

GAS INTERACTION STUDIES WITH LUNAR ORANGE SOIL 74220,29, by D. A. Cadenhead and W. G. Buerger, Department of Chemistry, State University of New York at Buffalo, Buffalo, New York 14214.

Ever since its discovery during the Apollo 17 mission, the orange soil has been the subject of considerable interest because of its unusual nature.<sup>1</sup> The present consensus of opinion would seem to favor a volcanic origin and we would like to present here, outgassing and gas interaction studies which support this view. The sample was placed in an ultra high vacuum system previously described,<sup>2</sup> outgassed and then exposed to both molecular and atomic hydrogen. After each exposure, the sample was again outgassed and the nature and relative quantities of the various gases released were recorded.

Pretreatment. Since the initial outgassing of any lunar sample may provide valuable information on its origin, special precautions were made to insure minimum contamination. Our assumption was that an initial low temperature outgassing would remove all physisorbed (but not chemisorbed) contaminants. Prior to insertion of the sample in the U.H.V. system, the entire system was baked out at 350°C overnight to attain a background pressure of  $5 \times 10^{-10}$  torr. The sample was inserted in the system by opening up only the immediate vicinity of the sample chamber. Since it proved impossible to maintain a continuous nitrogen atmosphere during transfer, the sample was briefly exposed to air. System and sample were then outgassed at 150°C for eight hours and at 25°C for approximately six months. The final background pressure, while still at room temperature, was  $2 \times 10^{-10}$  torr. The temperature was then raised to 400°C at approximately 2°C/min. with outgassing spectra being recorded every five minutes. Gas release, as expected, was not substantial until approximately 150°C was reached.

1st Outgassing. The primary peak observed on this first outgassing was that of water vapor (250-275°), the amount observed being approximately double that obtained for equal amounts of samples 14163,111 and 15565,3G.<sup>2</sup> Moreover, the impression gained from the pressure measuring gauge, the residual gas analyser when under rapid scan and the ion pump controller current was that the gas was being released in a rapid sequence of small bursts rather than in a continuous stream; an observation not previously made.<sup>2</sup> In addition, substantial amounts of N<sub>2</sub>/CO, CO<sub>2</sub> and SO<sub>2</sub> with traces of H<sub>2</sub> and (somewhat surprisingly) HCl. These data should be compared with those of E. K. Gibson and G. Moore on a similar sample.<sup>3</sup> At the conclusion of this experiment, the temperature was raised to 450° for 2 hours to reduce gas emission below 400°C to a hopefully insignificant level. The final pressure attained, however, was only  $10^{-8}$  torr.

2nd Outgassing. The sample was now exposed to  $2.5 \times 10^{-2}$  torr of molecular hydrogen at 150°C for 36 hours. Previous experience has shown us that, under these conditions, hydrogen dissociatively chemisorbs on lunar samples.<sup>2</sup> At the end of this time, the sample was allowed to cool while being continuously Vac-Ion pumped. Substantial amounts of water vapor were

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seen at low temperatures but amounts appeared to fall off rapidly with increasing temperature. There was no doubt that this water was immediately formed on exposure of the sample to hydrogen since the enhancement of the water peak was apparent each time hydrogen was admitted to the system. Other peaks observed were  $N_2/CO$  and again some  $HCl$ . The precise quantitative data for this particular experiment are somewhat in doubt, however, because of the not insignificant background pressure of  $10^{-8}$  torr. At the conclusion of this experiment the entire U.H.V. system was baked out at  $200^\circ C$  for three hours using a triple-stage liquid-nitrogen trapped mercury diffusion pump. The working system was then isolated and Vac-Ion pumped until a background pressure of  $5 \times 10^{-10}$  torr was attained.

3rd Outgassing. The sample was once again exposed to  $5 \times 10^{-2}$  torr of molecular hydrogen, this time at room temperature. A thoroughly outgassed tungsten filament was switched on for one hour at  $1250^\circ C$  in order to produce high concentrations of hydrogen atoms. The system was then outgassed from 25 to  $400^\circ C$ . Substantial amounts of water vapor were again observed at low temperatures ( $25-75^\circ C$ ), the amount of water released with increasing temperature decreased until the temperature approached  $400^\circ C$ . In addition, substantial amounts of methanol were observed at low temperatures ( $\sim 75^\circ C$ ). At the conclusion of this experiment, the sample was again outgassed for 2 hours at  $450^\circ C$ . A satisfactory background pressure of  $5 \times 10^{-10}$  torr was then obtained at room temperature.

4th Outgassing. The conditions prior to the second outgassing were essentially repeated [ $5 \times 10^{-2}$  torr  $H_2$  at  $150^\circ C$  for 36 hours]. Again, a low temperature water peak was observed ( $\sim 150^\circ C$ ) with a rapid fall in amount until the temperature exceeded  $400^\circ C$ . Methanol was not detected during this experiment.

Conclusions. The large amount of water vapor seen at higher temperatures ( $200-400^\circ C$ ) during the first, but not subsequent, outgassing experiments suggests that it, at least in part, arises from origins other than the solar wind. The presence of  $CO_2$  and  $SO_2$ , as has been pointed out by Gibson and Moore,<sup>3</sup> are indicative of a volcanic origin for 74220. Our water vapor observations, including the curious release pattern, would seem to support this conclusion and the small amounts of  $HCl$  provide further evidence. [Part of the water vapor and possibly all of the  $HCl$  during the second outgassing would appear to have been due to improper cleaning of the system after the 1st outgassing].

The failure to produce large amounts of water in the  $200-400^\circ C$  range is ascribed to the absence of an extensive amorphous layer in the orange soil.<sup>4</sup> The more rapidly released water vapor at lower temperatures (2nd, 3rd, 4th outgassing) is assumed to originate primarily from hydrogen interaction with the external (accessible) surface.

The methanol observation during the 3rd outgassing experiment undoubtedly arose through the tungsten filament having trace carbon impurities.

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This carbon and atomic hydrogen must have interacted with the oxygen in the soil to produce methanol. Since carbon is present in the solar wind, particularly during solar flare periods, the observation is not without some significance.

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