

^{58}Fe AND ^{54}Cr IN EARLY SOLAR SYSTEM MATERIALS

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ABSTRACT

Stepwise dissolution of primitive meteorites exhibits large ^{54}Cr anomalies but no collateral effects on ^{58}Fe and ^{48}Ca , two other neutron-rich nuclides from the iron peak. These results suggest that ^{54}Cr must have been produced in particular zones of the rare Type Ia supernovae or that ^{48}Ca and/or ^{58}Fe were produced together in Type II supernovae and were chemically separated into some mineral phase that favors Cr over Ca and Fe, and it is the dissolution properties of that phase that is driving the isotopic effect in leaching. The recent findings of nanometer-size oxide grains with very large ^{54}Cr excesses favored the latter scenario for the origin of the mono-isotopic Cr isotopic effect. In addition, the absence of isotopic variations in the $^{58}\text{Fe}/^{54}\text{Fe}$ ratio at the mineral scale confirms that the short-lived nuclide ^{60}Fe ($T_{1/2} = 2.62$ Myr) was homogeneously distributed to a less than 15% dispersion in the early solar nebula.

Key words: astrochemistry – minor planets, asteroids: general – nuclear reactions, nucleosynthesis, abundances – protoplanetary disks

Online-only material: color figures

1. INTRODUCTION

CI carbonaceous chondrites are among the most primitive samples of our solar system's materials available for laboratory examinations. They have incorporated presolar solids relatively unmodified by nebular processes. For example, carbonaceous chondrites are hosts for several types of circumstellar grains (diamond, graphite, silicon carbide, corundum, silicon nitride, and silicates; Anders & Zinner 1993; Zinner 1997; Zinner et al. 2011; Nguyen & Zinner 2004; Nagashima et al. 2004). These grains are identified as presolar because of the very large isotopic anomalies they carry which are attributable to nucleosynthesis in specific stellar environments. The known types of presolar grains constitute only a very small fraction of total meteorite mass and their identification and characterization relies on their efficient isolation from other meteoritic constituents. The first grains discovered were thermally refractory and very resistant to chemical reagents that dissolve most of the other phases in the meteorite; the latter feature in particular has been crucial in their successful isolation. But there are also cases in which presolar materials are carried in phases that are not chemically resistant, as first revealed in simple acid leaching of whole rock meteorites (Rotaru et al. 1992; Podosek et al. 1997; Trinquier et al. 2007), and more recently identified in cosmic dust particles (Messenger et al. 2002) and later in carbonaceous chondrites (Nguyen & Zinner 2004; Nagashima et al. 2004; Mostefaoui & Hoppe 2004).

The different leaching fractions of Orgueil (CI1 chondrite) show large isotopic anomalies in ^{54}Cr (Rotaru et al. 1992; Podosek et al. 1997; Trinquier et al. 2007). Initial treatment with acetic and nitric acids was found to dissolve most of the Cr (and other cations as well); the Cr thus dissolved is nearly uniform in composition but deficient (with respect to the normal composition) in ^{54}Cr by some 5–6 ϵ (per ten thousand). Further treatments with hydrochloric acid and then other reagents liberate the remainder of the Cr, which can have variable compositions, mostly with excess ^{54}Cr (up to more than 200 ϵ).

There is no known way to generate such isotopic variations within the solar system; this effect must be interpreted in terms of isotopic anomalies, i.e., non-homogenization of distinct presolar nucleosynthetic components. Recently, Dauphas et al. (2010) and Qin et al. (2011) have shown presolar spinel grains as possible carrier phases for these anomalies.

Dense Type Ia supernovae provide the neutron-rich, low-entropy environment thought responsible for the neutron-rich iron-group isotopes (e.g., Meyer et al. 1996). Collateral isotopic effects on other elements associated with the ^{54}Cr enrichment would provide compelling evidences to refine the nucleosynthetic origin of the ^{54}Cr anomalies. For example, no Ca isotopic anomalies have been found in leachates from the CI1 chondrite Orgueil containing large ^{54}Cr anomalies (Moynier et al. 2010). This absence of ^{48}Ca anomalies suggests that the ^{54}Cr anomalies must be produced either in massive stars during *s*-process nucleosynthesis without accompanying ^{48}Ca or in particular zones in a rare Type Ia supernovae (Moynier et al. 2010). On the other hand, ^{50}Ti enrichment is correlated with ^{54}Cr in Orgueil leachates (Trinquier et al. 2009) which has led the authors to suggest a common carrier phase for these two anomalies. However, the difference in volatility between Cr ($T_c = 1296$ K) on the one hand and the refractory elements Ti ($T_c = 1582$ K) and Ca ($T_c = 1517$) on the other hand (see Lodders 2003 for T_c , the 50% condensation temperature) may lead to fractionation and decoupling between these elements during thermal events within the solar system. Therefore, it would be very important to compare the ^{54}Cr excesses with another element with similar volatility such as Fe ($T_c = 1334$ K).

Fe is composed of four stable isotopes: ^{54}Fe (5.84%), ^{56}Fe (91.76%), ^{57}Fe (2.12%), and ^{58}Fe (0.28%). The different Fe isotopes are formed by different nucleosynthetic mechanisms in stars. ^{54}Fe , ^{56}Fe , and ^{57}Fe are formed by nuclear statistical equilibrium (NSE) while the neutron-rich ^{58}Fe (as well as other neutron-rich isotopes from the Fe peak) are formed by neutron-rich version of the equilibrium process in Type Ia or Type II supernovae (Clayton 2003). The neutron-rich ^{60}Fe

Table 1
Fe and Cr Isotopic Compositions of Leaching Meteorites (See the Text for Details of the ϵ Notation)

Sample Name	Type	Treatment	$\epsilon^{56}\text{Fe}^a$ ($\pm 2\text{SE}$)	$\epsilon^{58}\text{Fe}^a$ ($\pm 2\text{SE}$)	$\epsilon^{57}\text{Fe}^b$ ($\pm 2\text{SE}$)	$\epsilon^{58}\text{Fe}^b$ ($\pm 2\text{SE}$)	n^c	$\epsilon^{53}\text{Cr}^d$ ($\pm 2\text{SE}$)	$\epsilon^{54}\text{Cr}^d$ ($\pm 2\text{SE}$)
Semarkona (536 mg)									
S-I-2	LL3	50% Acetic acid, RT ^e	-0.04 ± 0.21	-0.75 ± 1.79	0.06 ± 0.31	-0.67 ± 1.96	5	0.99 ± 0.16	-6.2 ± 0.4
S-I-3	LL3	25% HNO ₃ , RT	-0.10 ± 0.08	0.04 ± 0.79	0.15 ± 0.11	0.24 ± 0.76	5	0.44 ± 0.15	-1.4 ± 0.3
S-I-4	LL3	6N HCl RT	-0.02 ± 0.09	0.67 ± 1.25	0.02 ± 0.13	0.70 ± 1.40	5	0.14 ± 0.15	-3.4 ± 0.3
S-I-5	LL3	6N HCl 40°C	-0.06 ± 0.15	-0.48 ± 0.76	0.09 ± 0.23	-0.36 ± 0.75	5	0.19 ± 0.23	4.4 ± 0.5
S-I-6	LL3	6N HCl 80°C	0.07 ± 0.15	3.5 ± 0.3
S-I-7	LL3	Conc. HF/HCl 100°C	-0.19 ± 0.14	0.68 ± 0.55	0.28 ± 0.21	1.06 ± 0.71	5	0.38 ± 0.15	1.0 ± 0.3
S-I-8	LL3	Conc. HF/HNO ₃ RT	0.03 ± 0.09	0.50 ± 0.78	-0.05 ± 0.14	0.44 ± 0.80	5	-0.15 ± 0.15	-0.5 ± 0.3
Orgueil IV (59.5 mg)									
O-IV-2	CI1	50% Acetic acid, RT	-0.22 ± 0.10	-1.45 ± 2.14	0.33 ± 0.15	-1.01 ± 2.11	7	1 ± 1	-4 ± 2
O-IV-3	CI1	25% HNO ₃ , RT	-0.99 ± 1.73	0.35 ± 0.70	0.19 ± 0.36	0.48 ± 0.81	7	0 ± 1	-5 ± 2
O-IV-4	CI1	6N HCl RT	0.07 ± 0.13	0.69 ± 0.80	-0.06 ± 0.20	-0.15 ± 0.64	8	0 ± 1	-5 ± 2
O-IV-5	CI1	6N HCl 40°C	0.16 ± 0.28	-0.43 ± 2.00	-0.24 ± 0.41	-0.74 ± 1.89	7	0 ± 1	8 ± 2
O-IV-6	CI1	6N HCl 80°C	-1 ± 1	71 ± 2
O-IV-7	CI1	9N HCl 80°C	-2 ± 2	98 ± 4
O-IV-8	CI1	Conc. HF/HCl 100°C	-0.18 ± 0.30	-2.63 ± 4.33	0.27 ± 0.45	-2.27 ± 4.14	4	-1 ± 2	33 ± 2
Murchison II (550 mg)									
M-II-2	CM2	50% Acetic acid, RT	-0.07 ± 0.13	-0.48 ± 1.06	0.11 ± 0.20	-0.34 ± 1.03	6	0.3 ± 0.3	-15.5 ± 0.7
M-II-3	CM2	25% HNO ₃ , RT	-0.05 ± 0.15	0.37 ± 1.24	0.08 ± 0.22	0.48 ± 1.20	6	0.4 ± 0.4	-2.5 ± 0.4
M-II-5	CM2	6N HCl RT	-0.31 ± 0.20	-0.31 ± 0.91	0.47 ± 0.30	0.31 ± 0.98	5	0.0 ± 0.4	14.5 ± 1.0
M-II-6	CM2	6N HCl 40°C	0.1 ± 0.5	34.2 ± 1.2
M-II-7	CM2	6N HCl 80°C	-0.29 ± 0.28	-0.10 ± 1.01	0.43 ± 0.42	0.47 ± 1.24	7	0.1 ± 0.4	21.1 ± 0.7
BCR-2	(basalt)	(Bulk)	-0.04 ± 0.15	0.21 ± 1.48	0.06 ± 0.22	0.29 ± 1.60	5
BIR-1	(basalt)	(Bulk)	-0.07 ± 0.22	0.37 ± 1.48	0.10 ± 0.28	-0.20 ± 1.92	8
BHVO-2	(basalt)	(Bulk)	-0.05 ± 0.07	-1.03 ± 0.89	0.02 ± 0.13	-1.55 ± 1.47	7

Notes.

^a $x\text{Fe}/^{54}\text{Fe}$ normalized to $^{57}\text{Fe}/^{54}\text{Fe} = 0.362566$. $x = 56$ and 58 .

^b $x\text{Fe}/^{54}\text{Fe}$ normalized to $^{56}\text{Fe}/^{54}\text{Fe} = 15.69786$. $x = 57$ and 58 .

^c Number of sample measurements.

^d $x\text{Cr}/^{52}\text{Cr}$ normalized to $^{50}\text{Cr}/^{52}\text{Cr} = 0.051859$. $x = 53$ and 54 .

^e Room temperature.

($T_{1/2} = 2.62$ Myr) is produced by neutron capture in Type II supernovae or asymptotic giant branch (AGB) stars. However, the probability of having both a passing AGB star and a molecular cloud is very low (Kastner & Myers 1994) and AGB stars are usually not considered as viable sources of the live ^{60}Fe in the early solar system (Huss et al. 2009). Early measurements of Fe isotopic ratios for most chondritic materials yield a normal Fe composition (at a resolution of ≈ 0.3 ϵ -units, 2 standard error, 2SE; Tang et al. 2009; Dauphas et al. 2008, 2004), except for effects in rare FUN inclusions that contain Fractionated isotopic compositions and Unusual Nuclear isotopic effects (Völkering & Papanastassiou 1989). Here, we report the relative isotopic abundances of Fe and Cr isotopes in leaching CI1 chondrite Orgueil, CM2 chondrite Murchison, and LL3 chondrite Semarkona to search for possible collateral effects and document the Cr isotopic composition of leachates from ordinary chondrites. Another motivation for this work is to use the variability of the ^{58}Fe to evaluate the extent of the heterogeneity within the solar nebula of the extinct nuclide ^{60}Fe ($T_{1/2} = 2.62$ Myr) which is formed in the same stellar environments.

2. SAMPLES AND ANALYTICAL PROCEDURES

In keeping with the nomenclature system used by Podosek et al. (1997), each starting whole rock sample is designated by a letter and a Roman numeral. Letters O, M, and S designate Orgueil, Murchison, and Semarkona, respectively. Samples processed for this work are O-IV, M-II, and S-I for Cr and Fe isotopes. Successive leaching fractions of each whole rock

sample are further identified by an Arabic numeral following the Roman numeral (e.g., Table 1).

2.1. Sample Treatments

All starting whole rock samples (see Table 1 for masses of starting materials) were crushed and then subjected to a series of leaches in different reagents. After each leaching step the sample was centrifuged and the supernatant decanted by pipette; the sample was then washed with water, centrifuged again, and similarly decanted. The washing procedure was repeated twice more, and all supernatants were combined to form the solution for that leaching step. In most cases these solutions were split, part being taken for the isotopic analysis and part reserved for other uses. In a few cases, a very small fraction of the solid residue was also removed for other studies.

The sequences of reagents applied to the various samples were similar but not identical. In general, the first reagent was 50% acetic acid. The treatment after the acetic acid was 25% nitric acid (HNO₃), then multiple steps with hydrochloric acid (HCl), finally further with hydrofluoric acid (HF) to obtain as complete a dissolution as possible.

Since the principal interest in the further analyses herein reported was constraining the nature of the HCl-soluble carrier phase with the largest excesses of ^{54}Cr , the Murchison samples were not processed beyond HCl treatment.

2.2. Isotopic Measurements

Prior to the mass-spectrometric analysis, Cr and Fe were purified by ion-exchange chromatography following procedures

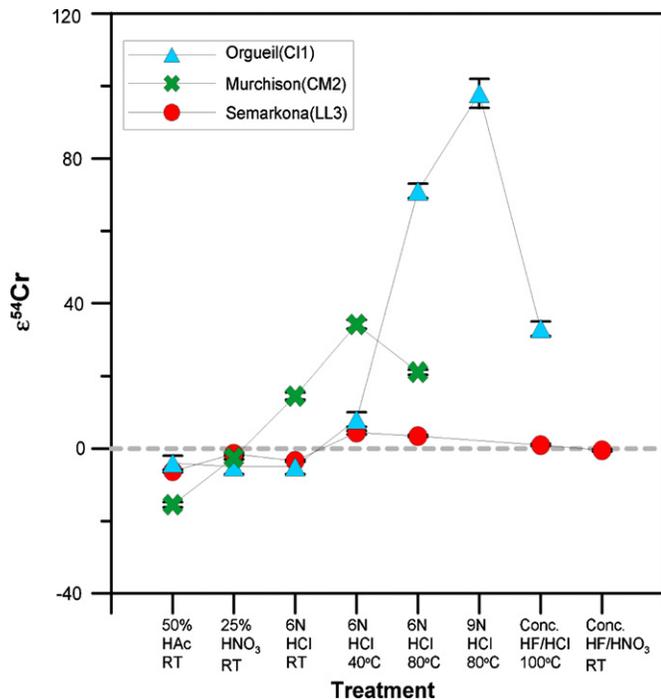


Figure 1. $\epsilon^{54}\text{Cr}$ in leaching fractions from Orgueil, Murchison, and Semarkona. Please refer to Table 1 for the exact data. HAc stands for acetic acid and RT for room temperature.

(A color version of this figure is available in the online journal.)

described previously (Podosek et al. 1997 and Moynier et al. 2007 for Cr, and Dauphas et al. 2004 for Fe). In order to minimize the Ni isobaric interference on the ^{58}Fe , the samples have been passed up to four times through the ion-exchange columns.

The Cr isotopic data reported here have been analyzed by TIMS Micromass Sector 54 at Washington University in St Louis (WUSTL), following the protocol described in Podosek et al. (1997). Fe isotopic data were measured using the MC-ICP-MS (Thermo-Finnigan Neptune Plus) at WUSTL. The Fe isotopic samples were introduced into the mass spectrometer using an Apex-Q+Spiro inlet system and a $100\ \mu\text{L}\ \text{min}^{-1}$ PFA nebulizer. The measurements were done in medium-resolution mode on the peak shoulder in order to resolve the isobaric interferences of $^{40}\text{Ar}^{14}\text{N}$ with ^{54}Fe , $^{40}\text{Ar}^{16}\text{O}$ with ^{56}Fe , and $^{40}\text{Ar}^{16}\text{OH}$ with ^{57}Fe . The intensities of masses 53, 54, 56, 57, 58, and 60 were measured on the Faraday cups L3, L2, central, H1, H2, and H4, respectively.

3. RESULTS

Cr and Fe isotopic data are given in Table 1 and in Figures 1 and 2 as epsilon units (deviation in parts per 10,000 relative to the composition of the Cr SRM 986 and Fe IRMM014 standards) after internal normalization to $^{50}\text{Cr}/^{52}\text{Cr}$ of 0.051859 and $^{57}\text{Fe}/^{54}\text{Fe}$ of 0.362566 using the exponential law (Maréchal et al. 1999). For Fe, we report an alternative normalization to $^{56}\text{Fe}/^{54}\text{Fe} = 15.69786$. The errors are reported as 2SE of the replicated measurements.

All of the samples analyzed in this study are found to have Fe isotope compositions similar to the terrestrial standard within the level of analytical precision of 30 ppm for the $^{56}\text{Fe}/^{54}\text{Fe}$ and of 150 ppm for the $^{58}\text{Fe}/^{54}\text{Fe}$ for a normalization to $^{57}\text{Fe}/^{54}\text{Fe}$. All samples also have a terrestrial Fe isotopic composition when

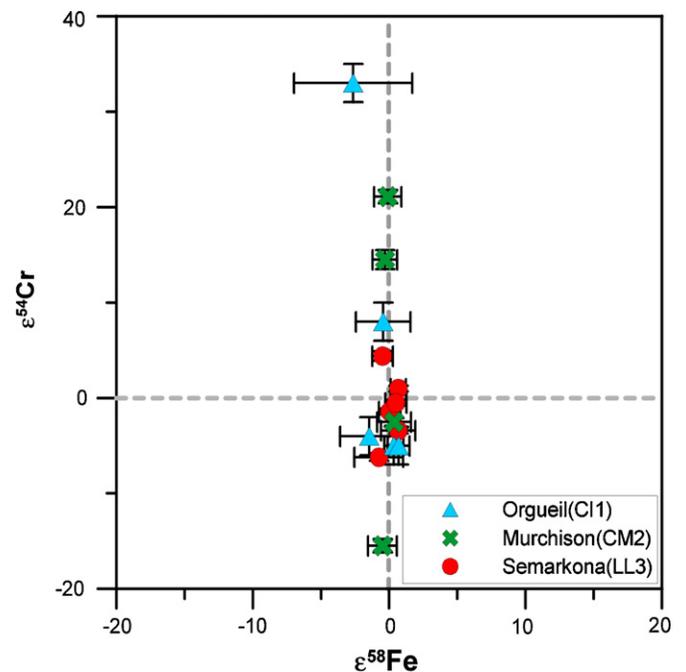


Figure 2. $\epsilon^{54}\text{Cr}$ vs. $\epsilon^{58}\text{Fe}$ in leaching fractions from Orgueil, Murchison, and Semarkona. Please refer to Table 1 for the exact data.

(A color version of this figure is available in the online journal.)

normalized to $^{56}\text{Fe}/^{54}\text{Fe}$ (Table 1). The fractions Semarkona I-6, Orgueil IV-6 and 7, and Murchison II-6 did not have enough iron to perform an isotopic measurement. The Cr isotopic compositions of Murchison and Semarkona show a similar pattern with Orgueil, in the sense that at least most of the Cr in these meteorites have anomalous relative abundance of ^{54}Cr . The sizes of the anomalies in Murchison and Semarkona are smaller (although still well resolved analytically) than in Orgueil, and the pattern in which the anomalies appear is different (Figure 1), but the essential features of the isotopic effects in Murchison and Semarkona are fundamentally similar to those in Orgueil: a HCl-soluble phase bears Cr with excess ^{54}Cr and other Cr carriers are deficient in ^{54}Cr . These results are in good agreement with Trinquier et al. (2007); Qin et al. (2010) for Murchison and the recent report of Cr isotopic data in leachates from the ungrouped carbonaceous chondrite Tagish Lake (Petitat et al. 2011). In addition, some fractions of Semarkona show ^{53}Cr excesses which is agreement with data on ordinary chondrites which have small ^{53}Cr excesses (Qin et al. 2010; Trinquier et al. 2007; Yin et al. 2007). To our knowledge, this study reports the first leaching experiment on an ordinary chondrite and shows that ordinary chondrites contain small ^{54}Cr anomalies.

4. DISCUSSION

The ^{54}Cr excess observed here is nucleosynthetic in origin as no known or plausible isotopic fractionation mechanism, energetic particle reaction (i.e., spallation), or short-lived nuclide progenitor (in particular ^{54}Mn) can account for the magnitude and mono-isotopicity of this effect. The only known stellar sites expected to produce significant excesses of ^{54}Cr are either the *s*-process in Type II supernovae (The et al. 2007) or particularly rare massive Type Ia supernovae that achieve significant neutron-richness during the NSE followed by the low entropy freezeout, often producing other Fe-group

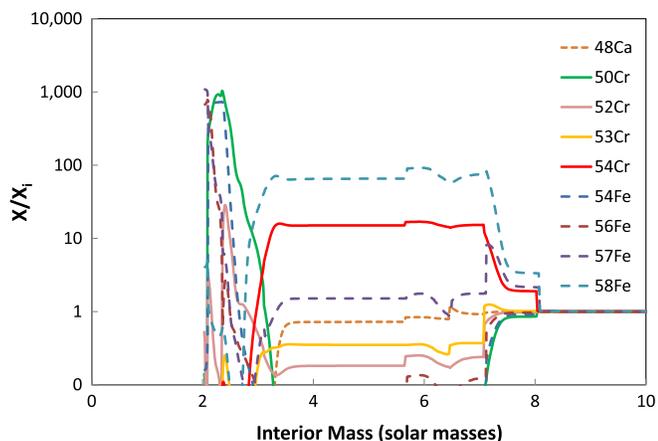


Figure 3. Iron and chromium overabundances (defined as mass fraction, X , relative to the initial mass fraction in the star, X_i) as a function of interior mass coordinate in a 25 solar mass supernova ejecta, stellar model s25a28d (figure generated from data of Rauscher et al. 2002). While most of the Fe and Cr isotopes are produced in the innermost supernova regions ($\leq 3 M_{\odot}$), the neutron-rich, ^{54}Cr , and ^{58}Fe are enriched in the mass range ≈ 3 to ≈ 8 solar masses. This is the material that experienced helium and some carbon burning. The other isotopes of Cr and Fe are either little changed or are depleted in these layers.

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elements with neutron-rich excesses, in particular ^{48}Ca , ^{50}Ti , ^{58}Fe , ^{64}Ni , and ^{70}Zn (Cameron 1979; Hartmann et al. 1985; Woosley et al. 1986, 1995; Woosley & Weaver 1997; Meyer et al. 1996; Woosley 1997). Only about 15% of the solar system's supply of ^{54}Cr is formed in Type II supernovae by s -process (The et al. 2007).

Correlated enrichments and depletions of the neutron-rich isotopes of Ca, Ti, Cr, Fe, and Zn have indeed been measured in a variety of inclusions, in particular FUN CAIs as well as in carbonaceous chondrites. These correlated variations have been attributed to material having formed under NSE conditions (Lee et al. 1978; Niederer & Papanastassiou 1984; Niederer et al. 1980; Papanastassiou 1986; Papanastassiou & Brigham 1989; Völkening & Papanastassiou 1989). While the effects in CAIs and in the ^{54}Cr -rich phase may be related, the carrier-phase does not contain excesses in either ^{48}Ca (Moynier et al. 2010) or in ^{58}Fe isotopes (this study), and the ^{54}Cr -rich carrier resides in a low-density and/or small grain size in a mineral soluble in HCl, unlike the higher density, more refractory phases present in CAIs. It has been recently proposed that the carrier phase of the ^{54}Cr anomalies measured in the stepwise leaching of Orgueil and Murchison may be related to nanoparticles (most likely spinels; Dauphas et al. 2010; Qin et al. 2011) with nucleosynthetic origins.

The absence of ^{58}Fe anomalies in the leaching fractions of carbonaceous chondrites with large ^{54}Cr effects can be used to refine the nucleosynthetic origin of the ^{54}Cr . Based on the absence of ^{48}Ca anomalies in the Orgueil leachates O-I, Moynier et al. (2010) proposed that the ^{54}Cr must have been synthesized either in the oxygen-rich, s -process enhanced ejecta from massive stars or in particular zones in the rare Type Ia supernovae. In the former origin, the grains should be enriched in ^{58}Fe with a $^{58}\text{Fe}/^{54}\text{Cr}$ overabundance of ~ 4 – 5 (Figure 3). Therefore our data suggest that the ^{54}Cr anomalies are either produced by rare Type Ia supernovae or ^{54}Cr was indeed co-synthesized with ^{58}Fe in Type II supernovae, but it was chemically separated into some mineral phase that favors Cr

over Fe, and it is the dissolution properties of that phase that is driving the isotopic effect in leaching. From our data it is not possible to tell which star produced the mono-isotopic ^{54}Cr excesses. Dauphas et al. (2010) and Qin et al. (2011) found nanoparticles with very large ^{54}Cr enrichments. Based on the oxygen-rich composition of these grains and on the rarity of Type Ia supernovae able to produce such ^{54}Cr excesses, Type II supernovae are the most likely origin for the ^{54}Cr isotopic anomalies (Dauphas et al. 2010; Qin et al. 2011).

Finally, the lack of large ^{58}Fe anomalies in leachates of Orgueil and Murchison confirm that Fe isotopes were homogeneously distributed in the early solar nebula (Dauphas et al. 2008), not only at the bulk rock scale but also at the mineral scale. These results confirm that the short-lived nuclide ^{60}Fe ($T_{1/2} = 2.62$ Myr) was homogeneously distributed to less than 15% dispersion in the early solar nebula (Dauphas et al. 2008; Moynier et al. 2009).

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