**SUPPLEMENTARY INFORMATION ON METHODOLOGY**

Selecting samples for extracting magnetic microspherules

We collected 32 sediment samples from the sediment sequence at depths ranging from -141 to +118 cm (Fig. 9A and Table S5). Thirteen of these samples were taken at continuous 1-cm-thick intervals from -27 to -22 cm and at 2-20-cm intervals from -141 to -27 cm. Of these 13 samples, 3 at the bottom of the sequence (-141, -121, -81 cm) were not analyzed because they were collected in the saturated zone below the water table and were potentially disturbed and mixed by slumping during collection. The available masses of the remaining 10 samples ranged from ~250 to 400 g. Nineteen 2-cm-thick samples were collected discontinuously from -71 to +118 cm, and the available masses of this group ranged from 20 to 51 g.

Extracting and counting magnetic grains

Magnetic grains were extracted from the 32 samples noted above using a neodymium magnet, according to the protocol of Firestone et al. (2007), as refined by LeCompte et al. (2012) and Israde et al. (2012), and described below in detail. For 19 samples, the available mass (20 to 51 g) was smaller than typically studied, and we detected no spherules in those samples, although small sample mass makes accurate quantification somewhat problematic. For the 10 large-mass samples, magnetic spherule candidates were identified, manually extracted from the magnetic grains, and mounted on SEM stubs. Of these 10 samples, SEM imagery showed that only two contained spherules that are morphologically the same as spherules from other YDB sites that are interpreted as having melted under high temperatures (Wittke et al., 2013). Two hundred g of sediment were available for each of those two samples, with the remaining mass set aside for other tests, including Pt and Ir. The 200-g sample from -30 to -33 cm contained 2 spherules (10 spherules/kg) that date to the YD at ~12,850 ± 60 cal BP. The 200-g sample from -26 to -27 cm yielded 9 spherules (45 spherules/kg) (Fig. 9). Both of these concentrations fall at the lower end of the range of YDB spherules counts at 18 sites on two continents that vary in concentration from 5 to 4900 spherules per kilogram (average: 955/kg; median: 388) (Wittke et al., 2013). Spherules in the upper zone post-date the YDB event, possibly because of reworking of 12,850-year-old spherules from the surrounding watershed. There are no detectable spherules for ~250 years preceding the YDB impact event or ~1700 years afterward. The distribution of these melted magnetic spherules is shown in Figure 9A.

We used energy dispersive X-ray spectroscopy (SEM-EDS) to analyze chemical compositions of Lake Hind spherules, as listed in Table S5. We identified two primary compositional types: one dominated by iron oxide (FeO) ranging from 95-99 wt.%, and the other type with <46 wt.% FeO and the remainder mostly as aluminosilicates. A composite image (Fig. 8) shows the 9 magnetic spherules from the upper zone at -26 to -27 and 2 spherules from the deeper zone. The Fe-rich type displays dendritic surface textures, as typically observed in spherules and meltglass from other YDB sites (Wittke et al., 2013). In Figure 8, the close-up inset images show these dendritic details. The aluminosilicate type of spherules typically display a smooth morphology with a few vesicles indicative of high-temperature melting, typical of similar glassy spherules from other YDB sites (LeCompte et al., 2012; Wittke et al., 2013).

Details of sediment processing for magnetic mineral extraction

Sample bulk sediment was first screened to remove any large organic particulates or pebbles, using a screen mesh-size of 8 x 7 strands per cm (18 x 20 per inch). The remaining sediment was then immersed in water and thoroughly agitated to create a well-mixed slurry into which a bagged Neodymium-Boron (N2dFe14B) super-magnet (manufacturers strength N52 or 76.5 lb. pull force, surface field 3411 Gauss) was immersed for ~20 iterations. Magnetic grains adhered to the bag’s surface in proximity of the magnet. The bag was immersed in a bowl of distilled water. The magnet was then withdrawn from the bag freeing the grains to fall into the bowl.

The magnetic grains thus extracted typically included some non-magnetic soil components and organic debris captured among the grains. The grains were now rinsed to initiate removal of such material. This was accomplished by repeating the bagged magnet extraction process with grains now deposited as before in a bowl of clean distilled water. The distilled water was decanted through a double layer of 20-μm filters to capture any small grains that may have floated due to surface tension. Magnetic grains were then air-dried or dried under a sunlamp.

All collected magnetic grains were heated to at least a temperature of 500° C to burn away most organic material. Magnetic material was then sonicated for a total of 24 minutes. A third and final rinse processing of the material was performed to remove remaining debris. Once the grains were collected and cleaned, they were size-sorted using ASTM screens into three groups. Size sorting the grains and separate examination of each size specific group is essential to ease identification of the candidate microspherules. One group contained all grains larger than 106 μm (ASTM 140). Another contained grains whose size was between 53 (ASTM 270) and 106 μm. The third contained grains smaller than 53 μm.

The size-sorted groups of magnetic grains were then ready for examination to identify and collect any magnetic micro-spherule candidates for Scanning Electron Microscope (SEM) and X-ray energy dispersive spectrographic (EDS) analysis. Three methods were employed to collect magnetic-microspherule candidates separated as described above from bulk soil samples. These were:

1. *Selected Candidates*: A well-mixed aliquot of about 10 milligrams of similar-sized magnetic grains was placed in a Petrie-dish cover. Micro-spherule candidates were identified by examination with an optical microscope with at least 180x maximum magnification. The individual candidates were removed using a single strand artist’s paint brush or sharpened orange stick and placed upon a double-sided tape affixed to an SEM sample-mount.
2. *Swept-Magnet*: The super-magnet was placed in a bag and swept over all or a portion of the magnetic grains at a vertical distance of about two cm. A fraction of the relatively few grains captured have a high probability of being spherules.
3. *Distributed Grains*: A small amount (typically between 1 to 3 mg) of magnetic grains were dusted across a double-sided adhesive SEM tape affixed to an SEM sample mount.

The *Selected Candidate* method provides the most accurate estimation of the number of spherules per kg. The *Swept Magnet* method provides the most reliable way to determine the presence of any magnetic microspherules, although it is the least accurate way to estimate the per kilogram abundance because some grains may be missed, depending in the relative abundance and grain magnetic properties. The *Distributed Grains* method provides good abundance accuracy and a survey of what other particulates is contained in the magnetic fraction that may be relevant to the cosmic impact investigation. It is most useful when there is substantial impact proxy material contained in the magnetic fraction.

Iridium and platinum group analyses

Abundances of Ir and Pt in sediment may be identified during geochemical tests at a suitably equipped laboratory. The analysis and results for this study were provided by Activation Laboratories in Ancaster, Canada. Tests performed may include instrumental neutron activation analysis (INAA) or in some cases the more sensitive inductively coupled plasma mass spectrometry (ICP-MS) or by nickel sulfide fire assay analysis (FAA) followed by ICP-MS. The platinum group elements (PGE) gold, (Au), platinum (Pt), and palladium (Pd) abundances were measured to a sensitivity of 1 ppb for Au, 0.1 ppb for Pt and Pd. Iridium is tested to a sensitivity of 0.1 ppb by subjecting the sample to FAA, followed by INAA.

References:

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