AN IODINE-XENON STUDY OF LABORATORY-SYNTHESIZED ENSTATITE,
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The I-Xe dating method has never lacked for critics. The method depends upon observing correlated release, in stepwise heating of a neutron-irradiated meteorite sample, of radiogenic $^{129}$Xe and pile-produced $^{129}$Xe. Typically it is found that the ratios $^{129}$Xe/$^{132}$Xe and $^{128}$Xe/$^{132}$Xe are linearly correlated for release temperatures above about 900°C. Such behaviour will result if the trapped xenon present is isotopically homogeneous and if retentively sited iodine and its identically sited $^{129}$Xe decay product are present in fixed ratio. Believers in the method extract chronological information from that ratio and additional import from the ratio $^{129}$Xe/$^{132}$Xe for the trapped component, obtained by extrapolation of the correlation line. Critics of the method have nevertheless implied that the correlations seen may be devoid of chronological significance because iodine and xenon (rich in $^{129}$Xe from earlier iodine decay) may have been indistinguishably trapped during the mineralization. In this case the "isochron" would simply be a mixing line between xenon from those trapped species and an irrelevant xenon component from (e.g.) terrestrial contamination. Other critics have supposed the high-temperature correlation to result from an artifact of the neutron-irradiation or of the low-temperature heating steps (or of both) whereby initially distinguishable iodine and xenon have merged into a component with a single high-temperature release pattern for its xenon which contributes to a mixing line. Indirectly, proponents of the method can meet such criticisms by successful application of the method to samples where the chronologies obtained can be judged on other grounds. There has been recent success of this type in dating silicates from iron meteorites (1) and single inclusions from the Allende meteorite (2). One sample from an iron meteorite (1) has shown an almost perfect correlation from temperatures of 600°C and upwards, an observation which generally weakens any possibility that low-temperature heating is the artifact responsible for the high-temperature correlation.

It has commonly been recognized that there are two more direct approaches to resolving the important questions involved. First would be successful identification and isolation of the iodine-bearing minerals in the meteorites. Thus far progress along this line has been disappointingly slow although there has been substantial progress in the related quest for the sites of the trapped xenon in meteorites (3). The other approach would be laboratory studies directed toward elucidation of the trapping (and release) properties for both iodine (via product xenon) and xenon in meteoritic minerals. The work reported here is a first step in that direction.

Enstatite was synthesized by the reaction

$$2\text{Mg}_3\text{[Si}_2\text{O}_5\text{](OH)}_4 + \text{Mg}_4\text{Si}_6\text{O}_{15}\text{(OH)}_2\cdot6\text{H}_2\text{O} \rightarrow 10\text{MgSiO}_3 + 11\text{H}_2\text{O}$$

Serpentine + Sepiolite + Enstatite + Water

Iodine vapor and $^{124}$Xe tracer were pipetted into an evacuated quartz vial (Figure 1) containing the staring material and maintained at -78°C. After sealing the vial at its constriction it was heated to 1000°C for 10 hours. The vial with its contents was then irradiated for 3 hours in the Lazy Susan position of the Berkeley Triga Mark III reactor at a flux of $4.5 \times 10^{12}$ n/cm²-sec. After irradiation the vial was placed in the quartz crucible
of a mass spectrometer sample system and opened by dropping a weight on the breakoffski. The Xe released from the vial containing the sample was measured at room temperature, 200 and 1000°C. The enstatite was then removed from the vial, wrapped in Al foil, and analyzed for Xe at higher temperatures in a tungsten crucible using RF induction heating.

As performed, the experiment enables determining the behaviour of three Xe components. Firstly, 128Xe results from neutron activation of 127I and therefore corresponds to Xe derived from I partitioned between the gas phase and the enstatite. Secondly, 124Xe corresponds to trapped xenon incorporated in enstatite from the gas phase by solution. Finally, a 126Xe component results from energetic implantation of recoiling 126I nuclei from the reaction 127I(n,2n)126I. The threshold for this reaction is so high that the recoiling compound nucleus has a minimum energy of about 1000eV, which corresponds closely to that for Xe ions in the present day solar wind.

A number of conclusions can be drawn from the experimental results summarized in Table 1. Since 128Xe/124Xe ratios increase with increasing temperature it is evident that 128Xe is more retentively bound than 124Xe dissolved in enstatite. In contrast, since 128Xe/126Xe ratios decrease with increasing temperature, 128Xe is less retentively bound than 126Xe acquired by implantation. Consequently the thermal release behaviour of trapped Xe components depends on the mechanism by which it was incorporated. This is an expected but not previously demonstrated result. In both cases variations in isotopic ratios of approximately a factor of 5 are found. This degree of variation is similar to those seen typically in high temperature gas fractions from meteorites. Of particular significance, and the main result of the experiment, is that trapped xenon and both iodine-derived xenon components are each released differently from the sample. In the case of 128Xe, where the recoil energy in the (n,γ) reaction is small, this fact implies that trapped iodine and xenon occupy different sites within the mineral, even after substantial heating of the lattice. This observation is at variance with both the criticisms of the dating method.

Very preliminary distribution coefficients for I and Xe between a gas phase and enstatite at 1000°C can be derived by assigning to the gas phase those Xe components released from the heatings in quartz and assigning to the dissolved phase those Xe components released from the Al-wrapped samples. Values obtained are 3.3 x 10^{-4} and 1.3 x 10^{-2} ccSTP/gm-atm for Xe and I respectively. The Xe value is higher than would be derived by extrapolation of Kirsten’s (5) measurements for He, Ne, and Ar solubilities for an enstatite melt at 1500°C (0.6 x 10^{-4}). In our case it is not certain that equilibrium solubilities have been obtained since path independence and reproducibility have not yet been demonstrated.

The distribution coefficients for I and Xe differ by a factor of 40, which is far less than the difference in depletion factors between cosmic and meteoritic abundances for those elements. Such a result is not unexpected – processes other than differing mineral solubilities at high temperatures probably determined the relative loss of iodine and xenon in the evolution of
In performing the preliminary experiment we encountered a number of difficulties in sample preparation that would require improvement in any extended study. The measured I and Xe contents of the vial were not those predicted from P-V-T relations. We found excess I$_2$ in the vial and that the I$_2$ vapor had a high affinity for nucleation during the procedure. I$_2$ was also preferentially adsorbed on the fine grained starting material and not returned to the gas phase without heating the sample to 150°C. Contrariwise a deficiency in Xe content was found in the vial and could be attributed to adsorption of Xe on portions of the sample system or in the I$_2$ reservoir during the aliquoting procedure.

In a separate experiment we found that Xe does not exhibit ideal gas behaviour in the presence of I$_2$. Equal quantities of Xe were pipetted into two evacuated pyrex ampoules at -78°C, one of which contained I$_2$. The ampoule containing I$_2$ was found to have three times the Xe abundance of the other. Apparently the presence of I$_2$ on glass surfaces substantially alters the adsorption properties for Xe. While these effects do not alter the conclusions stated in this paper, it is important to note that the maintenance of predicted abundances for these volatile components in closed systems is a non-trivial experimental problem.

### Table 1

<table>
<thead>
<tr>
<th>Temp. °C</th>
<th>$^{128}$Xe x $10^{-12}$ cm$^3$STP</th>
<th>$^{126}$Xe x $10^{-15}$ cm$^3$STP</th>
<th>$^{128}$Xe/$^{124}$Xe</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>5.61 x 10$^{-10}$</td>
<td>9.95 x 10$^{-8}$</td>
<td>177</td>
</tr>
<tr>
<td>200</td>
<td>2.35 x 10$^{-12}$</td>
<td>3.01 x 10$^{-10}$</td>
<td>128</td>
</tr>
<tr>
<td>1000</td>
<td>1.20 x 10$^{-12}$</td>
<td>2.51 x 10$^{-9}$</td>
<td>2092</td>
</tr>
</tbody>
</table>

### Notes and References:

3. Lewis, R.S. et al. (1975) Science 190, 1251.