carnivores (*7*). Not surprisingly, his material is identical in appearance with disaggregated placental carnivore scat.

To judge whether scat deposition can account for so many microvertebrate fossils, I refer again to Pearson (*5*), who obtained over 5000 rodent jaws from scat that had accumulated over a 9-month period on a 35-acre (14-hectare) study plot. That is an annual accumulation rate of over 100,000 jaws per square mile (400 per hectare).

The scat accumulation hypothesis explains why birds, bats, and arboreal mammals are so rare in Tertiary microvertebrate collections (*8*). When they do occur, they probably reflect capture by flying predators (*9*).