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Oxygen Isotopes in Refractory Stratospheric Dust Particles: Proof of Extraterrestrial Origin

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The oxygen and magnesium isotopic compositions of five individual particles that were collected from the stratosphere and that bear refractory minerals were measured by secondary ion mass spectrometry. Four of the particles exhibit excesses of oxygen-16 similar to those observed in anhydrous mineral phases of carbonaceous chondrites and thus are extraterrestrial. The oxygen and magnesium isotopic abundances of one corundum-rich particle are consistent with a terrestrial origin. Magnesium in the four extraterrestrial particles is isotopically normal. It is unlikely that these particles are derived from carbonaceous chondrites and thus such particles probably represent a new type of collected extraterrestrial material.

THERE IS MUCH EVIDENCE THAT most, and probably all, of the particles collected from the stratosphere that have elemental abundances similar to those of carbonaceous chondrites are samples of interplanetary dust particles (IDPs). The mineralogical, optical, and isotopic properties of these "chondritic" IDPs show that they are a diverse assemblage of primi-

tive materials from the solar nebula (1). Some IDPs have apparently undergone less chemical and thermal alteration on their parent bodies than have carbonaceous meteorites, and it is probable that many IDPs, especially the highly porous ones (2), are derived from comets. One notable difference between IDPs and most carbonaceous chondrites is the relatively low abundance of high-temperature mineral phases in IDPs. Although many chondritic IDPs contain enstatite laths and ribbons that have been interpreted as evidence for high-temperature vapor-to-solid condensation processes (3), in only two particles have the more refrac-

tory mineral phases that occur in the calcium- and aluminum-rich inclusions (CAIs) of carbonaceous meteorites been found (4, 5). One possible reason for this dearth of very refractory material in chondritic IDPs is that they formed at lower temperatures than those necessary for calcium-aluminum oxides to be stable in a gas of solar composition (6). Another possible explanation is that particles with high abundances of refractory oxide minerals are by definition not chondritic and so may have simply been overlooked, since most studies have concentrated on chondritic particles.

The primary difficulty in identifying non-chondritic IDPs in the National Aeronautics and Space Administration (NASA) stratospheric collection is distinguishing them from the abundant terrestrial contaminant particles. Circumstantial evidence for an extraterrestrial origin of single-mineral mafic silicate grains and of iron-sulfur-nickel particles is provided by their frequent association with chondritic material (7). The difficulties in identifying refractory extraterrestrial particles are exacerbated by pollution in the stratosphere, which includes refractory materials from rocket exhaust and spacecraft debris (8). In particular, the large abundance of aluminum-oxide spherules in the stratosphere leads to a possibility that these particles could get mixed with chondritic material on the collection surface. The only rigorous means for proving that small particles collected on Earth are of extraterrestrial origin is to measure either effects that can only be attributed to prolonged space exposure (for example, the presence of solar-flare nuclear particle tracks) or isotopic abundances that cannot be derived from the terrestrial composition by naturally occurring physical or chemical processes (for example, radioactive decay or isotopic mass fractionation). Previous hydrogen and mag-

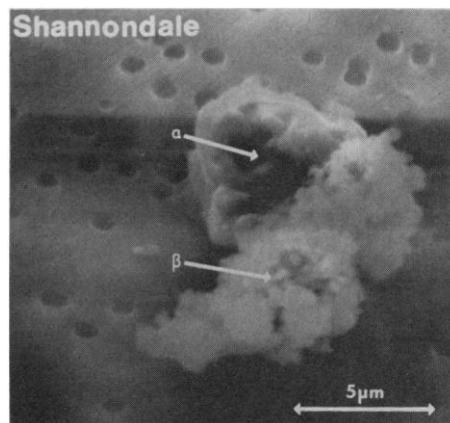


Fig. 1. Secondary electron micrograph of the IDP Shannondale. The energy-dispersive x-ray spectrum of spot α shows aluminum only, whereas that of spot β is chondritic.

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Table 1. Oxygen isotopic measurements in terrestrial oxide standards. *N* is the number of individual small grains analyzed. The quoted errors are 1 standard error of the mean. Gas-phase mass spectrometric measurements were provided by Clayton and Mayeda (25).

Standard sample	<i>N</i>	Oxygen isotopic composition relative to SMOW (per mil)			
		Gas-phase mass spectrometry		Washington University ion probe	
		$\delta^{18}\text{O}$	$\delta^{17}\text{O}$	$\delta^{18}\text{O}$	$\delta^{17}\text{O}$
Burma spinel	9	22.3 $\pm$ 0.2	11.6 $\pm$ 0.2	$\equiv$ 22.3	11.4 $\pm$ 1.4
Madagascar hibonite	7	10.0 $\pm$ 0.2	5.2 $\pm$ 0.2	11.0 $\pm$ 1.0	5.8 $\pm$ 1.4
Burma spinel	15	22.3 $\pm$ 0.2	11.6 $\pm$ 0.2	$\equiv$ 22.3	10.8 $\pm$ 0.9
New York spinel	13	15.1 $\pm$ 0.2	7.8 $\pm$ 0.2	15.4 $\pm$ 0.6	6.7 $\pm$ 1.0

nesium isotopic measurements of aluminum-rich stratospheric dust particles (SDPs) did not show any evidence for an extraterrestrial origin, and it is likely that most of these so-called Al-prime particles are man-made (9).

Zolensky has used transmission electron microscopy to identify SDPs that consist mainly of refractory mineral phases common in fine-grained CAIs. These particles are compositionally and morphologically unlike the Al-prime particles or any known contaminants, and thus he has suggested that they are IDPs (10). I have used a modified Cameca IMS-3f ion microprobe to measure the oxygen isotopic compositions of four of these particles, whose mineralogy is described by Zolensky in a companion report (11), and also of a $\sim 5\text{-}\mu\text{m}$ diameter aluminum-oxide grain that was attached to fluffy chondritic material in the particle Shannondale (Fig. 1). Oxygen was chosen because it is diagnostic for the origin of refractory oxide mineral phases. Anhydrous minerals from carbonaceous chondrites show excesses of ^{16}O that range up to $\sim 5\%$ relative to terrestrial materials (12). In addition, oxygen isotopes have provided a general classification scheme for meteorites and planetary bodies (13). The measurement of oxygen isotopic abundances (including ^{17}O) by secondary ion mass spectrometry is not well established (14), so some discussion of the experimental techniques is warranted.

Several technical problems must be overcome to make precise and accurate measurements of oxygen isotopes with the ion probe. First, to obtain good sensitivity the sample should be sputtered with a primary Cs^+ beam and negative secondary ions should be analyzed. However, this procedure causes severe charging of insulating samples. This problem is largely alleviated by embedding small particles in an ultra-clean gold foil. Residual charging effects (typically $< 5\text{ V}$) are monitored during the measurement and compensated by offsetting the accelerating voltage. Second, since the $^{16}\text{OH}^-$ interference can be ~ 2 to 100 times greater than the $^{17}\text{O}^-$ signal, measurements

are made at a high mass-resolving power of ~ 8000 (Fig. 2). Possible contributions to the $^{17}\text{O}^-$ signal from scattered $^{16}\text{OH}^-$ ions are corrected for by monitoring the tail of the $^{16}\text{O}^-$ peak at the appropriate mass that corresponds to the separation of the ^{16}OH and ^{17}O peak centers. In most cases this correction was < 1 per mil. Finally, since the ^{16}O and ^{17}O abundances differ by a factor of ~ 2500 , accurate ion counting must be performed over a wide dynamic range. The counting system deadtime was determined by titanium isotopic measurements to be 18 ± 1 nsec. The ^{16}O count rate for both samples and standards was kept below 5×10^5 count/sec, so there are no artifacts in the data due to incorrectly determined deadtime losses (15).

Measurements (16) were performed alternately on the samples and on small grains from terrestrial standards of known isotopic composition (Madagascar hibonite, Burma

spinel, and New York spinel). The standards were chosen to approximate the mineralogy of Zolensky's SDPs in order to minimize possible instrumental effects due to matrix-dependent mass fractionation. Corrections for instrumental mass fractionation in the measured isotopic ratios were made by normalizing the mean measured $^{18}\text{O}^-/^{16}\text{O}^-$ ratio for one of the standards to the SMOW (Standard Mean Ocean Water) scale according to an assumed exponential law (17). The data are reported as $\delta^{17}\text{O}$ and $\delta^{18}\text{O}$ relative to the SMOW scale:

$$\delta^i\text{O} = \left[\frac{(^i\text{O}/^{16}\text{O})_{\text{Sample}}}{(^i\text{O}/^{16}\text{O})_{\text{SMOW}}} - 1 \right] \times 1000$$

where $(^{18}\text{O}/^{16}\text{O})_{\text{SMOW}} \equiv 0.0020052$ and $(^{17}\text{O}/^{16}\text{O})_{\text{SMOW}} = 0.00038309 \pm 0.00000034$ (18). Table 1 shows the results of measurements obtained by treating Burma spinel as the primary standard and Madagascar hibonite and New York spinel as "unknowns." No significant matrix effects exist between the two types of spinel and the hibonite.

As a test of the ability of the ion probe to accurately measure non-mass-dependent isotopic effects in oxygen, I attempted to reproduce the ^{16}O -rich mixing line observed for refractory minerals from CAIs (19). Individual $\sim 10\text{-}\mu\text{m}$ sized spinel and hibonite grains were picked from the oxygen-ashed acid residue CFOc (20) of the CM chondrite Murchison, pressed into gold foil, and analyzed in the same manner as were the SDPs.

Table 2. Oxygen isotopic compositions of Murchison oxide grains and stratospheric dust particles. The quoted errors are 1 standard error of the mean.

Sample	Mineralogy*	Oxygen isotopic composition relative to SMOW (per mil)			
		$\delta^{18}\text{O}$	$\delta^{17}\text{O}$	$^{16}\text{OH}_{\text{corr}}^\dagger$	$^{16}\text{O}_{\text{excess}}^\ddagger$
<i>Murchison CFOc oxide grains</i>					
211	Sp	-43.2 $\pm$ 2.3	-47.7 $\pm$ 2.1	0.2	52.6 $\pm$ 5.0
212	Hib	-36.1 $\pm$ 2.4	-43.4 $\pm$ 4.9	0.2	51.3 $\pm$ 10.5
213	Sp, Chr	-44.3 $\pm$ 13.4	-43.2 $\pm$ 8.6	0.4	42.0 $\pm$ 23.1
214	Sp	-39.3 $\pm$ 2.2	-47.1 $\pm$ 2.6	0.1	55.5 $\pm$ 5.9
215	Sp	-47.6 $\pm$ 4.7	-57.4 $\pm$ 5.2	0.3	68.0 $\pm$ 12.0
216	Hib	-37.0 $\pm$ 1.7	-41.2 $\pm$ 2.6	0.2	45.7 $\pm$ 5.7
218	Sp	-39.8 $\pm$ 1.4	-38.1 $\pm$ 1.6	0.2	36.3 $\pm$ 3.7
134	Chr, ?	-15.9 $\pm$ 4.1	-20.6 $\pm$ 7.5	2.3	25.7 $\pm$ 16.2
131	Sp	-30.6 $\pm$ 8.1	-15.5 $\pm$ 9.1	2.2	-0.9 $\pm$ 20.9
132	Sp	-4.4 $\pm$ 4.8	-5.6 $\pm$ 4.7	6.2	6.9 $\pm$ 11.1
135	Sp	-24.5 $\pm$ 2.2	-27.0 $\pm$ 2.6	2.6	29.7 $\pm$ 5.9
<i>Stratospheric dust particles</i>					
W7017 1A92	Cor	-13.5 $\pm$ 3.7	-5.8 $\pm$ 2.6	0.3	-2.5 $\pm$ 6.7
W7017 1A114a	Hib, ?	-5.1 $\pm$ 2.6	-11.3 $\pm$ 3.7	0.1	18.0 $\pm$ 8.2
W7017 1A114b	Hib, ?	-7.8 $\pm$ 3.1	-13.9 $\pm$ 3.3	0.1	20.5 $\pm$ 7.6
W7017 1A55	Sp, Hib, Mel, Pv	-41.2 $\pm$ 1.2	-37.1 $\pm$ 1.0	1.1	32.7 $\pm$ 2.5
W7029 H15	Sp, Hib, Mel, Pv	-35.4 $\pm$ 2.0	-36.5 $\pm$ 2.6	0.2	37.7 $\pm$ 5.8
Shannondale α	Cor	-14.3 $\pm$ 2.2	-15.3 $\pm$ 2.3		16.4 $\pm$ 5.4

*For the Murchison grains and Shannondale α , the minerals listed are those determined to be present by energy-dispersive x-ray analysis. See (11) for a description of the mineralogy of the SDPs. Abbreviations: chromite, Chr; corundum, Cor; melilite, Mel; hibonite, Hib; perovskite, Pv; spinel, Sp; uncertain or unknown phases, ?. † Correction to $\delta^{17}\text{O}$ for scattered $^{16}\text{OH}^-$ ions. For Shannondale α this correction was not made, but high mass-resolution spectra indicate that $^{16}\text{OH}^-$ contributions were less than 1 per mil. ‡ This term is defined as (13):

$$[0.52/(1 - 0.52)]\delta^{18}\text{O} - [1/(1 - 0.52)]\delta^{17}\text{O}$$

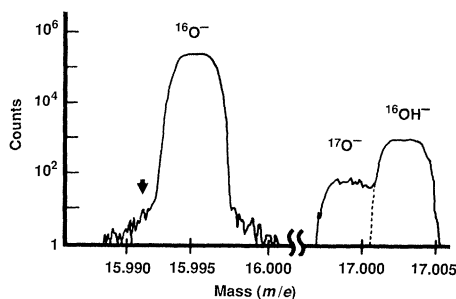


Fig. 2. High mass-resolution spectrum of the $m/e = -16$ and $m/e = -17$ regions for a terrestrial spinel grain. The contribution to the $^{17}\text{O}^-$ signal from scattered $^{16}\text{OH}^-$ ions is monitored by measuring the relative proportion of scattered $^{16}\text{O}^-$ ions at a mass (denoted by the arrow) that corresponds to $m_{16\text{O}} - (16/17) \cdot \Delta m_{17}$, where Δm_{17} is the mass difference between ^{16}OH and ^{17}O . There are no significant interferences at $m/e = -18$.

The results are shown in Table 2 and Fig. 3A. The line that best fits the data for the 11 Murchison grains is:

$$\delta^{17}\text{O} = (1.13 \pm 0.15) \cdot \delta^{18}\text{O} + (2.1 \pm 5.5)$$

(with $\chi^2 = 1.1$), which is consistent with the mixing line defined by more precise data (on much larger samples) by Clayton and Mayeda (19). Thus the ion probe can accurately measure ^{16}O excesses characteristic of high-temperature meteoritic minerals in small (~ 10 ng) particles.

The results of the oxygen isotopic measurements for the SDPs (21) are shown in Table 2 and are plotted in Fig. 3B. With the exception of the blocky corundum grain 1A92, the points lie along the ^{16}O -rich mixing line. Four of the particles exhibit well-resolved ^{16}O excesses, which confirms Zolensky's hypothesis of an extraterrestrial origin (10). This conclusion holds whether the ^{16}O excesses in CAIs are due to the incomplete mixing of a distinct nucleosynthetic component with "normal" solar system oxygen [see, for example, (13, 19)] or due to non-mass-dependent chemical fractionation effects that may have occurred in the solar nebula (22). Also, since the oxygen in these particles is indigenous, these ^{16}O excesses could not have been generated by exposure of the grains in the stratosphere to oxygen of unusual isotopic composition (23).

The data for 1A92 are consistent with mass-fractionated terrestrial oxygen; however, it cannot be rigorously concluded that this fractionation is intrinsic to the sample since it could be due to a difference in the degree of instrumental mass fractionation between corundum and spinel. This is unlikely, given the magnitude of the fractionation and the lack of observed matrix effects between hibonite and spinel. Thus 1A92 is probably isotopically light compared to most terrestrial oxides, but this provides no

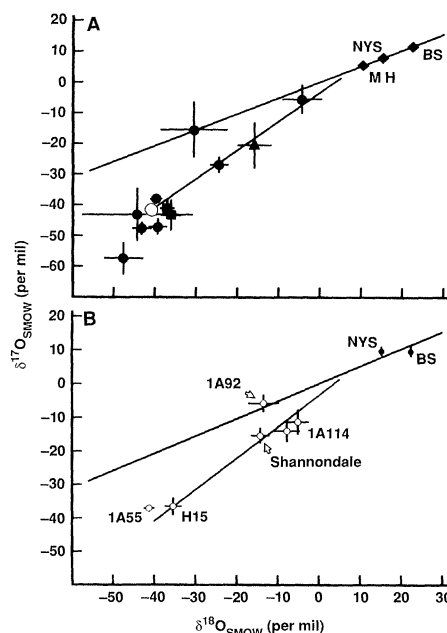


Fig. 3. (A) The oxygen isotopic compositions of 11 Murchison CFC oxide grains relative to SMOW (●, spinel; ■, hibonite; ▲, chromite). The terrestrial standards [shown as ◆; Madagascar hibonite (MH), New York Spinel (NYS), and Burma spinel (BS)] plot along a mass-fractionation line with a slope of 0.52. Also shown are the ^{16}O -mixing line (slope = 0.95) for anhydrous phases of Murchison and the composition of Murchison spinel (shown as ○) from Clayton and Mayeda (19). Error bars are 1 standard error of the mean. (B) The oxygen isotopic compositions of five stratospheric dust particles that bear refractory minerals and of terrestrial spinel standards. Also shown are the terrestrial mass-fractionation line and the Murchison ^{16}O -mixing line (19). Error bars are 1 standard error of the mean.

evidence for an extraterrestrial origin for this grain.

The ion probe was also used to measure the magnesium isotopic compositions of the SDPs by techniques similar to those reported in (24). No anomalies were found. For the corundum samples 1A92 and Shannondale α (Fig. 1), limits on the initial $^{26}\text{Al}/^{27}\text{Al}$ abundances are $<10^{-6}$ and $<7 \times 10^{-6}$, respectively. In the other SDPs, phases with high concentrations of aluminum relative to magnesium could not be spatially resolved so that meaningful limits on any possible initial concentration of ^{26}Al could not be set.

Based on textural evidence and the observation of noncrystalline phases, Zolensky has indicated (11) that these refractory dust particles are probably not derived from fine-grained CAIs by ablation of carbonaceous meteorites during atmospheric entry. This conclusion is strengthened by the demonstration of the extraterrestrial nature of an aluminum-oxide grain from Shannondale, a particle which in other respects resembles a typical chondritic aggregate IDP. Thus it is

likely that these particles represent a new type of collected extraterrestrial material.

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- $(R_m^{18}/R_i^{18}) = (18/16)^P$, where R_m^{18} and R_i^{18} are the measured and true ($^{18}\text{O}/^{16}\text{O}$) ratios, respectively. A similar expression holds for ($^{17}\text{O}/^{16}\text{O}$). The exponent P is determined for a measurement sequence from the R_m^{18} and the known R_i^{18} of a terrestrial standard. See (15) for a detailed discussion of the instrumental mass-fractionation correction.
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A Simple Model for Neutrino Cooling of the Large Magellanic Cloud Supernova

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A simplified analytic model of a cooling hot neutron star, motivated by detailed computer calculations, describes well the neutrinos detected from the recent supernova in the Large Magellanic Cloud. The observations do not require explanations that invoke exotic physics or complicated astrophysics. The parameters in this simple model are not severely constrained: $6.1^{+3.5}_{-3.6} \times 10^{52}$ ergs emitted in electron antineutrinos, a peak temperature of $4.2^{+1.2}_{-0.8}$ mega-electron volts, a radius of 27^{+17}_{-15} kilometers, and a cooling time of $4.5^{+1.7}_{-2.0}$ seconds.

THE DETECTION OF NEUTRINOS from the Large Magellanic Cloud (LMC) supernova (SN1987a) by the Kamiokande II (1) and the Irvine-Michigan-Brookhaven (IMB) (2) detectors was an epochal event in astronomy and physics. Most of the energy of the recent LMC supernova was emitted in neutrinos, a confirmation of the standard picture of core collapse (3, 4).

The physics of the explosion was complicated and requires detailed models for a correct description. However, the success of the simplified model described here suggests that the data from the LMC supernova are too sparse to discriminate among more sophisticated models or to justify inventing exotic new physics. In order to test in more detail our understanding of stellar collapse, we must await detection of a galactic core collapse [~ 50 times as many events expected at a rate of order one collapse event every 8 years (5)] or the availability of much larger detectors to observe stellar collapses in other galaxies.

The fluences and temperatures inferred from the neutrino observations were consistent with pre-supernova expectations (6, 7). However, there is one unexpected feature of the LMC supernova neutrino data: the 7.3-second gap between the first eight and last three events in the Kamiokande II

data. The Kamiokande II detector observed eight events in the first 1.9 seconds, followed by a quiet period of 7.3 seconds, and then three events were detected within 3.2 seconds. The IMB detector observed six events in the first 2.7 seconds, followed by a quiet period of 2.4 seconds, and then two events were detected within 0.6 second. Many investigators have claimed that exotic new physics or complicated astrophysics is required to understand the arrival times of the neutrinos (8–10). Figure 1 shows that the average energy of an event appears to decline with time and shows the agreement of the data with a cooling blackbody model. We show that a cooling hot neutron star model fits well all the observed data and provides an estimate of the radius of the hot neutron star. We focus on this simple model to show that it is not necessary to invent new physics to explain the observation; the observations can be fit by a simple model motivated by detailed calculations performed before the occurrence of SN1987a.

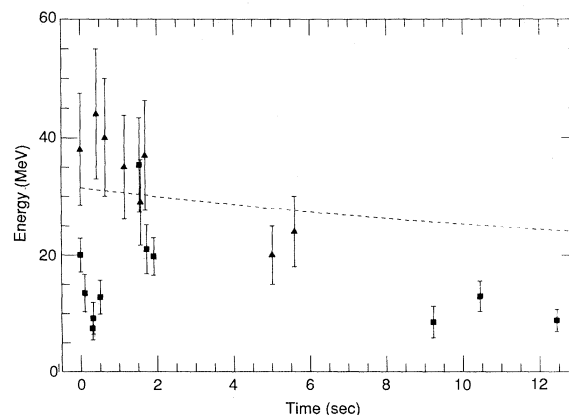


Fig. 1. Energy of the observed neutrino events in Kamiokande II (▲) and IMB (■) as function of time of arrival. The dashed line shows the expected average energy of an event in IMB based on the cooling blackbody model with $T_0 = 4.2$ MeV and $\tau = 4.5$ seconds. The dotted line shows the expected average energy of an event in Kamiokande II. [Data and error bars from (1, 2).]

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We combine the IMB and Kamiokande data sets with the assumption that the first neutrino in IMB arrived at the same time as the first neutrino observed by Kamiokande; the offset time is not known precisely. Given the observed rates, the expected time lag is ≈ 0.25 second. Our conclusions do not change if we include a time lag of this order. We also neglect neutrino scattering events. Bahcall *et al.* (6) considered the angular distribution and concluded that zero to three of the first Kamiokande events (2% significance) and zero to two of the IMB events (5% significance) may have been the result of scattering; the inclusion of scattering did not alter the estimates of neutrino temperature and would not change any of the conclusions of this report. These significance levels represent the fraction of Monte Carlo simulations of the angular distribution of events that have a larger Kolmogorov-Smirnov (KS) measure than the observations.

The temporal structure of the combined data can be fitted by an exponentially decaying flux $F \propto \exp[-(\ln 2)t/t_{1/2}]$, where $t_{1/2} = 2^{+0.9}_{-0.8}$ seconds is the decay constant (see Fig. 2). (All numbers in this report are quoted with 95% confidence limits.) Monte Carlo simulations of data drawn from this function show that a worse fit for the KS measure would be obtained in 10% of the cases. The exponential decay is not unique, and other functional forms [such as $F \propto \exp[-(t/\tau)^{1/2}]$ with the first half of the events arriving in the first $t_{1/2} = 2.8\tau = 1.4^{+0.8}_{-0.5}$ seconds] can provide even better fits to the data (see Fig. 2). Although these functions fit the observed temporal structure, they do not explain the apparent relation between time of arrival and average energy of the events.

When the core of a massive star can no longer support itself, it collapses rapidly on a dynamical time scale of milliseconds. When the density in the core reaches nuclear densities, nuclear pressure stops the collapse, and in this core bounce the gravitational binding energy is converted into thermal energy.