Separation of YD Event Markers (8/10/2007)

1) Separation of the Magnetic Fraction from Sediment.

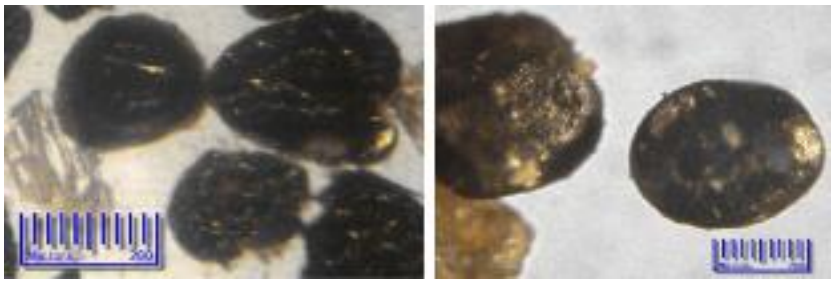

Fig. 1. Magnetic grains.

We used only **grade-42 or -52 neodymium magnets,** having found that nearly all other magnets are too weak and will fail to extract enough magnetic grains. Typically, we used the size 2”×1”×0.5”, which was convenient for both field and laboratory work. One source for these is K&J Magnetics, Inc., 1-888-746-7556, (http://www.kjmagnetics.com/), item # BY0X08-N52.

CAUTION: These powerful magnets can be dangerous. Keep them well away from metal objects, which they strongly attract. Also, credit cards, motel card keys, and electronic devices can be damaged.

Sonication with a ceramic magnet in a test tube is a common way to separate magnetic grains. However, we did not use the process since it typically collects only the smallest and most magnetic grains, excluding up to 90% of the rest, including small microspherules. Also, ceramic magnets are too weak.

Instead, we used several methods to separate magnetic grains, depending upon the sediment. For large-scale processing, the following basic procedures were used with automated equipment and a bank of magnets, which was placed in a moving stream of either wet or dry sediment. Small samples were processed manually.

A) Loose or sandy sediment.

- Friable sand or silt was first dehydrated at about room temperature and weighed. Bulk sediment from a single stratum was selected and typically weighed **at least 500-1000 g** (Fig. 2). If smaller samples were used, insufficient magnetic grains or microspherules often were recovered.

Fig. 2

- If necessary, the sample was gently pulverized. All processing was done with non-metallic tools to avoid adding foreign metal to the sample, and care was taken not to damage the magnetic grains, microspherules, charcoal, or carbon spherules.
- Next, the magnet was placed in a 4-mil plastic bag to prevent grains from sticking directly to the magnet (Fig. 3).

Fig. 3

- The sediment sample was poured into an empty container over the bagged magnet, which was held at an angle (Fig. 4). **NOTE:** The sediment was poured slowly and gently in order to retain small, weakly magnetic particles. Also, the bag was gripped tightly, since air gaps reduce magnetic strength.

Fig. 4

- Magnetic grains stuck to the edges of the magnet (arrow, Fig. 5).

Fig. 5

- The extracted grains were dropped into a separate container by withdrawing the magnet from the bag. The total process was repeated ~5-10 times until nearly all of the grains were recovered.
- If the grains were very clean, which was rare, the sample could be analyzed immediately. If dirty, as was typical, the magnetic fraction was **rinsed in water and extracted as described in B) below.**
- After drying, the magnetic fraction was weighed and catalogued. Grain abundances were calculated in grams per kg of bulk sediment.
- The entire magnetic fraction (Fig. 6) was tested by PGAA, INAA, or ICP-MS for ~50 elements, including iridium (Ir) and nickel. Most test sites showed anomalously high levels of Ir in the YDB magnetic fraction, apparently due to magnetic "nuggets" containing high concentrations of Ir.

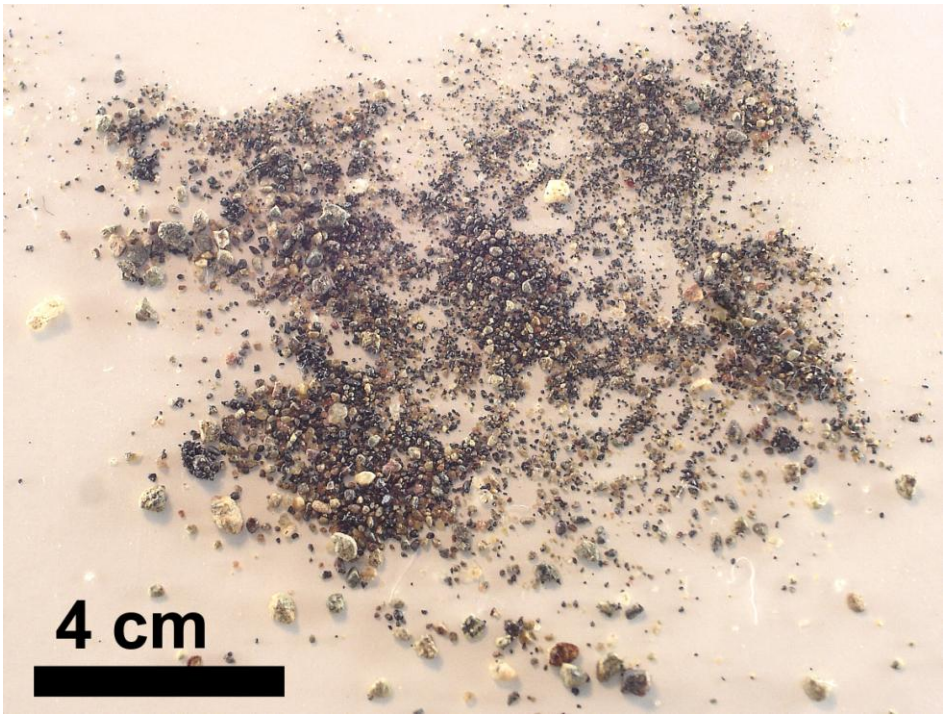

Fig. 6

B) Sticky or clayey sediment.

- For sediment that was difficult to pulverize, we added adequate water to each sediment sample to create a slurry (Fig 7). In convenient batches, we processed ~**500-1000 g** for each stratum.

Fig. 7

- The magnet, tightly stretched in a bag, was then immersed in the mixture (Fig. 8). **NOTE:** The magnet should be moved slowly and gently, otherwise water action will dislodge the smallest grains.

Fig. 8

- The magnetic fraction was withdrawn with the magnet (arrow, Fig. 9).

Fig. 9

- The bag, magnet, and grains were then immersed in a second container of clean water. Then, the magnetic grains were released from the magnet into the water by withdrawing the magnet from the bag (Fig. 10).

Fig. 10

- The above steps were repeated until only minimal additional grains could be extracted.
- Next, in order to separate excess dirt from the magnetic grains, the bag and magnet were used to retrieve the magnetic fraction from the second container (arrow, Fig 11).

Fig. 11

- After removing the magnet, the wet grains stuck to the bag and could be transferred to a lab dish by touching the bag to the dish surface (Fig. 12).

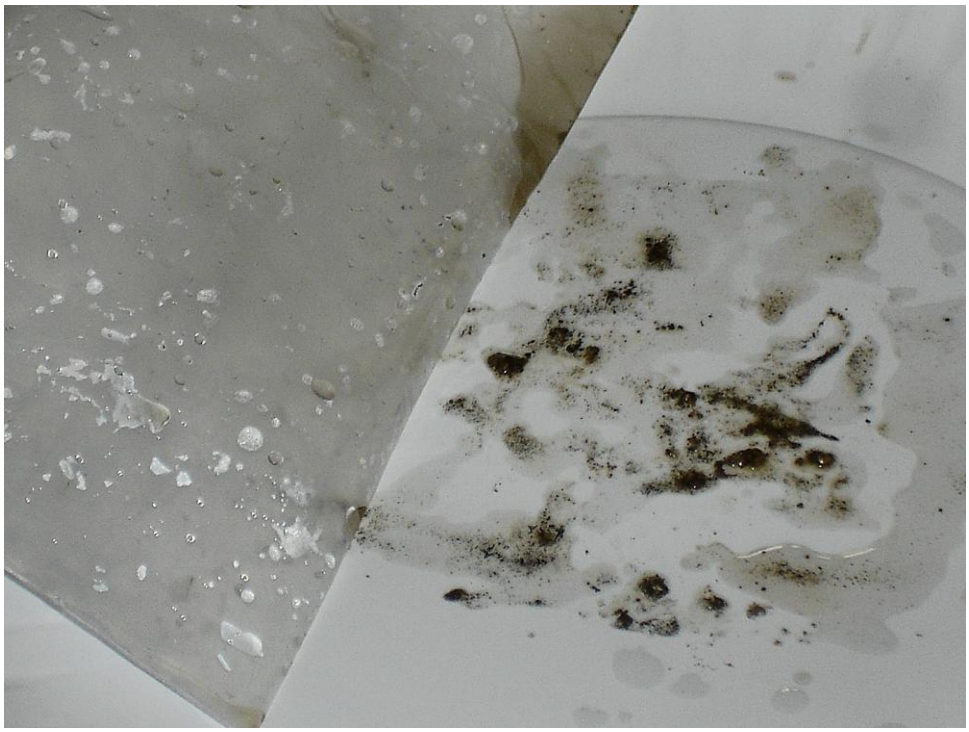

Fig. 12

- After drying, the magnetic fraction was weighed, catalogued, and analyzed as above.

2) Extraction of Magnetic Microspherules.

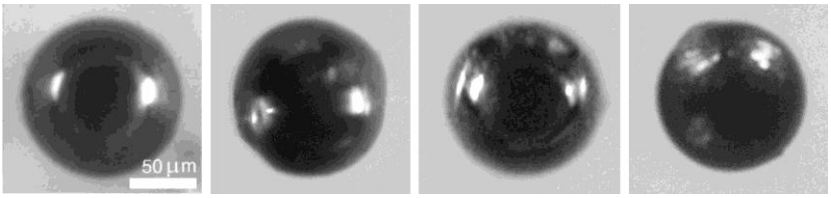

Fig. 13. Typical magnetic spherules.

- To find microspherules, the magnetic fraction was extracted as described above. To identify the maximum microspherules, we nearly always found it necessary to **clean the magnetic fraction with water, as outlined in 1B) above.**
- Since most microspherules are <150-µm in size, we used ASTM sieves to screen the magnetic grains into separate fractions and worked mostly with the <150-µm samples (ASTM #100 screen).
- One or more ~100-200 mg aliquots of the magnetic fraction were separated and weighed.
- To find spherules, we dusted the magnetic grains lightly across a microscope slide (Fig 14), being careful to **avoid leaving dense clusters of grains**, which made it difficult to distinguish the spherules. A white background makes it easier to locate the spherules.

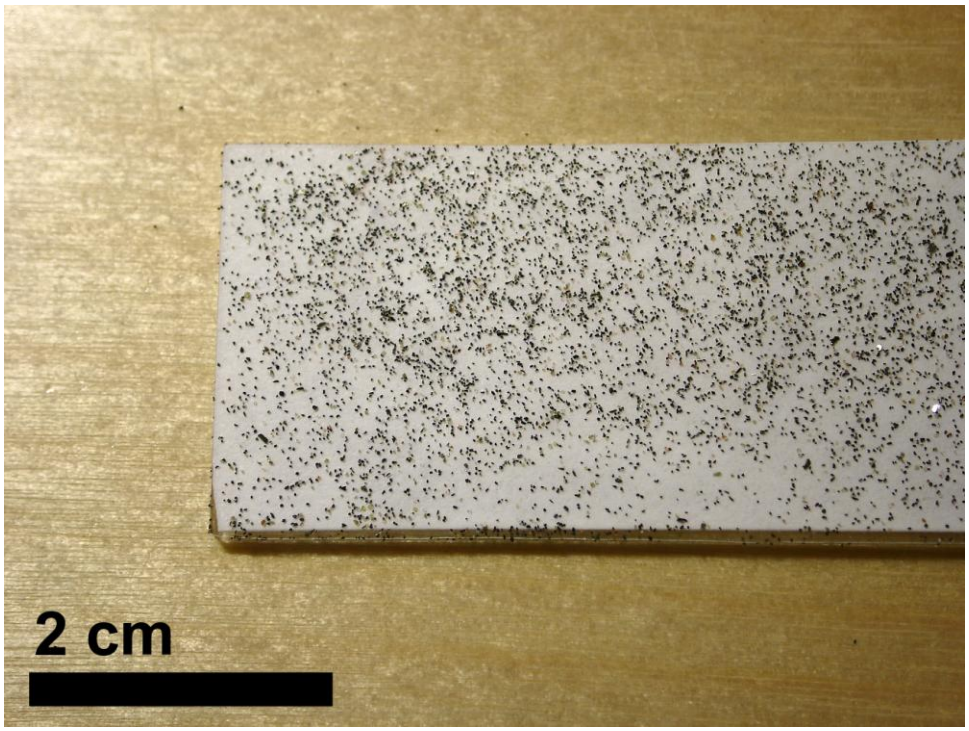

Fig. 14

- We scanned using a top-lit microscope, which had a mechanical stage, at no less than **100× to 150× magnification.** At lower resolution, the spherules were easily overlooked.
- Microspherules are usually rare, often making it necessary to inspect the entire magnetic fraction from 500-1000 g of sediment. Sometimes, there was only 1 microspherule per 20 grams of sediment.
- When found, microspherules were removed manually using the microscope and a sharpened, moistened, wooden probe or a single-bristle brush. They were placed on a microprobe/SEM mount or were temporarily placed on double-sided tape adhered to a microscope slide. Later, if it was necessary to remove them from the tape, a suitable solvent was used, such as alcohol. Once the glue was softened, the spherules were removed under the microscope with a magnetized needle.
- Next, they were tallied, photographed, and analyzed. Abundances were extrapolated to determine number of microspherules per kg of bulk sediment.
- Selected microspherules were mounted, sectioned, and analyzed by XRF and/or laser ablation.

SUMMARY. Extracting the magnetic spherules can be complicated, since the microspherules are difficult to see. To increase the chance of success, here is a repeat of the most important points:

1) Use a grade-42 or -52 neodymium magnet to extract the magnetic fraction.
2) Be prepared to extract the magnetic fraction from up to 500-1000 grams of sediment. Stop when you have found a representative number of microspherules.
3) Wash the magnetic fraction to remove the dust.
4) Spread the magnetic sample evenly across a microscope slide.
5) Search for microspherules at 100×-150× magnification on a white background with a top-lit microscope with a mechanical stage.

3) Extraction of Carbon Spherules, Glass-like Carbon, and Charcoal.

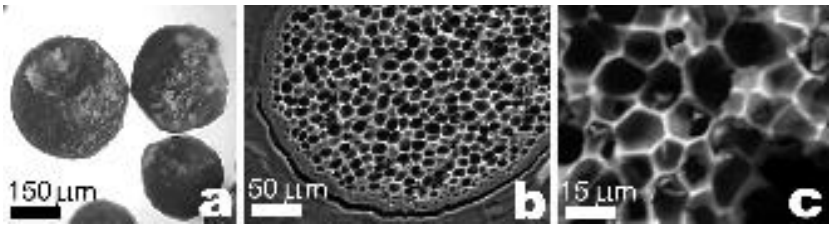

Fig. 15. Carbon spherules.

Fig. 16. Glass-like carbon.

- Carbon spherules have a low specific gravity, and water floatation was used to separate them. Ample water was used for dilution, and the slurry was agitated to free the floating fraction (arrows, Fig 17).

Fig. 17

- The floating fraction was captured with a 150-μm ASTM screen (Fig 18).

Fig. 18

- The floating fraction was placed onto a lab dish to dry (Fig. 19).

Fig. 19

- This was repeated until the entire floating fraction was removed.
- Then, to recover the less buoyant fraction of carbon that did not float, the remaining slurry was rinsed and agitated repeatedly. This stratified the sediment and brought the remaining non-floating carbon fraction to the surface of the sediment sample, but beneath the water. Obvious carbon, which included charcoal and glass-like carbon, was separated manually.

- The sample was then dried at room temperature, so as not to destroy the carbon.
- The carbon spherules were collected either visually or gravimetrically by agitating the dried sample on a smooth, inclined surface, down which they roll easily (Fig. 20).

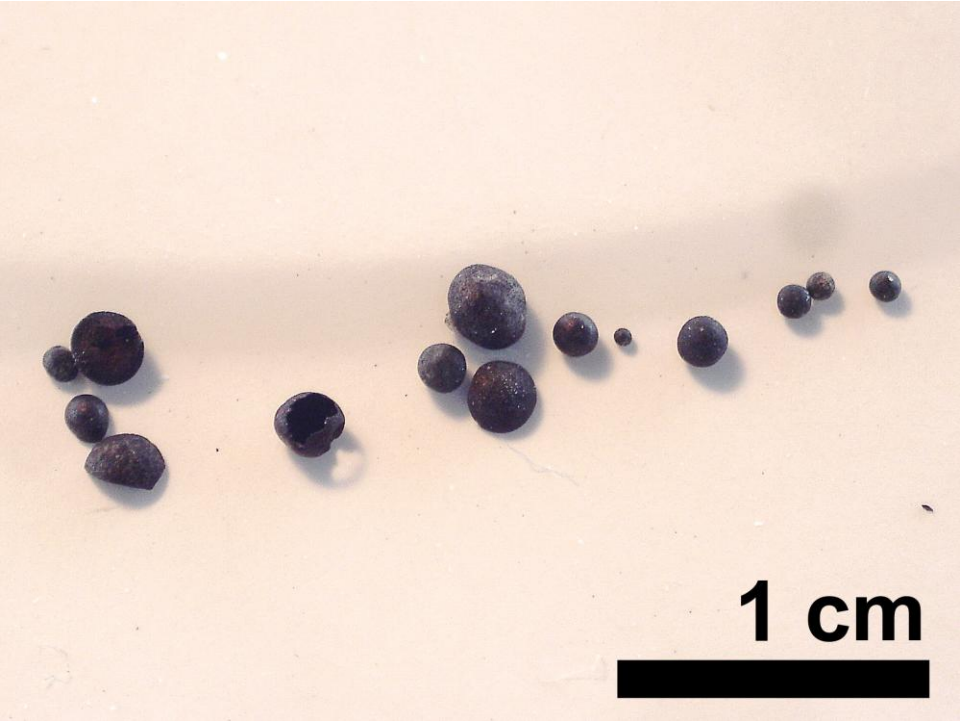

Fig. 20

- Glass-like carbon and charcoal, contained in the same sample, were identified visually and extracted manually, using a thin, sharpened, moistened, wooden probe.
- All three types of carbon were weighed separately, and abundances were calculated in #/kg for spherules or g/kg for charcoal and glass-like carbon.
- Carbon spherules and glass-like carbon were tested for fullerenes and helium-3. Other analyses, including PGAA, INAA, and MS-ICP, were performed to determine composition.
- Most of the carbon spherules and some of the glass-like carbon contain nanodiamonds. The diamonds were detected with TEM and ^{13}C-NMR. They were confirmed with selected area electron diffraction.