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AN ATLAS OF EXTRATERRESTRIAL PARTICLES

COLLECTED WITH NASA U-2 AIRCRAFT - 1974-1976

D. E. Brownlee and D. Tomandl University of Washington Seattle, Washington 98195

M. B. Blanchard and G. V. Ferry Ames Research Center Moffett Field, California 94035

and

F. Kyte San Jose State University San Jose, California 95192



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16. Abstract

Since March 1974, extraterrestrial particles have been collected from the stratosphere on flights with U-2 aircraft. Over 150 extraterrestrial particles (> 3 μ m and <40 μ m in size) have been collected from 1.4×10^5 m³ of stratospheric air. The majority of these particles have relative Mg, Fe, Si, C, S, Ca, and Ni abundances within a factor of 2 of C₁ and C₂ carbonaceous chondrites. Because no known terrestrial or lunar material matches the composition for these seven cosmically abundant elements, this is a strong diagnostic criterion for identifying extraterrestrial material. A final proof of the extraterrestrial origin for some of these particles is the occurrence of solar wind implanted helium.

These extraterrestrial particles have been divided into 4 groups: chondritic (60%), iron-sulfur-nickel (30%), mafic silicates (10%), and others. The chondritic type particles can be subdivided into aggregates and ablation products. The chondritic aggregates have grain sizes of 1,000 Å and their intergranular packing ranges from porous to nonporous. The carbon content in 5 of these particles is 2-15%. Their typical elemental composition is Fe, Mg, Si, C, S, Ca, and Ni. Detectable levels of ⁴ He implanted from the solar wind occurs in 5 of these particles. Minerals identified include: olivine, spinel, pyrrhotite (?) and a hydrated layered-lattice silicate (?). The chondritic ablation particles are not porous, contain no sulfur, and their shapes imply they have been melted. In texture, composition, and minerology, these particles resemble fusion crusts of chondritic meteorites. Minerals identified include: magnetite, olivine, and pyroxene. The iron-sulfur-nickel type particles resemble meteoritic iron sulfide with a small amount of nickel. The sulfur content is deficient relative to stoichiometric FeS. Minerals identified include mixtures of magnetite and troilite. The mafic silicate type particles are iron-magneisum silicate grains with clumps of chondritic aggregate particles adhering to their surfaces. Minerals identified include: olivine, spherules. Minerals identified include and pyroxene (?). The iron-nickel type particles have Ni/Fe ratios between 0.05-0.4. Most are spherules. Minerals identified include taenite and wustite. The other type particles are characterized by the occurrence of micron-size nickel-iron (Ni/Fe ≈ 0.05) mounds on spheroidal glassy-like grains having approximate chondritic-like elemental abundances. These particles are not ablation debris because the nickel-iron mounds could not have been produced without oxidizing the iron.

This atlas contains 27 figures of representative particles from each of these four groups. Along with each particle is an x-ray emission spectra and information describing the mineralology.

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AN ATLAS OF EXTRATERRESTRIAL PARTICLES

COLLECTED WITH NASA U-2 AIRCRAFT - 1974-1976

D. E. Brownlee and D. Tomandl Astronomy Department, University of Washington Seattle, WA 98195

> M. B. Blanchard and G. V. Ferry NASA-Ames Research Center Moffett Field, CA 94035

> > F. Kyte San Jose State University San Jose, CA 95192

In March 1974 a program was undertaken to collect extraterrestrial particles with NASA U-2 aircraft. Using an inertial impaction collector, over 150 extraterrestrial particles were collected from $1.4 \times 10^5 \text{ m}^3$ of stratospheric air. This atlas is a compendium of scanning electron microscope (SEM) pictures and results of analytical studies performed on a representative sample of those particles. Included with the pictures in section V of this report are x-ray emission spectra taken in the SEM.

I. COLLECTION TECHNIQUES

Particles are collected by ramming a 20 cm² (5 cm x 4 cm) oilcoated collection surface through the ambient air at the aircraft's cruise velocity of 200 m/s. Particles larger than ~ 3 µm are collected by inertial deposition while those <1 µm follow flow lines around the impaction surface and are not collected. Particle bounce-off is prevented by coating the collection surface with a >10 µm coating of 5 x 10⁵ centistokes silicone oil.

The basic collector was designed at Ames (Ferry and Lem, 1974) for collecting submicron stratospheric aerosols. For collection of extraterrestrial particles, minor modifications were made so that larger surface areas could be exposed.

II. ANALYTICAL PROCEDURE

Collected particles are analyzed by individually removing them from collection surfaces, mounting them on special surfaces for SEM analysis and then washing them with xylene to remove the silicone oil. The particles are then analyzed in the SEM for morphology and relative elemental abundances as determined with a solid state Si(Li) x-ray detector. By raster scanning on the portion of the particle facing the x-ray detector and calibration with mineral standards (similar to the unknowns), elemental ratios are routinely determined to an accuracy better than a factor of two.

A number of particles have been mounted on micron-size glass fiber and exposed to x rays in modified Debye-Scherrer pcwder diffraction cameras. Particles were exposed in both a 57.3 mm diameter camera with a continuous helium-purged atmosphere, and a 28.7 mm diameter camera insert placed inside a 57.3 mm diameter camera which was evacuated during exposure to x rays. Exposure times for these particles were typically 120-168 hours. Both Cr and Cu radiation was used depending upon the amount of Fe in the particle (Fe fluoresces with Cu radiation darkening the film) and the wavelength range of the diffraction pattern to be recorded on the film. Identifications were made using both overlays of x-ray diffraction patterns from known mineral standards and version 10 (Kyte and Blanchard, 1975) of the Johnson-Vand computer program for identification of x-ray diffraction patterns.

Some particles were crushed and mounted on thin carbon films for transmission electron microscopy to observe crystallite morphology in the 50-1,000 Å size range.

III. THE COLLECTED PARTICLES

Six hundred particles in the 2 μ m - 40 μ m size range were removed from collection surfaces and analyzed in the SEM. The vast majority of

these particles were aluminum oxide, a common stratospheric aerosol produced by solid fuel rockets (Brownlee et al., 1976). Disregarding particles which are largely aluminum, however, more than half of the collected particles have elemental abundances which closely match bulk abundances of primitive meteorites or minerals which are common in C1 and C2 carbonaceous chondrite meteorites. These particles have compositions uniquely different from obvious stratospheric, laboratory, and aircraft contaminant particles (Al particles, skin flakes, TiO₂ paint, Cd plating, etc.)

Particle Groups. On the basis of elemental abundances we have identified >150 particles from the U-2 flights which we believe are extraterrestrial. The majority of these particles have relative Mg, Fe, Si, C, S, Ca, and Ni abundances within a factor of 2 of C1 and C2 carbonaceous chondrite meteorite abundances (Mason, 1971). Importantly, we have not detected even small quantities of elements which were not cosmically abundant (i.e., Cu, Cl, Zn, Cd, etc.). Because no known terrestrial (or lunar) material matches the composition for these seven cosmically abundant elements, we feel this is a very strong diagnostic criterion for identifying extraterrestrial material. The particles which closely match cosmic abundances we refer to as "chondritic." No genetic association with chondrules is intended.

In addition to the chondritic particles other composition groups have been identified as extraterrestrial by their physical association with chondritic particles. These composition groups have been found as single parcles, as particles with chondritic material adhering to their surfaces, as particles imbedded in single chondritic particles, and as particles found

inside chondritic partic in hich broke into fragments during collection or were intentionally crushed in the lab. From the observed associations we believe that all extraterrestrial particle groups identified were at one time in intimate contact with each other. A final proof of the extraterrestrial origin of these particles was the recent detection of large and the soft solar wind implanted He in some of the particles.

We have defined four major compositional groups into which nearly all of the collected extraterrestrial particles can be placed. Sixty percent of the particles classify as chondritic, 30% as iron-sulfur-nickel, and 10% as mafic silicates. The properties of these groups follow.

A. <u>CHONDRITIC - (Chondritic Elemental Abundances</u>) Chondritic particles have chondritic (some exceptions) elemental abundances. Based on differences in morphology and S abundance, the chondritic particles fall into two subgroups: chondritic aggregates, and chondritic ablation.

<u>Chondritic Aggregate</u>. Ninety percent of the chondritic particles are aggregates of 1,000 Å sized grains. Typically, the aggregates are compact with little pore space. However, in some particles the component grains are loosely bound and the particle structure is quite porous. These aggregate particles typically have chondritic abundances (within a factor of 2) for Fe, Mg, Si, C, S, Ca, and Ni (Brownlee et al., 1976). Mn and Cr can often be detected at concentrations approaching the limits of detection. Half of the particles have carbon contents >5%. Optically the aggregates are very black, undoubtedly the result of high carbon contents.

Six chondritic aggregate particles were analyzed for carbon in an ARL electron microprobe. During analysis the carbon content of the particles was observed to decrease with time. After analysis all the particles were surrounded with large halos of condensed carbonaceous material from the particles. Apparently some of the carbon in the particles is in a volatile organic form. The results from the carbon analyses are listed in Table 1.

Table 1

Particle	<u>% C</u>
SP-4	2.2
SP-5	5.8
SP-6	13.5
SP-7	2.7
SP-8	> 15.1
MP-1	2.3

Ten chondritic particles were analyzed for ⁴He at Atomics

International, Canoga Park, California. Five of the particles contained detectable ⁴He at levels of $\approx 10^{-2}$ cm³ g⁻¹. The highest ⁴He concentration was $> 10^{-1}$ cm³ g⁻¹. This is a level of ⁴He higher than most gas-rich meteorites but comparable to typical lunar soils. The ⁴He is undoubtedly implanted solar wind and proves that the particles are not only extraterrestrial but, also, that they are true micrometeorites. Those particles without ⁴He presumably outgassed during atmospheric entry.

X-ray diffraction patterns from four of these particles show strong lines of a spinel phase (either Fe_3O_4 or $MgFe_2O_4$), and a sulfide which is probably pyrrhotite (FeS or $Fe_{1-x}S$). In addition, two of these patterns include lines from olivine. Because of elemental composition, the major phase must be a silicate but evidently it produces only weak lines and has not yet been identified. In one particle a 7 Å line suggests evidence for the existence of a hydrated layer-lattice silicate phase. X-ray powder patterns from these particles are very similar to powder patterns obtained from matrix material from the Murchison (C2) meteorite which had been heated to $450^{\circ}C$ (Fuchs et al., 1973).

Four chondritic aggregate particles have been crushed and mounted on thin carbon films for transmission electron microscopy (TEM). The particle structures observed in TEM are highly diverse and complex. Many of the 1,000 Å sized grains seen in the SEM are actually aggregates of much smaller grains (50 Å - 500 Å) as seen in the TEM. Many of the larger grains are covered with 300 Å coatings of a low atomic weight amorphous material (carbon?). These coatings have not been observed on meteorite samples used as controls.

At least 50% of the grains in the particles are crystalline and many of the grains produce good electron diffraction patterns. A few of the grains are euhedral. Several hexagonal platelets have been observed which are opaque to the electron beam (pyrrhotite?).

Direct comparison of the chondritic aggregates with the matrix of type 1 and 2 carbonaceous chondrites suggests that they are different. The carbonaceous chondrite matrix material consists largely of a layer-lattice

silicate which in the TEM has the appearance of crumpled foils and fibrous masses. Related textures have been observed in the stratospheric particles but only rarely. The stratospheric particles are aggregates of more-or-less equidimensional grains with widely diverse properties. An additional apparent difference is that the hexagonal electron diffraction patterns (with large d-spacings) frequently observed in carbonaceous chondrites have not been observed for the several hundred grains examined from the stratospheric particles.

<u>Chondritic Ablation</u>. Ten percent of the particles with chondritic compositions are spherules (or spheroids) which are not porous and do not contain sulfur. The particle shapes imply the particles were molten at one time. The absence of sulfur is probably the result of thermal alteration. X-ray diffraction of one particle revealed a composition of magnetite and olivine. In composition, texture, and mineralogy these spherules are very similar to fusion crusts of chondritic meteorites (Blanchard and Cunningham, 1974). We believe that these particles experienced ablation during atmospheric entry (Brownlee et al., 1975). The one particle analyzed with x-ray diffraction has a mineralogical composition of magnetite, olivine (~Fo 60), and pyroxene (probably enstatite).

B. IRON-SULFUR-NICKEL - (An iron-sulfur mineral with a few percent

<u>nickel</u>). Iron-sulfur-nickel (Fe-S-Ni, or FSN) particles are roughly similar to meteoritic troilite or pyrrhotite containing a few percent Ni. In many of the particles, sulfur is deficient relative to stoichiometric FeS by factors of 50% (sometimes more). The FSN particles may be related to the poorly

characterized Fe, S, O, and Fe, S, C phases reported in carbonaceous chondrites (Fuchs et al., 1973), or they may be mixtures of FeS and Fe_3O_4 . X-ray diffraction of a single 8 µm spherical FSN particle showed it to be a mixture of magnetite and troilite.

Unlike the chondritic particles, the FSN particles come in a wide variety of forms. The majority of these particles are spheres, but they also have been found as solid irregular masses, aggregates, well-defined single crystals (octahedron with cubic truncation), and stacks of platelets. Some of the nonspherical FSN particles show remarkable similarities to forms of magnetite found in Cl meteorites (Jebwab, 1971). The FSN spheres may be ablation debris, but the irregular shapes are probably not.

C. <u>MAFIC SILICATES</u> - <u>(Olivine or Pyroxene)</u>. These particles are iron-poor olivine and pyroxenes with clumps of chondritic aggregates adhering to their surfaces. One euhedral crystal has been found, but typically they are subhedral to irregular. Eight of the particles have elemental abundances similar to pyroxene and eight to olivine. The x-ray diffraction pattern taken from one particle was identified as a coarse-grained olivine (FO 70) with minor phases tentatively identified as pyrrhotite and pyroxene.

IRON-NICKEL. Seven particles have been collected in which only Fe and Ni were detected. Ni to Fe ratios fall within the 0.05 to 0.10 range except for one particle which has a ratio of 0.4. Most of the particles are spheres and are almost certainly ablation debris. Two of the Fe,Ni particles are irregular with very odd shapes and possibly indicative of ablation. Because the FSN particles show a continuous trend of S/Fe ratios approaching

zero, it is quite possible that the Fe,Ni spheres are FSN particles which have experienced total depletion of sulfur during ablation. The x-ray diffraction pattern taken from one spherical particle contained taenite (Fe,Ni) and wustite ($Fe_{1-x}O$). The presence of wustite is sufficient proof (Davis, 1976) that it has experienced ablation and is extraterrestrial.

E. <u>OTHER PARTICLES</u>. Three particles were collected which are covered with Ie,Ni (Fe/Ni ~ 20) mounds (one micron and smaller in size). Two of these are spheroidal, glassy-like objects with approximate chondritic abundances. These particles are morphologically very similar to glassy agglutinates found in lunar coils. The third particle is a very strange porous particle composed of Fe,S and Si which is covered with large numbers of Fe,Ni mounds. These particles are probably not ablation debris because it is not possible that small metallic Fe mounds could be produced by ablation in an atmosphere containing oxygen without being oxidized to an iron oxide. Because of similarities to impact-produced features in lunar soils, we believe that these particles may have been produced by meteoroid collisions in space

The FSN and most of the mafic silicate particles are believed to be extraterrestrial because they have been found in physical association with (e.g., actually inside) chondritic aggregate particles. Other particle types, although rare, have been found in crushed chondritic aggregates. For example, on one flight a chondritic aggregate particle was collected that broke into ~ 100 fragments upon impacting the collection surface. Most of the fragments were small pieces of chondritic aggregate material but also found in the debris were FSN particles, enstatite, olivine, an opaque high Si mineral (possibly SiC) and two fragments of a Si, Al, Ca, Ti mineral.

Other extraterrestrial particle types probably exist in our collections but have not been identified either because they have not been found in physical association with the three major cosmic dust groups, or because they do not have distinctive compositions. Since almost all of the collected particles are either high Al particles, identified micrometeorites, or obvious contaminants, we believe other cosmic dust types probably constitute only a minor component (<10%) of the extraterrestrial particles found normally in the stratosphere.

IV. SUMMARY

The U-2 collections indicate that the flux of extraterrestrial dust in the state sphere is 3×10^{-6} particles m⁻²s⁻¹ (diameter $\geq 10 \ \mu m$). In the 2-30 μm size range most of the particles are true micrometeorites and have not melted during atmospheric entry. Although a variety of particle types has been observed it presently appears that they could be genetically related and derived from a common parent body type because of their association. The parent body matrix appears to consist of an opaque fine-grained matrix material containing minor amounts of inclusions. The matrix is an aggregate of 1,000 Å sized grains whose cumulative composition is close to cosmic abundances; it is very black and contains $\geq 5\%$ finely dispersed carbon. Imbedded in the fine-grained matrix are occasional micron-sized inclusions, primarily Ni-bearing iron sulfides (similar to troilite) and olivines and pyroxenes with compositions clustering towards forsterite and enstatite. The only known materials which are similar to these recovered

cosmic dust particles in terms of elemental abundance, texture, mineralogy, and inclusion content are type 1 and the matrix of type 2 carbonaceous chondrite meteorites.

The following properties of the micrometeorite parent material indicate a strong similarity to C1 and C2 carbonaceous chondrites and strong differences from other meteorite types:

- (1) Extremely fine-grained particle size
- (2) High carbon abundance
- (3) Low iron, olivine, and pyroxene abundance
- (4) Magnetite content
- (5) Nickel-bearing iron sulfides

The major difference between the micrometeorite material and Cl meteorites is - the detailed morphology of the fine-grained material, and the existence of mineral types not observed in Cl's.

V. INTERPRETATION OF X-RAY SPECTRA SHOWN IN FIGURES 1-27

The x-ray emission spectra included in this atlas were made in the SEM with an EDAX solid state x-ray detector. X-ray emission from the particles was produced by bombardment with a 20 keV electron beam. The beam was raster scanned on the particle half facing the detector, producing an averaged analysis with an effective take-off angle of $\sim 45^{\circ}$.

The spectra are displayed as x-ray photon counts (Y axis) vs. x-ray photon energy in keV (X axis). The spectra were nominally integrated so that the highest peak would have 10^4 counts in the channel containing the highest number of counts. In all cases the X-axis ranges from 0 keV to 10 keV.

The x-ray lines on the spectra are primarily K_{α} emission lines. The positions of important lines are:

Mg K _a	1.3 keV	
Al K _a	1.5 keV	(Al is usually unresolved due to the close- ness of large Mg and Si peaks)
Si K _a	1.8 keV	
s κ _α	2.3 keV	
Pd L a	2.8 keV	
Са К а	3.7 keV	
Cr K _a	5.4 keV	
Mn K _a	5.9 keV	
Fe K _a	6.4 keV	
Fe K a	7.1 keV	
Ni K a	7.5 keV	

The Pd line on all particles results from a thin palladium coating which was sputtered onto the samples to provide a conductive coating. Three particles U2-5A 21, 29, and 30 were coated with gold-palladium. The gold on these samples produces a line coincident with S and a second line (I_{tt}) at 9.7 keV. A few spectra show small differences in energy values due to electronic drift. An example of line identification is given in Figure 1 for an analysis of a representative sample of the Allende meteorite.

A material containing cosmic abundances produces the following approximate peak height ratios as observed on the EDAX unit under our operating conditions.

Mg/Si ~ 0.5 Fe/Si ~ 0.5

S/Si	~0.3
Ca/Si	~ 0.05
Ni/Si	~ 0.05
Mn/Si	~ 0.01
Cr/Si	~ 0.01

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Figure 1. - An example of peak height identification. The sample is a 0.1 cm polished piece of the Allende carbonaceous chondrite, coated with carbon.





Figure 2. – U2-5A (21) Chondritic aggregate unusually high porosity. Major elements: Si, Fe, S, Mg. Minor elements: Ca, Ni (Au-Pd coating). (all scale bars are 1 µm).





Figure 3. – U2-5B (24) Typical chondritic aggregate; contains a 1 x 2 μ m silicate inclusion. Major elements: Si, Mg, Fe, S. Minor elements: Ca, Ni, Cr.





Figure 4. – U2-5A (30) Chondritic aggregate, high porosity with 3 μm inclusion. Major elements: Si, Mg, S, Fe. Minor elements: Ca, Ni, Au (coating).





Figure 5. – U2-5B (47) Chondritic aggregate, small. Major elements: Si, Mg, Fe, S. Minor elements: Ca, Ni.





Figure 6. – U2-5B (35) Chondritic aggregate. Major elements: Si, Fe, Mg, S. Minor elements: Ni.





Figure 7. – U2-5A (29) Chondritic aggregate, note smooth, rounded areas. Major elements: Si, Nig, Fe, S. Minor elements: Ca, Cr, Ni, Ac (coating).





Figure 8. – U2-5D (13) Chondritic aggregate, low porosity. Major elements: Si, Mg, Fe. Minor elements: S, Ca, Ni.

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Figure 9. – U2-5D (7) Chondritic aggregate, non-porous. Major elements: Si, Fe, Mg, S. Minor elements: Ca, Ni.

Figure 10. - U2-5B (43) Chondritic aggregate, very low porosity. Major elements: Si, Mg, S, Fe. Minor elements: Cr, Ni.

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Figure 11. - U2-2E (28A) Chondritic aggregate that fragmented upon impaction on the collection surface. (a) Low magnification showing most of the particle. The fine grained portions are chondritic aggregate material. Most of the particles with smooth surfaces are either FSN or mafic silicates. Also found are high Ca, Al, Si, Ti minerals and an opaque high Si mineral. Note the rod shaped particles.

(b) Mafic silicate grain on left connected to chondritic aggregate material.

Figure 11. - U2-2E (28A) Chondritic aggregate that fragmented upon impaction on the collection surface.

(c) FSN hexagonal crystal with attached chondritic aggregate material.

Figure 12. - U2-6A (9) Parts of a fragmented particle.

(a) FSN, an unusual stack of platelets.

Figure 12. – U2-6A (9) Parts of a fragmented particle.

(b) Chondritic aggregate (uppermost particle) and FSN (all other particles).

Figure 13. – U2 5B (49) Chondritic ablation sphere with submicron magnetite grains. Major elements: Fe, Si, Mg. Minor elements: Ca.

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Figure 14. – U2-6A (4) Chondritic ablation, spheroidal, showing submicron magnetite grains. Major elements: Fe, Si. Minor elements: Mg, Ca, Cr.

Figure 15. – U2-6A (18) Chondritic ablation. Major elements: Fe, Si, Mg. Minor elements: Ca, Cr, Ni.

Figure 16. – U2-6E (5) FSN, typical sphere with surface texture. Major elements: Fe, S. Minor elements: Ni.

Figure 17. – U2-58 (3) FSN, octahedron with cubic truncation. Note growth steps. Major elements: Fe, S. Minor elements: Ni.

Figure 18. – U2-6D (1) FSN, Major elements: S, Fe. Minor elements: none detecte/

Figure 19. – U2-6B (6) FSN, aggregate very rare. Major elements: S, Fe. Minor elements: Si, Ni.

Figure 20. – U2-6D (8) FSN, unusual surface structure, very little sulfur. Major element: Fe. Minor elements: S, Ni.

Figure 21. - U2-62 (29) Fe-Ni, very unusual. Major elements: Fe, Ni. Minor elements: none detected.

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Figure 22. – U2-5B (20) Mafic silicate with adhering chondritic aggregate material. Silicate: Fo 93 olivine. Major elements: Mg, Si. Minor elements: Fe. Chondritic material: Major elements: Si, Fe, Mg, S. Minor elements: Ca, Ni.

Figure 23. – U2-5B (54) Mafic silicate, Fo 55 olivine. Surface features not detectably different in composition from crystal (perh.ps because they are small). Major elements: Si, Fe, Mg. Minor element: Ca.

Figure 24. – 112-5B (30) Mafic silicate grain, Fo 95 olivine. Surface features not detectably different in composition (perhaps because they pre small). Major elements: Mg, Si. Minor elements: Fe, Cr.

Figure 25. – U2-6C (35) Chondritic aggregate with a 6 µm smooth, transparent, high Si-Ca-Al grain. Chondritic material: Major elements: Si, Mg, S, Fe. Minor elements: Ca, Ni. Si-Ca-Al material: Major elements: Si, Ca, Al. Minor elements: S, Fe (probably from chondritic portion of particle).

Figure 26. – U2-5A (39) Rounded chondritic particle covered with Fe-Ni mounds. Particle is possibly the result of a collision in space. Major elements: Si, Mg, Fe. Minor elements: Ca, Ni, Cr.

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Figure 27. – U2-9A (4) Very strange and unusual particle, covered with Fe-Ni mounds. Major elements: Si, Fe, S. Minor element: Ni.

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