DEUTERIUM ANALYSIS OF HYDROGEN EXTRACTED FROM LUNAR MATERIAL.

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I - EQUIPMENT AND PROCEDURE.

1 - About 200 mg aliquots of a sample are transferred from the shipment container to a gold foil wrapping, in a glove box, under dry nitrogen. They are inserted in a silica tube, connected to a ground joint and a stopcock, then attached to a vacuum line where they can be heated to 1100°C by a D.C. current furnace.

A liquid air trap removes condensable vapors from desorbed gases. A vanadium furnace absorbs non rare gases. Pressure is followed by a Pirani gauge.

Non absorbed gases are pumped out. Hydrogen desorbed from the vanadium is transferred to a charcoal trap at -195°C, then taken to the mass spectrometer line.

2 - On the mass spectrometer the sample, attached to the double inlet system, is compared with a protonium standard ([D/H ratio = 1.0 x 10^{-6}) at the same pressure. Precision (2σ) of ratio measurements is ± 0.1 x 10^{-6} for 20 mm² samples, ± 0.5 x 10^{-6} for 2 mm², and ± 2 x 10^{-6} for 0.5 mm² samples.

II - PRELIMINARY CHECKS.

Terrestrial basalt in contact with protonium gas is used to investigate operating conditions (T, P, treatment time) and contamination.

a) Under 600°C recovery yield of hydrogen is higher than 95%, and protonium in contact with basalt and the line under extraction conditions, is not contaminated by deuterium by more than 0.4 to 0.6 parts per million.

b) Above 600°C the recovery yield drops. At 800°C protonium in contact with the basalt is completely absorbed in twenty minutes.

III - RESULTS.

The gases of a first 235.1 mg aliquot from the sample 14,003,78 (fines) have been desorbed after pumping 1/2 hour at 275°C. Volumes of fractions collected in each temperature interval, and corresponding D/H ratios are given in Table I part 1. The results can be compared to those of [1] and [2]. To investigate further whether the deuterium found can originate from water adsorbed on the fines, by exchange with desorbing hydrogen, two experiments have been carried out.

a) Ordinary water vapor saturated at room temperature was kept in contact 72 hours, with the sample previously outgassed at 800°C.
Protonium is then introduced on the extraction line, the sample heated and the gas sampled under the same conditions as in Table I part I. The deuterium ratio of the fractions collected is less than $3 \times 10^{-6}$. Though, as in the case of basalt, $2/3$ of the last fraction of protonium is absorbed, when heating it up to $800^\circ C$, the remaining third is not more contaminated.

b) A fresh 255.6 mg aliquot of the sample is exposed for 24 hours, at room temperature to water vapor of 700 ppm deuterium content. The sample is then outgassed and analysed as above. Results are given in Table I part II.

**DISCUSSION AND CONCLUSION.**

The first small ($1.1 \, \text{mm}^3$) fraction is not representative because sampled during water elimination. A constant excess of deuterium (5 ppm) is then observed compared to the first experiment. This increase is much smaller than expected contamination from 700 ppm water vapor adsorbed on the fines and exchanging with inbedded hydrogen during desorption.

Further experiments to trace the origin of the deuterium found are underway on samples 14,003 - 14,259 - 10,019. Up to now our experiments show that:

a) No hydrogen can be observed to desorb above $800^\circ C$ as then it reacts rapidly with the fines.

b) The last collected fraction is richer in deuterium than all others. Our line is not the cause of this enrichment unobserved in preliminary tests.

**TABLE I**

<table>
<thead>
<tr>
<th>Part I</th>
<th>Part II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating temperature ($\pm 10^\circ C$)</td>
<td>Heating temperature ($\pm 10^\circ C$)</td>
</tr>
<tr>
<td>275-310</td>
<td>20-275</td>
</tr>
<tr>
<td>410-515</td>
<td>275-445</td>
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<td>435-550</td>
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<td>540-730</td>
<td>535-635</td>
</tr>
<tr>
<td>730-825</td>
<td>635-825</td>
</tr>
<tr>
<td>Total</td>
<td>Total</td>
</tr>
</tbody>
</table>

15 $\mu$ moles/g (excluding first fraction)
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Fig. 1: Sketch of apparatus for extraction of hydrogen
A - Quartz tube containing the sample.
B-D - Liquid nitrogen traps.
C - Pirani gauge.
E - Tube containing vanadium.
F - Line to introduce reference samples.
G - Tube containing charcoal.

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