Galactic cosmic ray-produced $^{129}\text{Xe}$ and $^{131}\text{Xe}$ excesses in troilites of the Cape York iron meteorite

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Abstract–The flux of galactic cosmic rays (GCR) in the solar system appears to change with time. Based on the abundances in iron meteorites of cosmogenic nuclides of different half lives, Lavielle et al. (1999) found that the GCR flux increased in recent times (<100 Ma) by about 38% compared to average flux in the past 150 Ma to 700 Ma ago. A promising technique for calibrating the GCR flux during the past ~50 Ma, based on the $^{129}\text{I}$ and $^{129}\text{Xe}$ pair of nuclides, was discussed earlier (Marti 1986; Murty and Marti 1987). The $^{129}\text{I}$-$^{129}\text{Xe}$ chronometer provides a shielding-independent system as long as the exposure geometry remained fixed. It is especially suitable for large iron meteorites (Te-rich troilite) because of the effects by the GCR secondary neutron component. Although GCR-produced Xe components were identified in troilites, several issues require clarifications and improvements; some are reported here. We developed a procedure for achieving small Xe extraction blanks which are required to measure indigenous Xe in troilites. The $^{129}\text{Xe}$ and $^{131}\text{Xe}$ excesses ($^{129}\text{Xe}_n$, $^{131}\text{Xe}_n$) due to neutron reactions in Te are correlated in a stepwise release run during the troilite decomposition. Our data show that indigenous Xe in troilite of Cape York has isotopic abundances consistent with ordinary chondritic Xe (OC-Xe), in contrast to a terrestrial signature which was reported earlier. Two methods are discussed which assess and correct for an interfering radiogenic $^{129}\text{Xe}$ component from extinct $^{129}\text{I}$. The corrected $^{129}\text{Xe}_n$ concentration in troilite D4 of Cape York yields a cosmic ray exposure (CRE) age of 82 ± 7 Ma consistent, within uncertainties, with reported data (Murty and Marti 1987; Marti et al. 2004).

INTRODUCTION

Galactic cosmic rays provide information on the discrete energetic particle sources in the galaxy and are thought (e.g., Axford 1981) to derive from shock acceleration in supernovae explosions, as they travel through the surrounding interstellar gas. If, as at present, our solar system is located in galactic spiral arms with star formation regions (OB associations), an increased GCR flux can be expected. The stellar content of OB associations within 1 kpc from the Sun was reported by De Zeeuw et al. (1999), in a study of the evolution of nearby young stellar groups in star-forming regions. The OB associations are unbound moving groups of stars which can be detected kinematically because of their small internal velocity dispersion.

Cosmochemical approaches to study the variability of the fossil GCR flux were proposed by several workers (e.g., Voshage 1962; Schaeffer et al. 1981; Lavielle et al. 1999) based on production rate calibrations of cosmogenic nuclides in iron meteorites. The proposed methods use abundance ratios of GCR-produced radionuclides of varying half-lives, coupled with stable products, which integrate over the time of GCR exposure. These chronometers are expected to show variations in integral exposure times, but geometric complexities during the irradiation in general were neither studied nor delineated. Lavielle et al (1999) reported an evaluation of the constancy of the GCR flux over the last 1 Ga. Their inferred average production rates of $^{36}\text{Cl}$ and $^{36}\text{Ar}$ in the time interval 150 to 700 Ma before the present were ~28% lower than present-day values of these production rates. Their study does not constrain the time of flux increase, nor the possibility of a cyclic variation. A possible recent (<100 Ma) flux increase may be assessed by a GCR-produced nuclide of appropriate half-life, such as $^{129}\text{I}$ which is produced by neutron reactions on Te and its $t_{1/2} = 16$ Ma is ideal for monitoring changes in the GCR flux over the last 50 Ma. The proposed $^{129}\text{I}$-$^{129}\text{Xe}_n$ chronometer (Marti 1986) which is based on the pair $^{129}\text{I}$ and its integrating stable decay product...
\[ F(T) \equiv \frac{\lambda_{129} T}{1 - e^{-\lambda_{129} T}} = 1 - \frac{129\text{Xe}_T}{129\text{I}} \text{,} \]

where \( \lambda_{129} = 4.41 \times 10^{-8} \text{ a}^{-1} \) is the decay constant of \(^{129}\text{I}\), and \(^{129}\text{Xe}_T\) represents the neutron-produced excess concentration. The resolution of several Xe components in troilite requires that samples are analyzed using a step-heating technique for progressively higher temperatures until troilite decomposition is complete. As the release systematics of the components apparently differ, partial component separation is achieved by this procedure. The isotopic abundances of indigenous Xe have to be known for the determination of isotopic excesses \(^{129}\text{Xe}_n\) and \(^{131}\text{Xe}_n\) and also of \(^{128}\text{Xe}_n\) for the correction of interfering radiogenic \(^{129}\text{Xe}_e\) component.

**EXPERIMENTAL TECHNIQUES**

In an earlier study of troilite from Cape York (Murty and Marti 1987) a rather good correlation between neutron-produced excesses \(^{129}\text{Xe}_e\) and \(^{131}\text{Xe}_n\) was observed, but the authors ignored a possible contribution by radiogenic \(^{129}\text{Xe}_e\). In their study the indigenous Xe isotopic abundances in some temperature steps did not agree with abundances in terrestrial Xe which was reported to be the trapped component in troilite (Hwuang and Manuel 1982). The experiments were carried out in a double-walled quartz system using an external heater, permitting inspections of the progress of the decomposition and of the reaction products. The setup revealed sulfur reactions with quartz. One troilite chip in the storage system accidentally fell out of the Pt container boat, while being transferred to the double walled quartz section for heating. While this chip was in contact with quartz of the inner wall the decomposition was monitored, as well as the products and the gas release. At 900 °C, it was clear that the sample had reacted with the quartz wall and released atmospheric Xe present in quartz. The measured concentration was \(~20\times\) higher than the expected trapped Xe abundance in Cape York troilite.

In order to achieve low Xe blanks, it was necessary to prevent reactions of the hot sulfur vapor with the quartz extraction system. We developed techniques which efficiently remove sulfur vapors produced in the decomposition of troilite. Figure 1 shows the experimental setup used for the in vacuo removal of sulfur, which consists of a Pt-covered spool with Fe wire wrapped on it. Low blanks were achieved by catching sulfur vapors during decomposition on the hot pre-degassed iron wire coil. The Fe-wire coil was heated with an external heater, together with the troilite sample in Pt foil boats, while the Fe-wire maintained a temperature gradient of a few hundred degrees below the temperature of the troilite sample. The formation of FeS on this catcher wire visibly eliminated S deposition and reactions on quartz of the extraction system at temperatures >1000 °C. The elimination of S deposition on the extraction system drastically reduced the Xe blank. An additional improvement was achieved by reducing S-vapor diffusion in the extraction system by adjusting one end of the Fe wire spool (low temperature side) to match the inside diameter of the extraction system. Although the observed Xe blanks increased with temperature, for a 30 min heating step at 1100 °C they were only about \(5 \times 10^{-15} \text{ cm}^3 \text{ STP}\) of \(^{132}\text{Xe}\). The \(~2\text{ cm} \) long spool-assembly is moved into the extraction system. The elimination of S deposition on quartz of the inner wall was monitored, as well as the products and the gas release. At 900 °C, it was clear that the sample had reacted with the quartz wall and released atmospheric Xe present in quartz. The measured concentration was \(~20\times\) higher than the expected trapped Xe abundance in Cape York troilite.
RESULTS AND DISCUSSION

Indigenous Xe in Troilite

The Xe concentration ($^{132}\text{Xe} = (1.49 \pm 0.10) \times 10^{-12} \text{cm}^3 \text{STP/g}$) in Cape York troilite D4 and the isotopic abundances (uncertainties at 95% confidence levels) are given in Table 1 and neutron-produced and radiogenic Xe components in Table 2. The measured concentration in troilite D4 is similar to values observed by Hwaung and Manuel (1982) and twice as large as those observed by Murty and Marti (1987) in troilites from other shielded locations in Cape York. The Xe isotopic abundances in temperature steps up to 700 °C are dominated by Xe of atmospheric composition, but above 900 °C the indigenous Xe component is observed to be consistent with the isotopic composition of OC-Xe (Lavielle and Marti 1992) for all isotopes except $^{128}\text{Xe}$, $^{129}\text{Xe}$, and $^{131}\text{Xe}$ (Fig. 2). Variable isotopic abundances are observed for $^{128}\text{Xe}$ but the $^{128}\text{Xe}/^{130}\text{Xe}$ ratios in the 1100° and 1110 °C steps are consistent with the OC-Xe signature. The identification of an indigenous OC-Xe component in troilite is at variance with the Xe reported by Hwaung and Manuel (1982) who observed a terrestrial composition. The agreement of $^{124}\text{Xe}/^{130}\text{Xe}$ and $^{126}\text{Xe}/^{130}\text{Xe}$ ratios with OC-Xe data also shows that the GCR-spallation component in troilite is very small. A very small spallation component is observed in our Ar data (Table 3) which is nearly two orders of magnitude lower than spallation Ar expected for unshielded locations for an iron with this CRE age. The observed very small GCR-spallation component and poorly known production rates for spallation Ar in heavily shielded locations may illustrate the value of using Xe components due to secondary neutron reactions, for reliable CRE ages.

Xe Components Due to Secondary Neutron Reactions

It is not clear whether the isotopic abundances of Xe released in the 900 °C step, although consistent with OC-Xe within error limits, still may contain a small contaminating atmospheric component. We do not use the 900 °C data for our component analysis. We note that Xe fractions from temperature steps 950°, 1000°, and 1050 °C all show small excesses at $^{128}\text{Xe}$ (Table 1, Fig. 3), indicating the presence of a neutron-produced $^{127}\text{I} (n, \gamma, \beta^-)$ $^{128}\text{Xe}_n$ component and, therefore, that I is present in troilite. This excess implies that $^{129}\text{Xe}$-excesses may be affected by radiogenic $^{129}\text{Xe}$ from extinct $^{129}\text{I}$ in troilites. This component was considered to be unimportant by Murty and Marti (1987). The Xe isotopic data for the 1100° and 1110 °C fractions (Table 1) are consistent with OC-Xe, except those affected by neutron reactions on Te. The excesses $^{129}\text{Xe}_n$ and $^{131}\text{Xe}_n$ are well correlated and permit the evaluation of secondary neutron energies (Fig. 4). Murty and Marti (1987) did calculate expected correlations for thermal and epithermal neutrons and showed that mainly epithermal neutrons are responsible for the observed excesses in Cape York troilite. They used thermal cross-sections and the resonance integrals of Browne and Berman (1973), as well as (n, 2n) reaction data from...
Table 1. Isotopic abundances of Xe in Cape York troilite (1.0581 g) D4, normalized to $^{130}$Xe. Concentrations are given in units of $10^{-12}$ cm$^3$ STP/g and 2σ uncertainties are given for isotope ratios.

<table>
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<th>Temp (°C)</th>
<th>$^{130}$Xe (10$^{-12}$ cm$^3$ STP/g)</th>
<th>$^{124}$Xe/$^{130}$Xe</th>
<th>$^{126}$Xe/$^{130}$Xe</th>
<th>$^{128}$Xe/$^{130}$Xe</th>
<th>$^{129}$Xe/$^{130}$Xe</th>
<th>$^{130}$Xe</th>
<th>$^{131}$Xe/$^{130}$Xe</th>
<th>$^{132}$Xe/$^{130}$Xe</th>
<th>$^{134}$Xe/$^{130}$Xe</th>
<th>$^{136}$Xe/$^{130}$Xe</th>
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<tr>
<td>&gt; 700</td>
<td>0.241</td>
<td>2.72</td>
<td>2.51</td>
<td>53.9</td>
<td>1119.8</td>
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<td>609.3</td>
<td>618.7</td>
<td>234.0</td>
<td>198.4</td>
</tr>
<tr>
<td>OC-Xe</td>
<td>2.86</td>
<td>2.55</td>
<td>51.1</td>
<td>642.0</td>
<td>=100</td>
<td>506.0</td>
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<td>1.1</td>
<td>0.5</td>
<td>0.4</td>
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</table>
Proton reactions on $^{130}\text{Te}$ and $^{128}\text{Te}$ may also produce $^{129}\text{I}$, but these are not important in heavily shielded locations, as discussed earlier. We use the $^{129}\text{Xe}/^{130}\text{Xe}$ ratio of OC-Xe for indigenous $^{129}\text{Xe}$ in troilite D4, although it is possible that the $^{129}\text{Xe}/^{130}\text{Xe}$ ratio in troilite was slightly evolved at the time of system closure due to decay of extinct $^{129}\text{I}$. We note that a Xe-rich inclusion in troilite studied by Murty and Marti (1987) showed a slightly evolved $^{129}\text{Xe}/^{130}\text{Xe}$ ratio, but this excess may also be interpreted as due to reactions on Te isotopes. We will explore this option later.

### Iodine in Troilite and Radiogenic $^{129}\text{Xe}$ from Extinct $^{129}\text{I}$

As was shown by Murty and Marti (1987), excesses $^{129}\text{Xe}_n$ and $^{131}\text{Xe}_n$ are well correlated. On the other hand, these authors identified an unusual halogen containing inclusion in their troilite sample of Cape York. They estimated that Cl and I concentrations in this inclusion were ~43 ppm and 0.8 ppb, respectively, and that some of their observed $^{36}\text{Ar}$ and $^{125}\text{Xe}$ excesses may be due to neutron capture reactions. These authors inferred an initial $^{129}\text{I}/^{127}\text{I}$ ratio of ~0.84 × 10$^{-4}$, based on the assumption that the total excess $^{125}\text{Xe}$ in this inclusion represents the radiogenic component. This inferred ratio, although uncertain, is consistent with reported extinct I-Xe chronological data in iron meteorites (Niemeyer 1979). We can correct the implied superimposed $^{125}\text{Xe}$, radiogenic component by using an unknown but uniform ratio $R = ^{129}\text{Xe}_n/^{125}\text{Xe}_n$. Figure 2 shows that neutron-produced excesses $^{128}\text{Xe}_n$ are differentially released and are observed in the early releases (950 to 1050 °C), but are absent in the temperature fractions 1100° and 1110 °C (Fig. 3). These latter data require no radiogenic $^{129}\text{Xe}_n$ corrections. Therefore, a straightforward method to obtain the total neutron-produced excess $^{129}\text{Xe}_n$ is to use the observed ratios $^{129}\text{Xe}_n/^{131}\text{Xe}_n$ in these two temperature steps and to calculate $^{129}\text{Xe}_n$ excesses in all steps from measured excess $^{131}\text{Xe}_n$ data and to sum these to get the total excess. An alternative which may be analytically more satisfactory and illustrate the uncertainties is to correct measured $^{129}\text{Xe}_n$ data. We do not know the ratio $R$ but we may infer a range of values which correct the $^{129}\text{Xe}_n$ data onto the correlation line in all temperature steps. Figure 4 shows results in three panels for corrected Te (n, γ)-produced excesses: correlations $^{129}\text{Xe}_n$ versus $^{131}\text{Xe}_n$ with $^{129}\text{Xe}_n$ corrections carried out for three different values $R = 10, 14, \text{and } 18$, selected from a spectrum of used $R$ values. We note that the middle panel with $R = 14$ represents a rather good fit, except for temperature steps 900° and 1150° (discussed below), while the panel for $R = 10$ may be considered to represent lower limits and the panel for $R = 18$ upper limits for the applied corrections. Both methods yield the same $^{129}\text{Xe}_n$ totals within uncertainties.

### Corrected $^{129}\text{Xe}_n$-Data and CRE Age

The concentration of $^{129}\text{Xe}_n$ listed in Table 2 is calculated for a radiogenic $^{129}\text{Xe}_n$ correction $R = 14 \pm 4$ (excluding the 950° and 1150 °C fractions). The correlation (Fig. 4) shows that Te-reaction products are dominated by epithermal neutron reactions, and support the reported conclusion (Murty and Marti 1987). The small excesses in the 950 °C data (as well as in the residual gas released at 1150 °C) suggest small
“unsupported” excesses of $^{129}\text{Xe}$ are present which are not related to Te reactions; these minor excesses are not included in the calculation of the $^{129}\text{Xe}$ concentration.

As mentioned earlier, we should not ignore the possibility that the ratio $^{129}\text{Xe}/^{130}\text{Xe}$ at the time of system closure of troilite might have been slightly evolved from an initial OC abundance. This option would slightly reduce the amount of unsupported excess $^{129}\text{Xe}$, but this evidence is questionable and contradicted. First, the $^{129}\text{Xe}/^{132}\text{Xe}$ ratio observed in a special troilite inclusion (Murty and Marti 1987) exceeds the OC-value by only 4%, although this ratio refers to a halogen-rich inclusion. Second, we have looked at simulated $^{129}\text{Xe}_{\text{en}}$-$^{131}\text{Xe}_{\text{en}}$ correlations resulting from excesses in indigenous $^{129}\text{Xe}/^{130}\text{Xe}$ ratios. The $^{129}\text{Xe}_{\text{en}}$-$^{131}\text{Xe}_{\text{en}}$ correlation break down with a $^{129}\text{Xe}_{\text{en}}$ excess of 7% above the OC-value. There is no evidence for an evolved indigenous $^{129}\text{Xe}/^{130}\text{Xe}$ ratio, and we adopt the OC-value in the calculation of $^{129}\text{Xe}_{\text{en}}$ excesses. A value $^{129}\text{Xe}_{\text{en}} = 26.6 \pm 1.8$ million atoms/g is calculated (Table 2) in troilite D4.
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which when coupled to the $^{129}$I concentration (Nishiizumi et al. 1985 and personal communication) slightly lowers the preliminary CRE age of Cape York reported earlier (Marti et al. 2004) to a value of 82 ± 7 Ma.

CONCLUSIONS

1. Our results show no evidence for indigenous Xe of terrestrial atmospheric composition as reported by Hwaung and Manuel (1982). The isotopic abundances of Xe in troilite of Cape York are consistent with OC-Xe. Excesses are observed on $^{128}$Xe, $^{129}$Xe and $^{131}$Xe.

2. Extraction blanks are successfully reduced by an order of magnitude by catching S vapors from decomposing troilite on hot Fe wires and by eliminating contacts of the troilite with the quartz extraction system. The low blanks permit identifications of the indigenous and neutron-produced components.

3. The $^{124}$Xe and $^{126}$Xe isotopic abundances show that the spallation component in Cape York troilite is very small. The excesses on $^{129}$Xe and $^{131}$Xe are mostly due to GCR secondary neutron reactions while excesses $^{128}$Xe can be used as a monitor for reactions on I. They also permit corrections to be applied for interfering radiogenic $^{129}$Xe from extinct $^{129}$I.

4. The neutron-produced excesses $^{129}$Xe and $^{131}$Xe show a linear correlation, and the slope identifies GCR epithermal neutrons as the prevalent source of particle reactions in Cape York troilite.

5. The reaction excess $^{131}$Xe serves as a suitable monitor of GCR reactions and permits the calculation of the total excess $^{129}$Xe. The corrected $^{129}$Xe concentration slightly revises earlier reported (Marti et al. 2004) values for the of the CRE age to 82 ± 7 Ma, which agrees within uncertainties with a value of 94 ± 20 Ma for Cape York reported by Murty and Marti (1987). For an evaluation of...
the constancy of the GCR flux this CRE age needs to be cross-calibrated with ages based on radionuclides with different half-lives.

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Editorial Handling—Dr. Ian Lyon

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