

The Murray Springs Clovis site, Pleistocene extinction, and the question of extraterrestrial impact

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Some of the evidence for the recent hypothesis of an extraterrestrial impact that caused late Pleistocene megafaunal extinctions [Firestone et al. (2007) *Proc Natl Acad Sci USA* 104:16016–16021] was based upon samples collected at Murray Springs, a Clovis archaeological site in southeastern Arizona. Here we describe sampling and analyses of magnetic separates from within, above, and below the lower Younger Dryas boundary (LYDB) black mat at Murray Springs, as well as radiation measurements from the LYDB at Murray Springs and two other well-stratified Clovis sites. The main magnetic fraction at Murray Springs is maghemite. Magnetic microspherules have terrestrial origins but also occur as cosmic dust particles. We failed to find iridium or radiation anomalies. The evidence for massive biomass burning at Murray Springs is addressed and found to be lacking. We could not substantiate some of the claims by Firestone and others, but our findings do not preclude a terminal Pleistocene cosmic event.

archaeology | cosmic dust | microspherules | radiation anomaly | biomass burning

Recently an extraterrestrial (ET) impact over North America has been proposed as the cause of Rancholabrean megafauna extinction, the onset of Younger Dryas (YD) cooling, the end of the Clovis culture, and extensive biomass burning across North America (1–4). One of the key sites providing their evidence is the Clovis site at Murray Springs in Curry Draw, Cochise County, of southeastern Arizona. The Clovis occupation surface at Murray Springs is a sharp stratigraphic contact upon which rests black organic clay (the black mat) that is believed to result from an algal bloom (5). The Murray Springs black mat covers and preserves the Clovis-age landscape. Hundreds of Clovis stone artifacts in direct association with skeletons of two mammoths, eleven bison, and bones of dire wolf and horse were exposed under the black mat by archaeological excavations (6). Charcoal of ash wood (*Fraxinus* sp.), probably originating from hearths, produced eight ¹⁴C dates that average 10,900 ± 50 B.P. (12,900 calendar years.). Radiocarbon ages of the black mat, stratum F₂, range from approximately 10,700 B.P. at the base to approximately 9700 B.P. at the top, indicating deposition during the YD cooling period, a reversal from the Bølling-Allerød warming trend (7, 8).

At Murray Springs the YD black mat (stratum F₂) is generally a 2- to 10-cm-thick layer of organic clay. At Profile B it covers a Clovis-age stream channel of sand and gravel (stratum F₁) as thin black stringers separated by several centimeters of white marl facies (Fig. 1). Away from the channel the black mat rises gently over an older surface where it is up to 10 cm thick at Trench 22 (9). The microstrata are shown in Fig. 2. On three occasions Haynes and Ballenger escorted Allen West and associates (1, 2, 4) to the Murray Springs site and collected sediment samples at, above, and below the LYDB at Profile B and Trench 22 North (Fig. 1).

After several analyses Firestone et al. (1) have reported that the Clovis surface at Murray Springs contains (a) a spike in magnetic particles, (b) including magnetic microspherules, (c) carbon microspherules, (d) fullerenes, (e) a helium-3 anomaly, (f) an

iridium anomaly, (g) a radiation anomaly, (h) abundant charcoal, and more recently, (i) nanodiamonds (4). This paper reports on our attempt to reproduce some of their most readily tested findings. Where they collected, we collected, and, therefore, we have essentially identical samples.

Here we report the concentration of magnetic separates from six samples collected from stratified alluvial sediments at the Murray Springs site, as well as comparative samples that include channel sands collected from the modern stream bed of Curry Draw (16MS07) and eolian fine sands and silts collected from a polymer-coated rooftop in Tucson, Arizona (1AZ07). The LYDB is represented in sample 9-5-3MS07, which includes about 0.5 cm either side of the contact (F_{2a1}/F_{1a2}) of the basal black mat, F_{2a}, and the top of the Allerød-age channel fill, F_{1a}, at Profile B₁ (Fig. 2A). Collection of sample 3-5MS07 was confined to the top 1.0 cm of the Clovis (F₁) stream channel sands. Sample 26MS07 included about 0.5 cm of basal black mat (F_{2a}) and about 1.0 cm of the top of stratum F_{1a} on either side of the F_{2a}/F_{1a} contact at Profile B₃ (Fig. 2B). Following Allen West (4), a fourth LYDB sample (7MS08) was collected from Trench 22 and included approximately 1.0 cm of sediment on either side of the F₂/D contact (Fig. 2C). Here the black mat rests on a much older surface, however, and we omitted the sample. Samples 1AZ07, 16MS07, 26MS07, and 3-5MS07 contained the magnetic particles and microspherules we examined using an electron microprobe and inductively coupled plasma-mass spectrometry (ICP-MS).

Firestone and others (1) also found a radiation anomaly at LYDB at Murray Springs and at the type Clovis site in Blackwater Draw, New Mexico. Using a Radiation Alert Monitor 4 Geiger Counter, 1-min counts were taken by the lead author at the Clovis occupation surface at two localities at the Murray Springs site (Profile B₁ and Trench 22), at Blackwater Draw and at the Sheaman Clovis site in eastern Wyoming. In addition 1-min counts were made at 5- and/or 10-cm intervals above and below the Clovis surface.

Results

Magnetic Separates. Sample 9-5-3MS07 was separated into 19 size fractions and the magnetic particles, 0.82% of the total, separated from each fraction. Magnetic particles are most concentrated in the silt to fine sand fractions (Table 1). Examination under 100× and 400× in both reflected and transmitted light revealed what appear to be subangular detrital magnetite grains and rock-forming silicates (quartz and feldspars) with a few silvery microspherules ranging in size from approximately 10 to 70 μm. They look like microscopic highly polished ball bearings (Fig. 3).

The percentage of magnetics separated from microstratigraphic context are shown in Fig. 4. It can be seen that the highest

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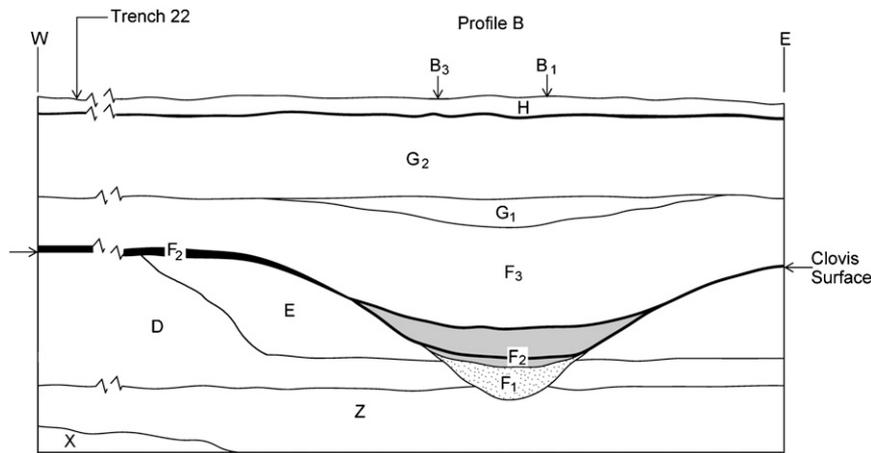


Fig. 1. Generalized stratigraphic cross-sections of Murray Springs site at Profile B in relation to Trench 22.

concentration (0.82%) occurs in the F_{2a1}/F_{1a} contact zone, the Clovis surface (Fig. 4A). However, sample 26MS07, also from the Clovis occupation surface (Fig. 4B), about 3 m away, had only 0.072% magnetics. Sample 3-5MS07 from the contact of F_{2a1} with F_{1a} at the top of the F_1 channel fill has the next highest percentage (0.56%) of magnetic particles. The lowest percentage of magnetics (sample 16MS02, 0.007%) occurs in the white marl of lower stratum F_{2b3} .

The F_1 channel sand sample 4MS07 has a low percentage (0.014) of magnetics, whereas sample 6MS08, about 12 cm lower, has an intermediate percentage of 0.15%. Because Stratum F_1 is a fluvial channel sand, much like the modern channel fill of Curry Draw, the amount of magnetite and maghemite is highly variable both vertically and laterally because of fluvial cut and fill dynamic equilibrium. The range of this variability has not been determined, but black streaks of magnetite are common; magnetic particles accounted for 14.1% of the modern channel sand sample (16MS07).

Microspherule Analyses. Two batches of the magnetics from sample 26MS07 were mounted on transparent tape attached to a cardboard pocket slide with a 13-mm-diameter pocket for micro-

scopic observation and counting using a mechanical stage. The first batch weighed approximately 7 mg and contained 104 microspherules; the second batch weighed approximately 3 mg and contained 411 microspherules. Portions of the magnetic particles of each batch were mounted in epoxy resin, ground, polished, and graphite-coated for electron microprobe analysis.

Sectioned microspherules were of two kinds; one type was solid, and the other was made of an aggregate of particles. Both types appear to be maghemite, with some being high in Ti and smaller amounts of Si and S being present in some examples. Using the same procedures as 26MS07, magnetic particles accounted for 3.32% of our rooftop sample (1AZ07). The slide count was 621 microspherules, some hollow, that had essentially the composition of maghemite but without Ti. These are probably airborne particles from diesel exhaust and emissions from coal-burning power plants in Tucson. Such findings are common around industrial areas (10–12) and reflect the multiple contexts in which microspherules are created and discovered.

ICP-MS Analyses. Portions of the magnetic fractions of 26MS07, 3-5MS07, the rooftop sediment (1AZ07), and channel sands (16MS07) from the modern streambed of Curry Draw were

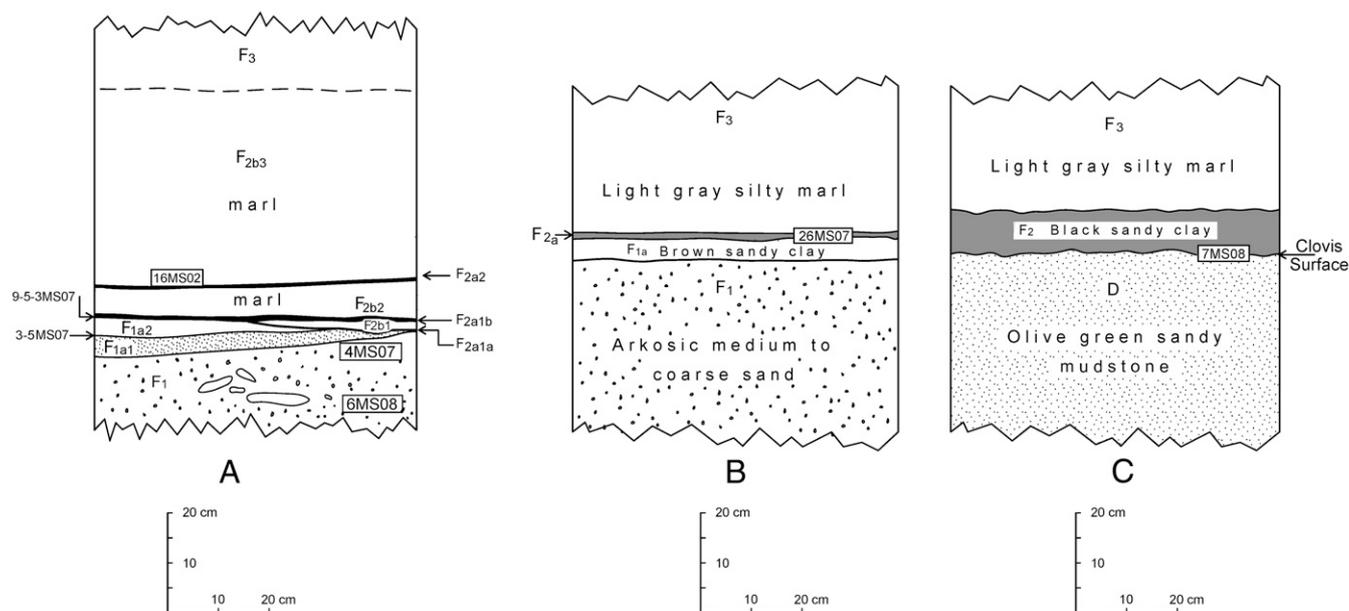


Fig. 2. Generalized microstratigraphic columns at (A) Profile B₁, (B) Profile B₃, and (C) Trench 22 north showing sample locations.

Table 1. Magnetic separates by size from LYDB sample 9-5-3 MS07.

ASTM mesh size	Weight, g	Weight of magnetics, g	wt %	Texture
+10	0.121	0.001	0.83	very fine gravel
+12	0.016	none	0.00	
+14	0.105	none	0.00	
+16	0.175	none	0.00	
+18	0.736	0.001	0.14	
+20	0.910	0.0005	0.05	
+25	1.205	0.002	0.17	medium-coarse sand
+30	1.399	0.002	0.14	
+35	1.919	0.013	0.68	
+40	1.778	0.012	0.67	
+45	1.540	0.007	0.45	
+50	1.290	0.008	0.62	
+60	1.374	0.006	0.44	
+70	1.063	0.019	1.79	
+80	1.217	0.018	1.48	very fine to fine sand
+100	0.757	0.019	2.51	
+200	3.974	0.045	1.13	
+325	1.145	0.027	2.36	silt
-325	1.594	0.004	0.25	
Totals	22.318	0.183	0.82	

analyzed by ICP-MS at the Lunar and Planetary Laboratory, University of Arizona. The analyses included iron, nickel, titanium, tungsten, iridium, uranium, and rare earths. Ti/Fe ratios of our LYDB samples range from 0.08 (26MS07) to 0.90 (3-5MS07). Similar values and variability are observed between our rooftop sample (0.02) and the modern stream bed sample from Curry Draw (0.72) (Table 2). The highest Ir value of 72 ppb is from the stream bed of Curry Draw and is over twice as high as the magnetics in sample 3-5MS07 from the top 1.0 cm of the Clovis-age stream bed. The LYDB at Profile B₃ (26MS07) has

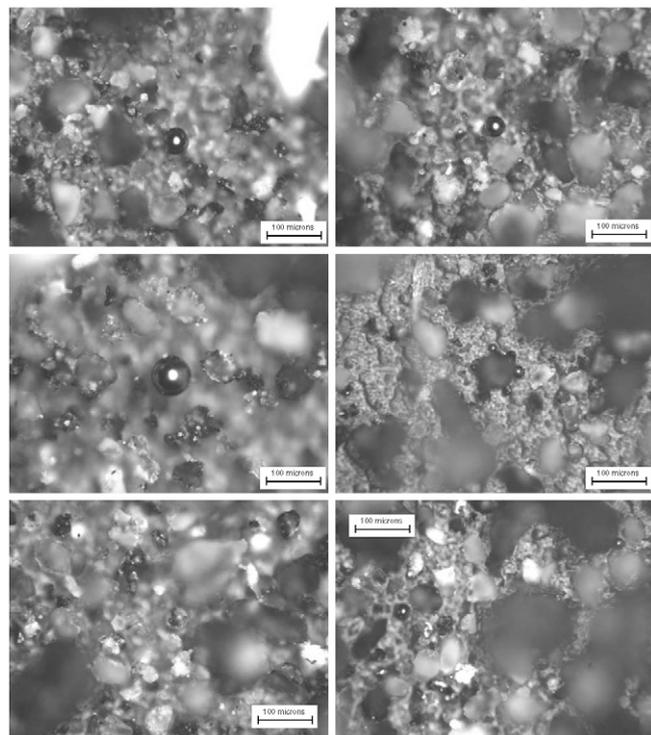


Fig. 3. Photomicrographs of magnetic microspherules in sample 26MS07 from the LYDB at Murray Springs.

64 ppb, statistically the same as the modern stream bed sample. The roof sample (1AZ07) has negligible Ir.

Discussion

One of the claims by Firestone and others (1) is a spike in Ti-rich magnetic particles at Murray Springs. The relative amounts of magnetic particles in sediments at the site are mainly a function of their local geological context and the rate of sedimentation. Magnetite and maghemite are ubiquitous in the late Quaternary alluvium of Curry Draw, including the bed load of the modern channel where magnetic particles concentrate as streaks of black sand (sample 16MS07).

Among the buried samples, 9-5-3MS07 contains the highest percentage (0.82%) of magnetics separated from the microstrata, and sample 3-5MS07 contains the second-highest percentage (0.56%) (Fig. 4A). However, sample 26MS07 is also from the LYDB (Fig. 2B) but contains as much as an order of magnitude less percentage (0.072%) of magnetic particles, probably because of variability in magnetite concentrations in the Clovis sands similar to the modern channel. Sample 16MS02 from the F_{2b3} marl at Profile B (Fig. 2A) had practically no magnetic particles (0.007%) because the marl, essentially a chemical precipitate of CaCO₃, was more or less continually aggrading, thereby diluting the accumulation of magnetic particles.

Firestone et al. (1) report a Ti/Fe ratio of 0.76 from magnetic particles from Murray Springs, a value they consider to be anomalously high and indicative of a Ti-rich terrestrial source or a new and unknown type of impactor. Our LYDB sample 3-5MS07 shows a similarly high ratio of 0.90, but so does our sample 16MS07, which was collected from the modern stream channel and has a ratio of 0.72 (Table 2). Microspherules from the rooftop sample lacked Ti. Franzén and Cropp (13), in their discussion of microspherules from Scandinavian peat bogs, describe criteria for distinguishing terrestrial from ET sources based upon proportions of rare-earth elements. In our samples, europium (Eu) is depleted with respect to gadolinium (Gd) and samarium (Sm), suggesting that most of the Murray Springs magnetic particles are derived from continental rocks rather than meteoritic material (Table 2).

Recently, Surovell et al. (14) were unable to replicate a concentration of magnetic particles or spherules at the LYDB at seven sites, arguing instead that parent material, surface stability, and fluvial sorting are largely responsible for the distribution and accumulation of magnetic particles. Additionally, Haynes (15) has drawn attention to the potential for cosmic dust (16, 17) as a source for some of the magnetic microspherules or the framboidal particles in the Murray Springs sediments. Cosmic dust derives from the ablation of meteoritic bodies, cometary dust, terrestrial impact, and passage of the solar system through an interstellar cloud (18, 19). Between 4,000–10,000 t of cosmic dust is thought to land on the earth’s surface annually (20). By some estimates, a square meter collects about one 10-µm particle per day and one 100-µm particle per year (17, 21).

Because of the unknown contribution from terrestrial sources and because our microspherule count has not been systematic, we cannot estimate or evaluate the number per unit area with any confidence. Sample 26MS07 came from an area of approximately 200 cm² or 0.02 m². This would be equivalent to about twenty 10-µm particles per 1 m² per century (21). The Clovis surface over the top of the F₁ channel probably was stable for less than a decade before burial (9). However, the farther one gets away from the channel the older the erosional surface becomes as it rises up the sides of the valley, and, therefore, the more time there is for accumulation of cosmic dust. The Clovis surface at the top of the F₁ channel fill might be expected to have more cosmic dust than the sides of the valley if it concentrated there during activation of the Clovis channel. Cosmic dust therefore cannot be overlooked as a reasonable explanation for the presence

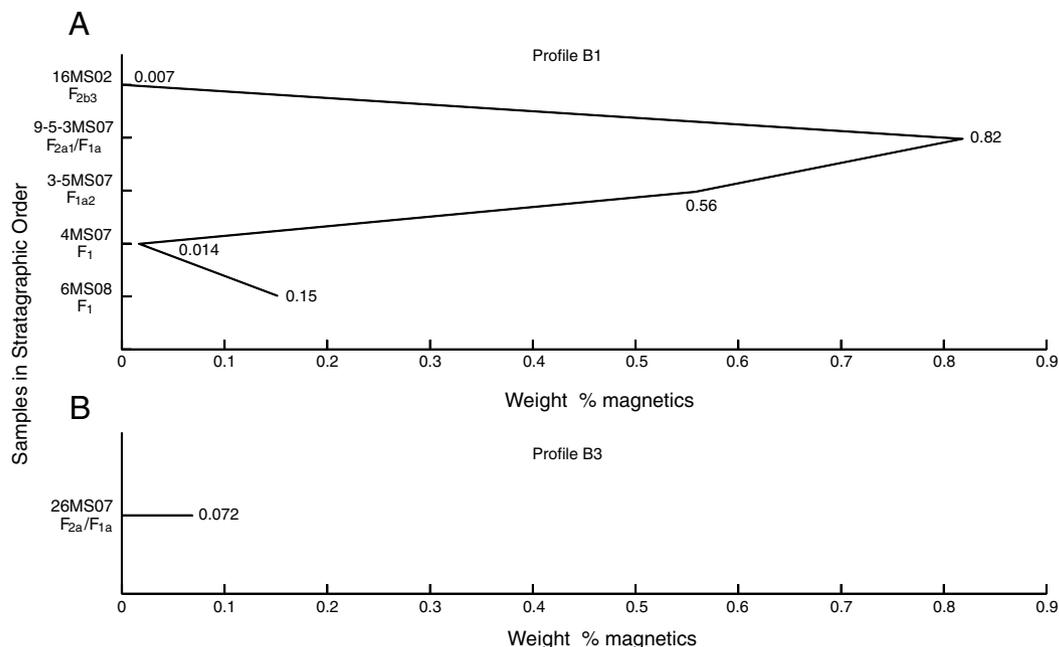


Fig. 4. Magnetic particle percentages from (A) Profile B₁ and (B) Profile B₃ showing LYDB samples 9-5-3MS07, 3-5MS07, and 26MS07.

of an ET component, including nanodiamonds (20, 22, 23), from the Murray Springs strata.

Radiation Anomaly. Radiation background varied from 22 cpm at Murray Springs to 22.7 cpm at Blackwater Draw. By placing the Geiger tube window against the stratigraphic surface counts ranged from a low of 18 cpm to a high of 37 cpm at Murray Springs Trench 22 North (Fig. 5B). The Clovis surface (F₂/D₁ contact) measured 27 cpm. At Profile B₁, only three counts were made, giving readings of 25 cpm at the Clovis surface (F_{2a1}/F₁), 27 cpm at 10 cm above, and 22 cpm at 10 cm below (Fig. 5A).

At Blackwater Draw, radiation ranged from 18 to 37.6 cpm, with the highest reading being at the Clovis occupation surface

(strata D/C contact) on the basis of the average of five individual 1-min counts (Fig. 5C). However, as Fitting (24) reported, uranium mineralization in bones at the Blackwater Draw Clovis site is due to ground water mineralization and not confined to any stratigraphic contact. The Sheaman site results (Fig. 5D) varied from 20 to 31 cpm across the Clovis surface (strata D/C contact). The higher readings at all of these sites are probably due to variations in detrital radioactive minerals such as allanite or monazite (25). Variations from 0.5 to 2.0 times background counts do not appear to be anomalous.

Biomass Burning. Kennett et al. (3) claim on the basis of the Stankiewicz and Tegelaar report in Haynes (5) that the presence

Table 2. ICP-MS analysis of magnetic separates.

Element	Unit	Tucson roof silt magnetics		Curry Draw streambed magnetics		Clovis surface F ₂ /F ₁ Profile B3 magnetics		Approximately 1 cm below Clovis Surface—F ₁ sand surface	
		1AZ07	2σ	16MS07	2σ	26MS07	26	3-5MS07	2σ
Fe	wt %	45.3	1.2	9.6	0.4	27.1	0.8	5.7	0.2
Ti	wt %	0.8	0.1	6.9	0.2	2.2	0.1	5.1	0.5
W	ppm (μg/g)	202	11	794	9	496	64	1132	85
Ni	ppm (μg/g)	6	5	53	25	67	32	28	20
La	ppm (μg/g)	19	15	124	60	38	28	347	219
Nd	ppm (μg/g)	20	3	95	5	27	2	225	16
Sm	ppm (μg/g)	3.6	1.7	18	2	4.8	1.6	35	3
Eu	ppm (μg/g)		BDL*	0.2†	3.5		BDL	1.2†	4.3
Gd	ppm (μg/g)	5.7†	7.2	22	11	5.0†	8.1	34	26
Tb	ppm (μg/g)	0.6†	1.4	2.7	1.5	0.4†	1.4	4.6	1.6
Dy	ppm (μg/g)	4.4†	4.9	16	4	3.3†	3.7	26	4
Ho	ppm (μg/g)		BDL	2.1†	3.5		BDL	3.8†	3.8
Er	ppm (μg/g)	1.2†	4.5	8.5	7.7	0.1†	4.2	16	6
Tm	ppm (μg/g)	0.2†	0.9	1.9	1.3	0.1†	0.8	2.6	1.0
Yb	ppm (μg/g)		BDL	8.5†	25.9		BDL	21†	54.0
Lu	ppm (μg/g)		BDL	13.4†	16.6	6.6†	10.5	5.4†	6.3
Hf	ppm (μg/g)	29	1	192	8	155	12	124	3
Ta	ppm (μg/g)	83	4	709	35	73	2	710	30
U	ppm (μg/g)	6.3	0.6	9.1	0.8	3.1	0.6	7.7	1.9
Ir	ppb (ng/g)	0.9†	34	72	30	64	22	31	29

*BDL: sample signal below blank.

†Sample signal overlaps with blank at 2σ level.

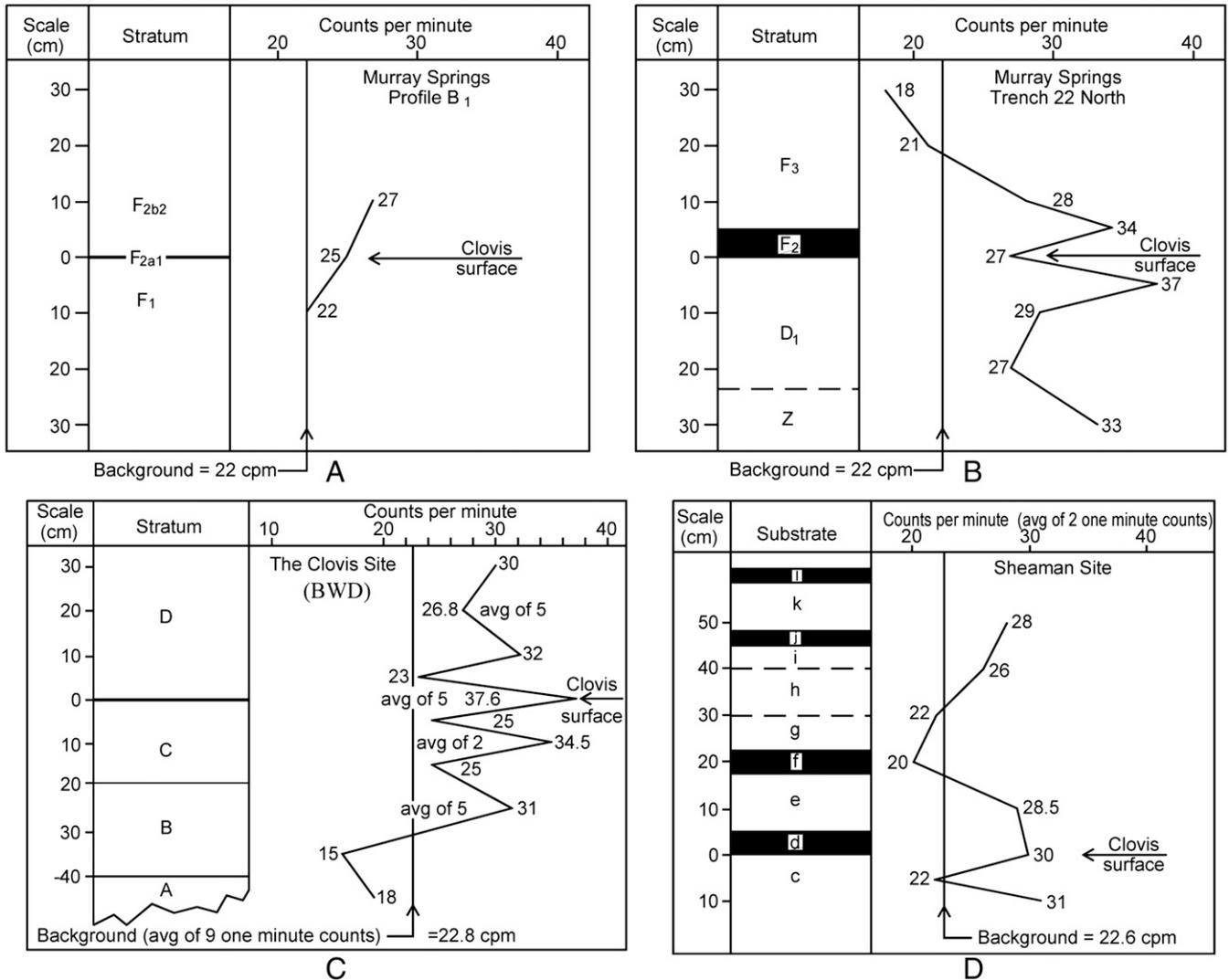


Fig. 5. Radiation counts per minute at (A) Murray Springs Profile B₁, (B) Murray Springs Trench 22N, (C) Blackwater Draw Clovis site, New Mexico, and (D) Sheaman Clovis site, Wyoming.

of charcoal, vitreous carbon, and vitrinite in the Murray Springs black mat (Stratum F₂) indicates there was intense biomass burning during the onset of the YD. The sample that provided this information (6AZ90) was just one of at least three others that did not yield such material. Over the past four decades the lead author has chemically pretreated hundreds of black mat samples for multifraction ¹⁴C dating (15). Very few YD-age black mats were found to contain adequate charcoal. The vitreous carbon, vitrinite, and charcoal in sample 6AZ90 are probably all a result of burning, but their amounts are miniscule in the sample, which was collected 6 m from a Clovis hearth (6). The sample's combustion products are probably derived from this hearth or possibly others nearby. Thus, there is no compelling evidence for extensive YD-age biomass burning at Murray Springs or any of the other Clovis sites in the San Pedro Valley of Arizona. On the basis of charcoal counts in pollen cores, others (26, 27) do not find evidence of continent-wide fires at the onset of the YD.

Conclusions

From the data presented here we find no compelling evidence for a cosmic catastrophe at the Murray Springs Clovis site. The stratigraphic distribution of magnetic particles and the included microspherules can be explained by the fluvial dynamics affecting these sediments, some of which originated from the normal flux

of cosmic dust to our planet as suggested by Pinter and Ishman (28). We do not observe a LYDB Ir anomaly relative to the local geological background. The careful chemical pretreatment of dozens of basal YD black mat samples from the upper San Pedro Valley also fails to find adequate charcoal to support the hypothesis of extensive biomass burning.

We retain the opinion that we have yet to understand what happened approximately 12,900 calendar years ago that abruptly terminated the major elements of Pleistocene megafauna (15). The ET impact hypothesis has sparked several investigations that are leading to new knowledge, and although our data from Murray Springs and elsewhere do not support it, neither do they preclude it.

Materials and Methods

Magnetic Separation. After sieving for the removal of coarse particles (>1.4 mm) and lumps of black clay (stratum F_{2a1}), and flotation for the removal of mostly rootlets and insect debris, the magnetic particles were separated with a 0.5 × 1 × 2-in. neodymium (Nd) magnet covered with shrink wrap. This was patted up and down on the sediment under water and patted systematically across the pan from one side to the other and top to bottom and then again at right angles to the first passes until the magnet had accumulated all the particles it could. The magnet was then moved to a dish of water and removed from the shrink wrap so the magnetic particles would settle to the bottom of the dish. This was repeated eight times, a point of

significant diminishing returns. The dry magnetics of sample 26MS07 weighed 0.243 g. The remaining sand was washed by decantation to separate clays and fine silt. The sand and silt, after being placed in water in a 40-cm-diameter plastic pan, was again subjected to magnetic separation 10 times. This yielded 0.118 g of magnetic particles for a total of 0.361 g, or 0.072% by weight. Both samples 9-5-3MS07 and 26MS07 revealed magnetite, rock-forming minerals, and a few microscopic spherules.

The material that floated in sample 26MS07 was subjected to magnetic separation and yielded 0.003 g, raising the percentage to 0.073%. Some magnetic particles were probably not recovered from the very fine grained material in suspension. The flotation material under 400× magnification revealed several blue-black shiny microspheres among irregular blue-black grains (magnetite?), black carbon microspherules, and much organic debris. The metallic microspherules may have been caught up in the debris that floated or they may be hollow (18). In attempting to replicate the methods of Firestone et al. (1), we find that the separation of magnetic particles with a hand magnet is as much art as it is science (14). For uniformity in comparing datasets, using an electromagnetic separator would provide better reproducibility.

Microspherule Analysis. Microspherules were analyzed using a Keck electron microprobe at the Lunar and Planetary Laboratory of the University of Arizona.

ICP-MS Analysis. The bulk composition of Murray Springs samples was determined using acid-dissolution ICP-MS. The sample solution for ICP-MS was prepared by placing the samples in Teflon digestion vessels with a solution containing 1 mL concentrated HNO₃ and 0.5 mL concentrated HF. The Teflon vessels were sealed and heated at 130 °C overnight. After cooling, visual inspection revealed that the samples did not completely dissolve. An additional 0.5 mL of concentrated HCl was added to all vessels, which were again sealed and allowed to digest overnight. An analytical blank solution was prepared using the same procedure. After dissolution, solutions were evaporated at 110 °C until only a small drop of solution remained. This solution was diluted with 5% HNO₃, yielding a solution with approximately 30 µg/ml total dissolved solids.

Solution standards were prepared that consisted of known amounts of the elements of interest using single-element solutions obtained from

High-Purity Standards (Charleston, SC). The initial standard solution contained 6 µg/ml Fe, Ni, La, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, and U; 2 µg/ml Ti; and 1 µg/ml Ir. This initial solution was diluted to produce three additional standard solutions containing 0.6, 0.06, and 0.006 µg/ml of most elements; 0.2, 0.02, and 0.002 µg/ml Ti; and 0.1, 0.01, and 0.001 µg/ml Ir. A standard-blank solution was prepared at the same time using successive dilutions of the 5% HNO₃ standard-carrier solution. All blanks, standards, and sample solutions were spiked with 1 ng/ml Rh solution, which served as an internal standard to monitor the instrument response over the course of the analysis.

Analyses were performed using an ELEMENT2 HR-ICP-MS. The following ions were monitored in low-resolution mode: ⁶¹Ni⁺, ¹³⁹La⁺⁺, ¹⁵³Eu⁺⁺, ¹⁵⁷Gd⁺⁺, ¹⁵⁹Tb⁺⁺, ¹⁶³Dy⁺⁺, ¹⁶⁵Ho⁺⁺, ¹⁶⁷Er⁺⁺, ¹⁶⁹Tm⁺⁺, ¹⁷³Yb⁺⁺, ¹⁷⁵Lu⁺⁺, ¹⁰³Rh⁺, ¹⁴⁶Nd⁺, ¹⁴⁷Sm⁺, ¹⁸⁰Hf⁺, ¹⁸¹Ta⁺, ¹⁸²W⁺, ¹⁹³Ir⁺, and ²³⁸U⁺ (Table 2). Many of the rare-earth elements were analyzed as doubly charged ions to eliminate interferences with oxides. In addition, ⁴⁷Ti⁺ and ⁵⁷Fe⁺ were analyzed in medium-resolution mode to eliminate interferences and prevent overly large signals from saturating the ion detector. Sample concentrations were determined by first subtracting blank signal intensities from those obtained from the sample and standard solutions. A calibration curve was obtained by performing a linear least-squares regression for each element using the blank-subtracted counts and the known concentrations in each standard solution. In all cases, the regression coefficients were 0.997 or higher. Uncertainties were calculated by taking the square root of the sum of the squares for the standard deviations on both the sample and blank solutions and are reported at the 2σ level.

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