

## A Method for Isolating Very Small Particles From Plastic Explosive Samples

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### KEYWORDS

Composition C4, dust, explosives, forensic investigation, plastic explosives, polarized light microscopy (PLM), provenance, scanning electron microscope-energy dispersive spectroscopy (SEM/EDS), small particles, X-ray diffraction (XRD)

### ABSTRACT

Plastic explosive samples may contain large numbers of fine particles adhering to their surfaces. Analysis of these particles can be useful in forensic investigations involving plastic explosives. Data obtained from particle analysis can be used to develop investigative leads regarding the origin of an unknown explosive device or to compare two or more samples to determine whether they share a common origin. A method for isolating fine particles from Composition C4 plastic explosive samples is described, and examples of particles recovered by this method in casework are provided.

### INTRODUCTION

A number of plastic explosive samples were submitted to Stoney Forensic, Inc. for very fine particle analysis to assist source attribution. The customer was interested in determining the geographic origin of the samples as well as determining whether any of the samples could be associated with each other. The samples were collected from inside sealed devices, and

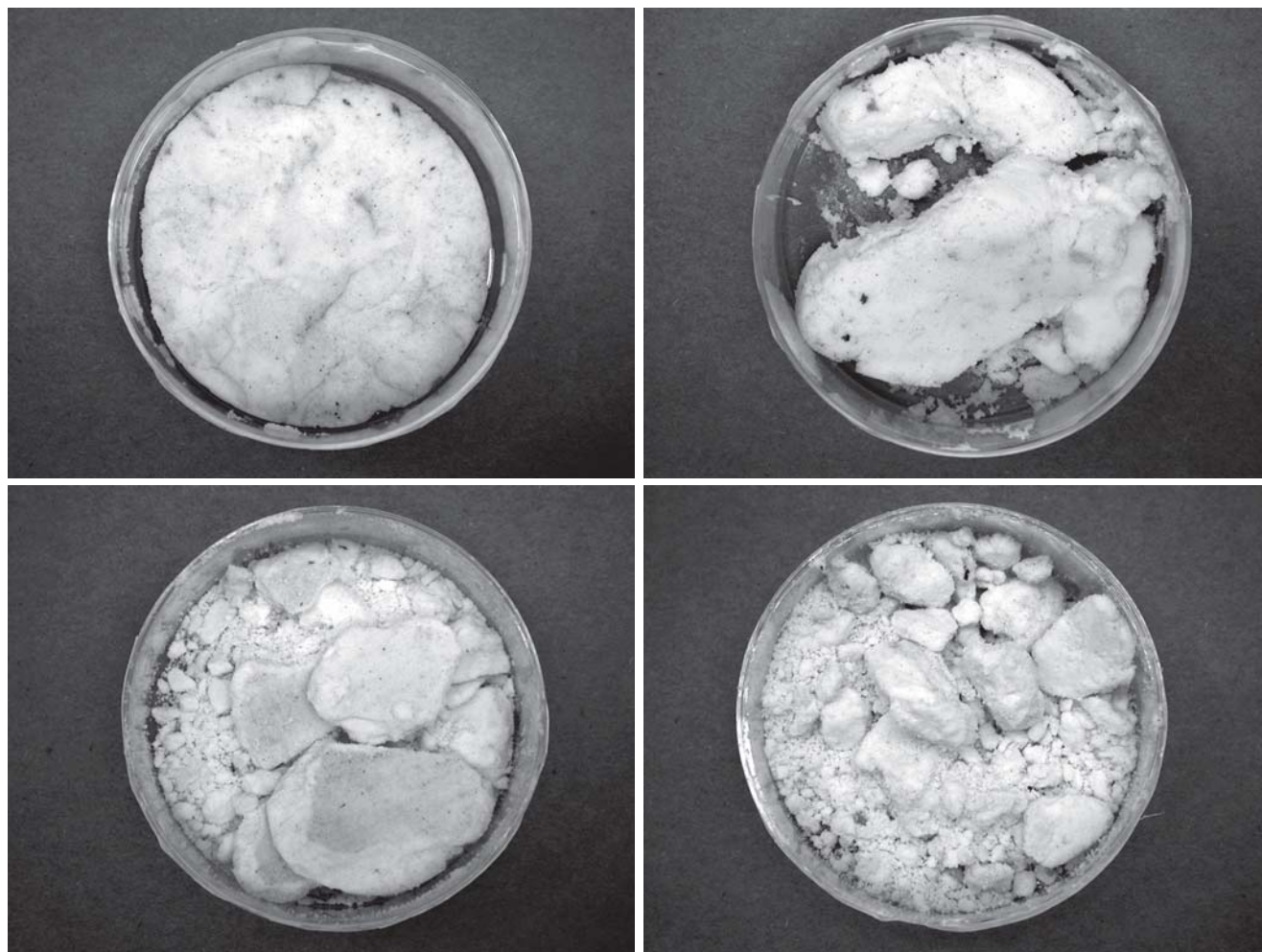
it was hypothesized that the fine dust particles trapped in the plastic explosive had originated from the location where the devices were assembled and that the plastic explosive had been isolated from the external environment since being sealed at that location. In order to analyze the dust particles trapped in the plastic explosive, a method for isolating the particles from the plastic explosive matrix was developed and is reported here.

### METHODS

The first step in recovering very fine particles from a Composition C4 sample involves manual isolation of all large trace evidence particles (easily visible to the naked eye) using non-magnetic forceps. These particles are set aside for subsequent characterization by appropriate trace evidence techniques and do not warrant further discussion here.

The next step in the developed method is recovery of the fine dust trapped in the tacky plastic explosive matrix. The very top layer of each sample, where the bulk of the dust is located, is carefully pulled off of the remaining material using clean forceps and transferred to an appropriately sized tube (such as a 15 mL centrifuge tube). This process is generally straightforward, although Composition C4 plastic explosive can be encountered with a variety of textures and some are more challenging to sample than others. Textures encountered by the author range from the consistency of very hard putty to soft molding clay to dry and crumbly (Figure 1).

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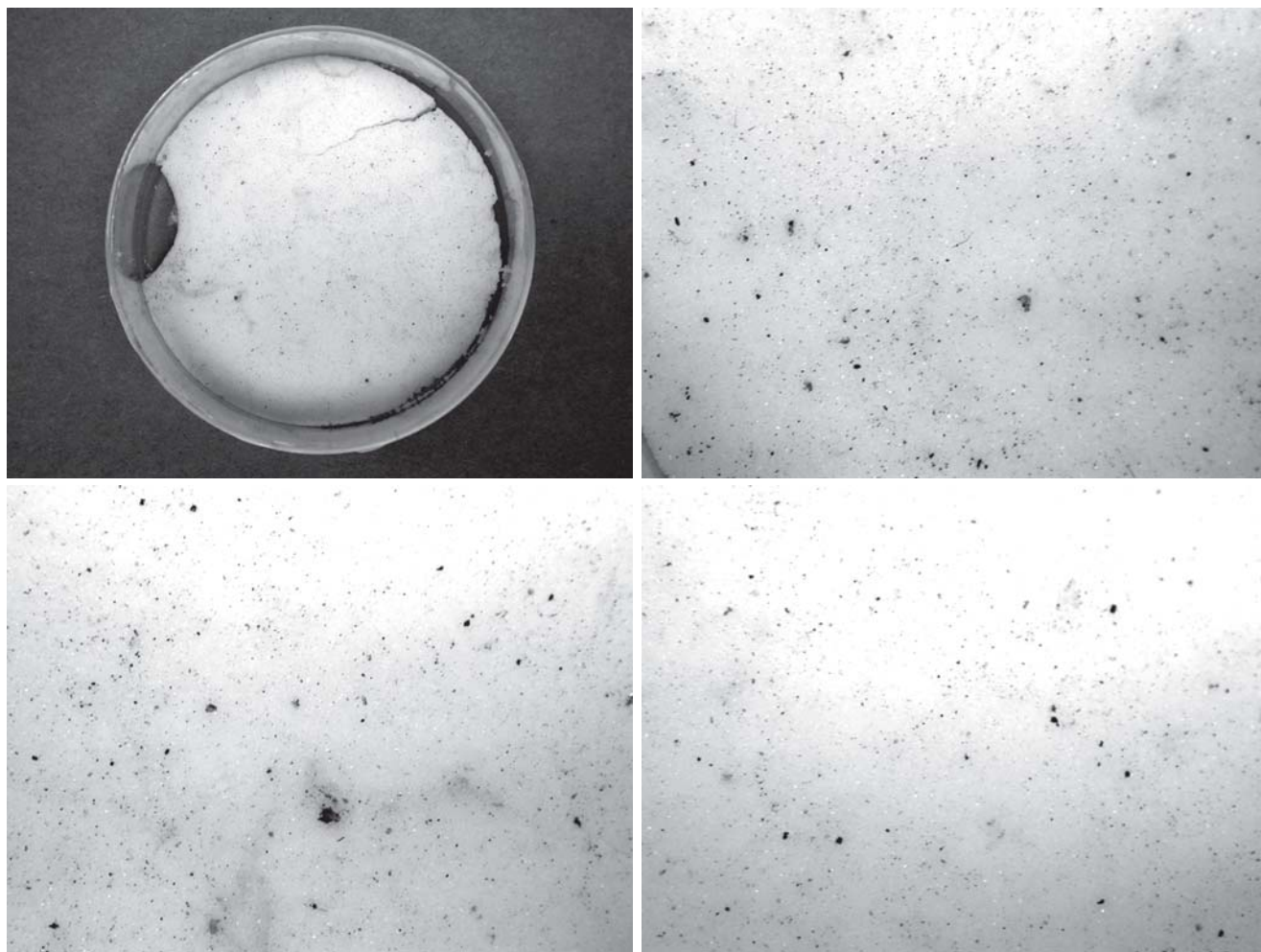
**Figure 1.** Photographs of several plastic explosive samples show the range of textures encountered by the author, from hard putty (upper left) to dry and crumbly (lower right). The diameter of the Petri dish is 9 cm.

The next step is the separation of the dust particles from the plastic explosive matrix for analysis and identification. In order to recover the fine particles in an efficient manner, the plastic explosive matrix is dissolved using appropriate solvents, concentrating the insoluble dust particles for analysis. The components making up Composition C4 plastic explosive include the high explosive RDX (approximately 91%); a plasticizer, generally dioctyl adipate, although dioctyl sebacate was used in the past (approximately 5%); polyisobutylene (approximately 2%); and hydrocarbon oil (approximately 2%) (1, 2). Taking this into account, the solvents selected to dissolve these components are acetone and hexanes.

Reagent grade acetone (>99.5%) is used first to dissolve the RDX. The sample to be analyzed is weighed, and 5 mL of acetone is added per 200 mg of C4. The

sample is agitated with a probe and vortexed. The sample is then sonicated briefly (long sonication times should be avoided as this can damage pollen and mineral grain surfaces). After sonication the sample is centrifuged to coagulate all released particles and the acetone is removed. This process is repeated as many times as needed (typically two or three times) to dissolve all of the acetone-soluble components. After this step, the sample generally resembles a wad of dirty chewing gum.

Hexane (mixed isomers, anhydrous, ≥99%) was used to dissolve the remaining ingredients; although polyisobutylene may not be dissolved by these solvents, its presence did not appear to negatively impact subsequent processing. Approximately 5 mL of hexane is added per 200 mg of C4 (the starting mass of the C4 prior to acetone digestion). The sample is agi-



**Figure 2.** Photographs of Sample 1 show a very clean sample of plastic explosive. The diameter of the Petri dish in the upper left image is 9 cm; the other images are close-up photographs of the same sample.

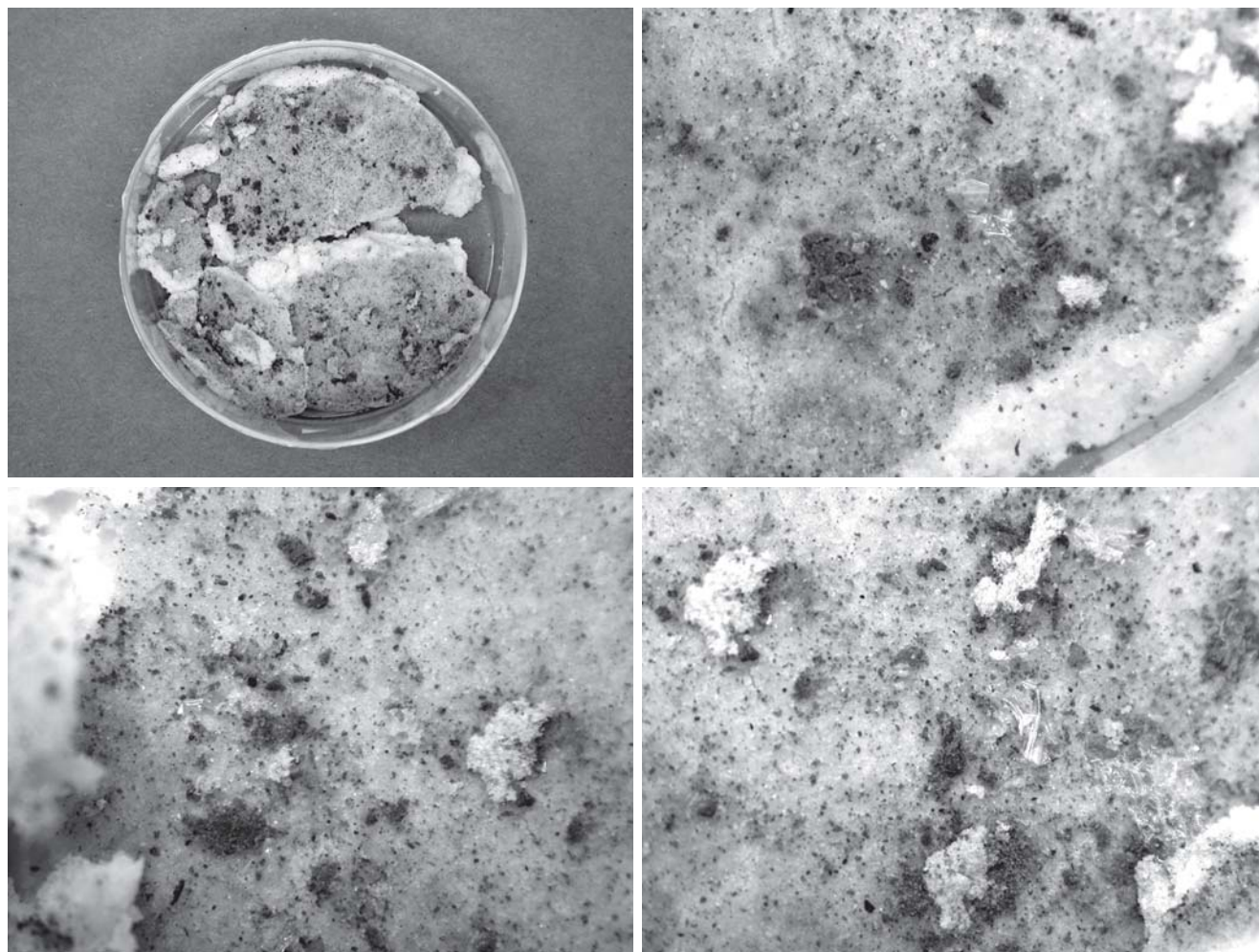
tated with a probe and vortexed. The sample is sonicated briefly and centrifuged to coagulate all released particles, and then the hexane is removed. This process is repeated as many times as needed (typically two or three times). After this step, loose particles are concentrated in the bottom of the tube.

The loose particles can then be prepared for analysis according to the standard operating procedures in place at each individual laboratory. For this project, the particles were separated using a soil processing protocol. The first step in this method involves separation into different-size fractions by settling in water. However, transferring the particles directly to water after the hexane washes results in aggregation, clumping, and flocculation of the sample. In order to avoid this, the sample is washed sequentially with acetone, ethanol and water, and centrifuging after each step.

The sample is then separated into a variety of fractions for subsequent analysis using the method advocated by Palenik (3) with a few minor differences. The sample is first separated into sand, silt and clay by settling in water. Sieving is performed on the sand fraction (when appropriate), followed by density separation of the fine sand (sample size permitting). For this project the minerals were analyzed by PLM and SEM/EDS as appropriate, pollen grains by acetolysis of the silt fraction and light microscopy, and clay by XRD.

## RESULTS

The method described above was applied to some 30 samples submitted for analysis. Microscopical analysis of the recovered particles produced extremely useful results for every sample, including hundreds of



**Figure 3.** Photographs of Sample 2 show a relatively dirty sample of plastic explosive. The diameter of the Petri dish in the upper left image is 9 cm; the other images are close-up photographs of the same sample.

particles of various types. The resulting source attribution inferences were extremely fruitful.

Two examples of Composition C4 samples processed using the method described above are provided below to illustrate the range of materials recovered. Sample 1 was the “cleanest” sample of those analyzed, both in macroscopic appearance (Figure 2, page 119) and in terms of the dearth of recovered particulate material. The total mass of the particulate material recovered from Sample 1 was only 2 mg, with a starting mass of approximately 0.5 g. Despite this very small sample size, microscopical analysis revealed sufficient sand-sized light mineral grains for statistically significant point count data (>300 grains) (4). However, there were many fewer sand-sized heavy minerals (only 171 grains) and only four pollen grains. There were no crystalline phases detectable by XRD in the

clay-sized fraction.

Sample 2 was one of the “dirtiest” of the samples analyzed, both in terms of its macroscopic appearance (Figure 3) and the amount of recovered particulate material. Sample 2 contained roughly 45 mg of recovered debris, with a starting mass of approximately 0.5 g. Microscopical analysis of the recovered material revealed sufficient sand-sized light and heavy mineral grains for statistically significant results (>300 grains) and more than 100 pollen grains. The clay fraction contained sufficient material for the detection of eight crystalline phases.

Overall, a large variety of particles were recovered from the samples. This included over 42 different minerals (Plate 1, pages 122-123), over 26 different taxa of foraminifera (Plate 2, page 124), over 30 different taxa of coccoliths, at least one dinoflagellate cyst taxon,

several types of plant opal phytoliths (Plate 3, page 124), over 33 different pollen taxa (Plate 4, page 124), botanical macerals (Plate 5, page 125), insect parts (Plate 6, page 125), starch grains (Plate 7, page 125), fungal spores, animal hair, and over 11 different types of anthropogenic materials (Plate 8, pages 126-127). (See plate captions on page 128.)

Useful XRD spectra were also obtained on the clay-sized fractions of numerous samples. The character of the particles in the recovered dust made it possible to significantly constrain the possible geographic origin of the samples, as well as provide strong evidence supporting conclusions about whether any of the samples were assembled in similar environments (many of which were).

## DISCUSSION

A method has been developed for recovery of fine particles from a plastic explosive matrix. This method was applied to a large number of samples and appears to efficiently recover a wide variety of particle types from plastic explosive samples. The particles recovered offer tremendous potential for provenance investigation and comparative analyses. They were extremely useful in the specific cases described above, and have great potential to contribute to future investigations.

The primary limitation of the method is the loss of those species soluble in the solvents used. As applied to these 31 samples, materials soluble in acetone, hexanes, ethanol and water were all lost. It may be possible to save the solvents and reduce their volumes by drying down the liquids in a particle-free environment in order to analyze soluble species. However, due to the large quantities of acetone-soluble and hexane-soluble materials derived from the plastic explosive itself, it is unlikely that materials present in trace amounts in the dust adhering to the samples could be detected. Alternatively, settling velocity separations could be conducted in hexanes, preserving the ethanol-soluble and water-soluble species.

Another limitation to this method is the amount of time required to isolate the particulate samples. The procedure required two days to process a sample and prepare it for analysis. However, the bulk of the time involved centrifugation and settling periods; there was relatively little hands-on time, and the analyst was able to complete other work during the two days of processing. There is clearly room to improve on the method as described above. However, it appears to be a good starting point for future work of this type.

## ACKNOWLEDGMENTS

The author would like to acknowledge David Exline for performing SEM/EDS and XRD analyses on the samples, and he is responsible for the SEM images presented in the color plates. The author would also like to thank Dr. Vaughn Bryant for conducting the pollen analysis and for providing the photomicrographs of pollen grains seen in the color plates. Finally, the staff of the McCrone Research Institute in Chicago contributed to analysis of the large particles recovered from these samples; their help is greatly appreciated.

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*See Plates 1-8 on pages 122-127, and plate captions on page 128.*

Plate 1. Minerals

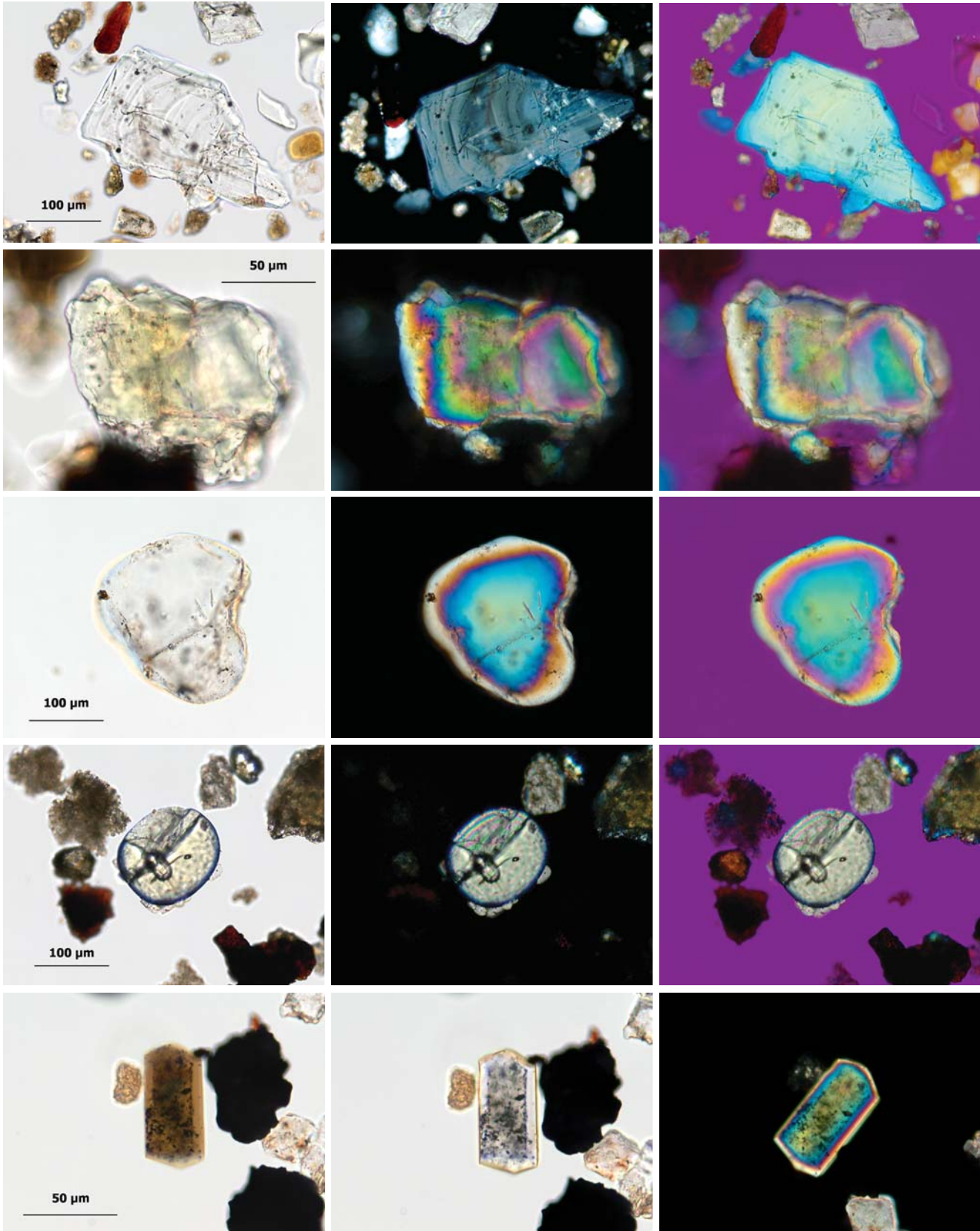


Plate 1. Minerals (continued)

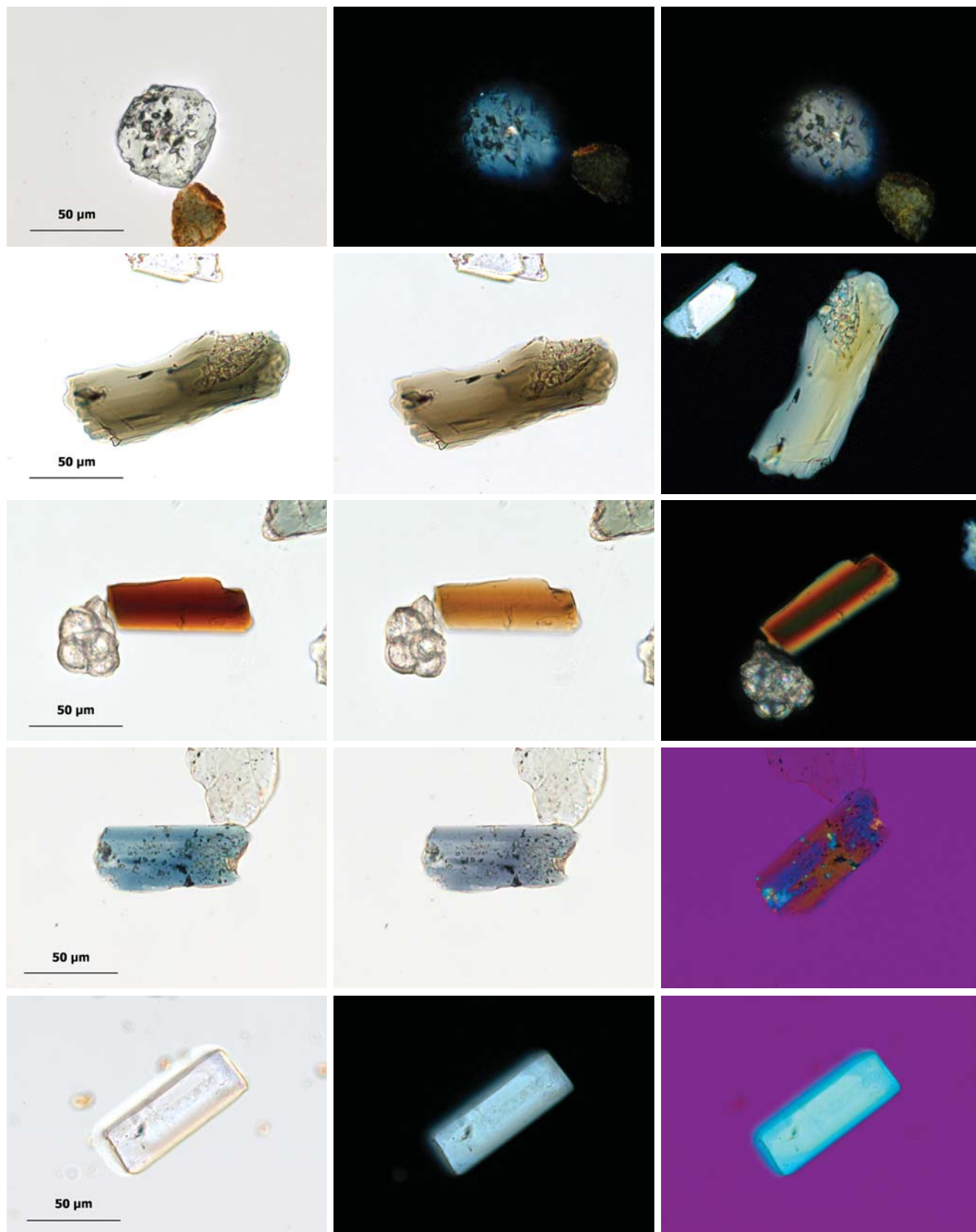


Plate 2. Foraminifera

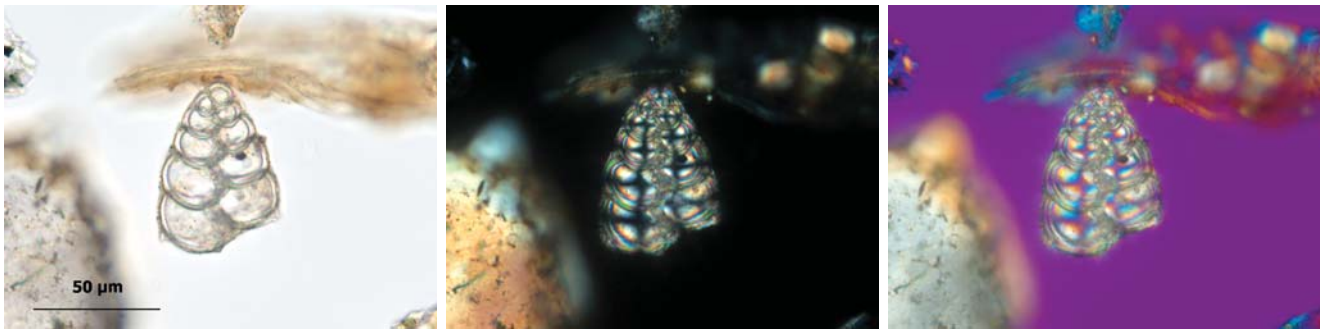


Plate 3. Plant Opal Phytoliths

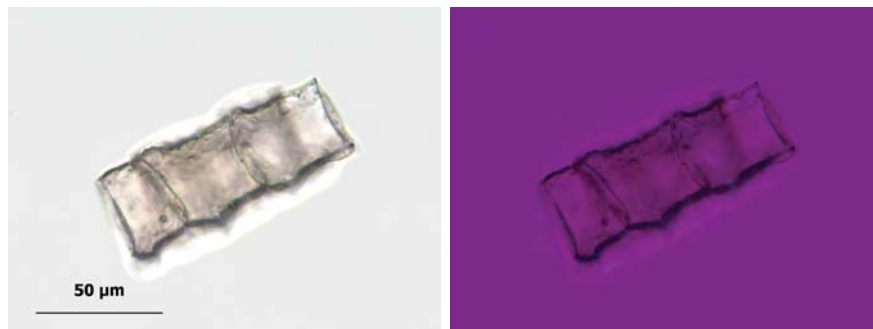


Plate 4. Pollen

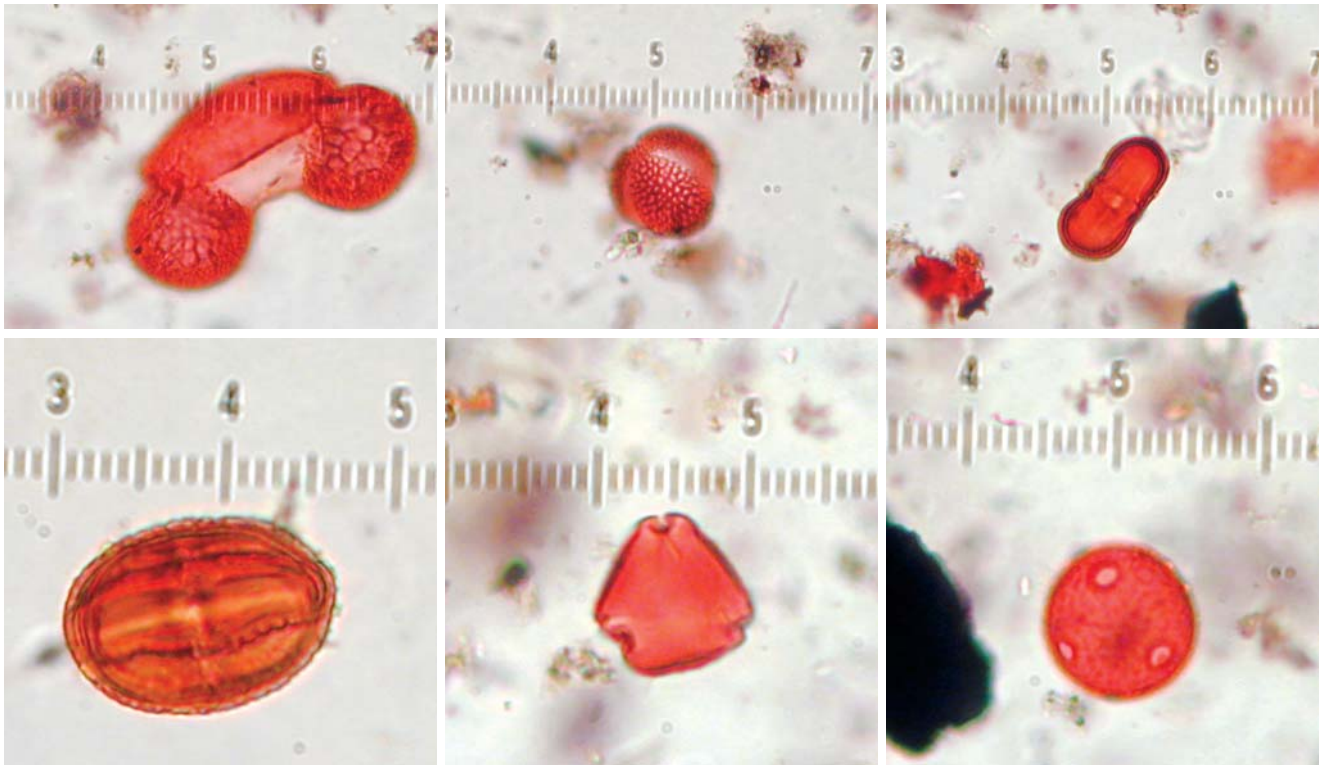


Plate 5. Botanical Macerals

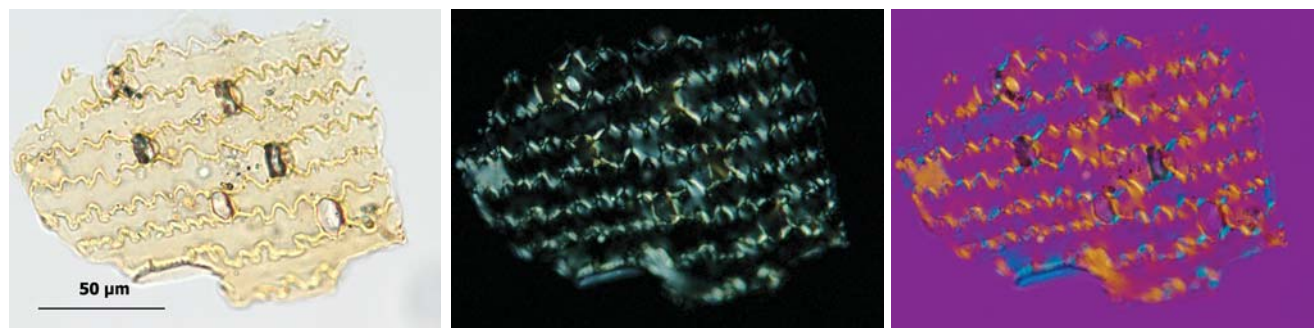
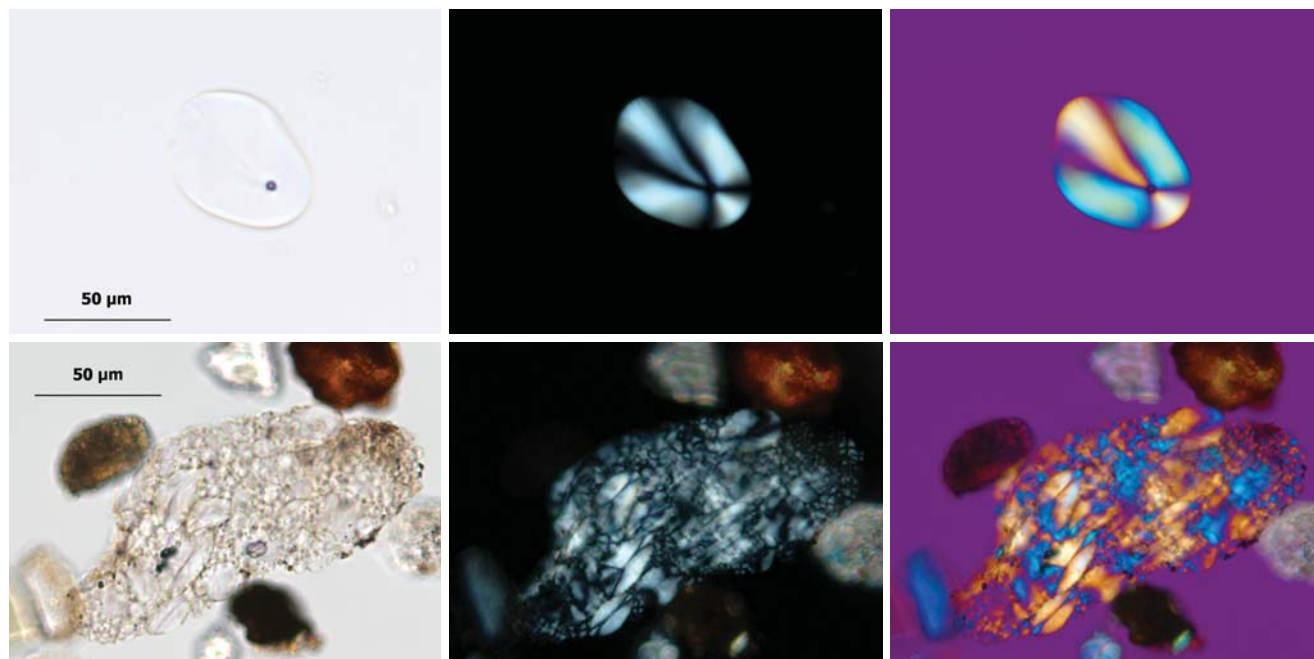


Plate 6. Insect Parts



Plate 7. Starch Grains



## Plate 8. Anthropogenic Materials

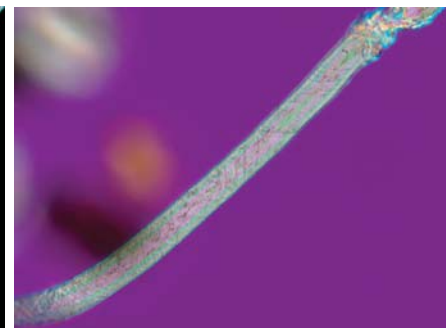
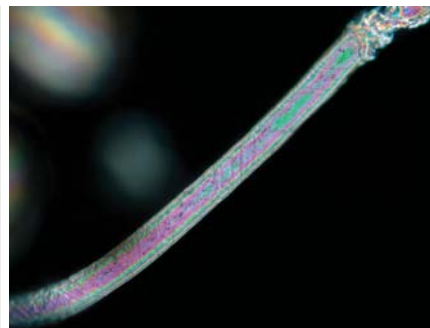
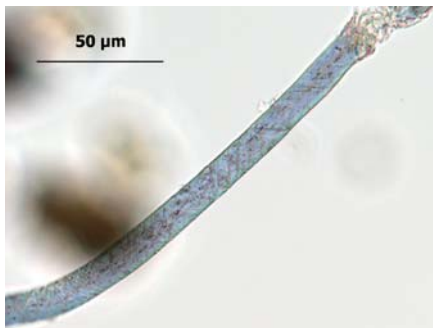
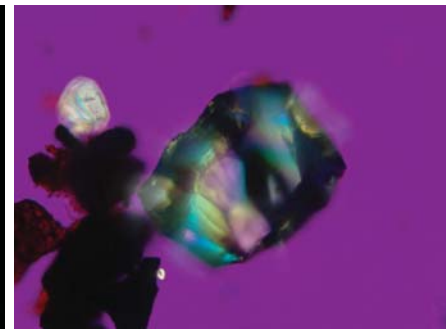
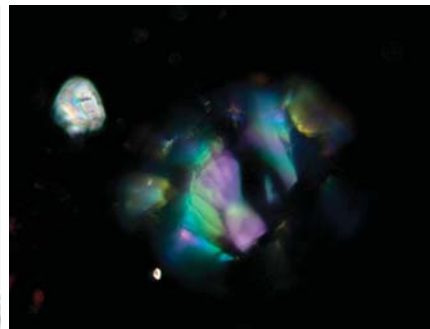
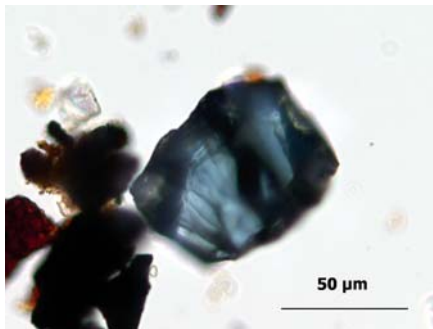
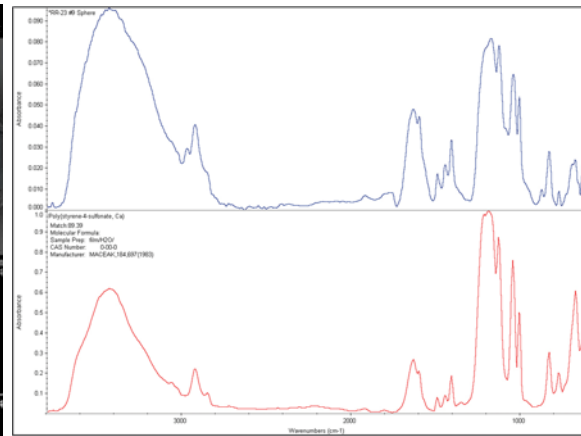
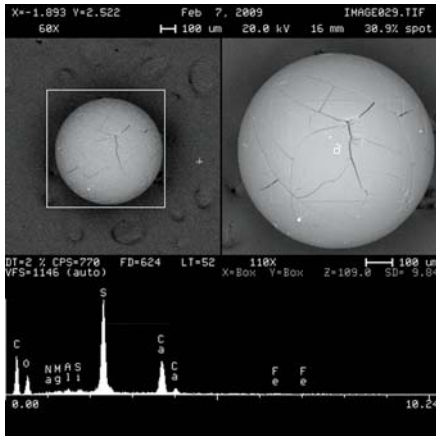
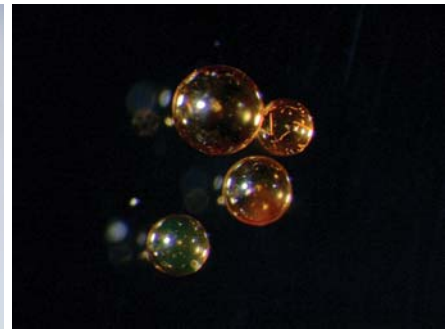
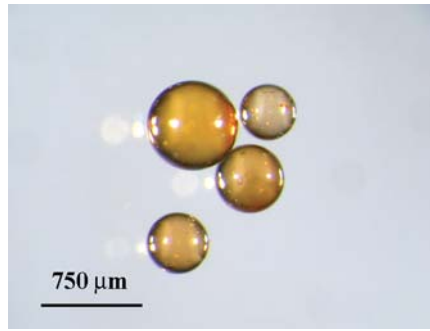
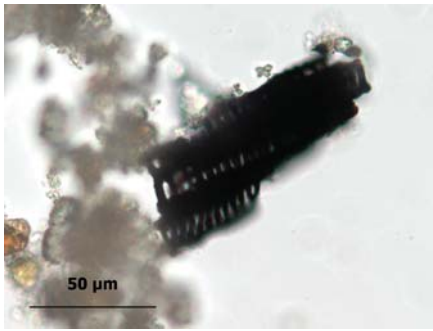
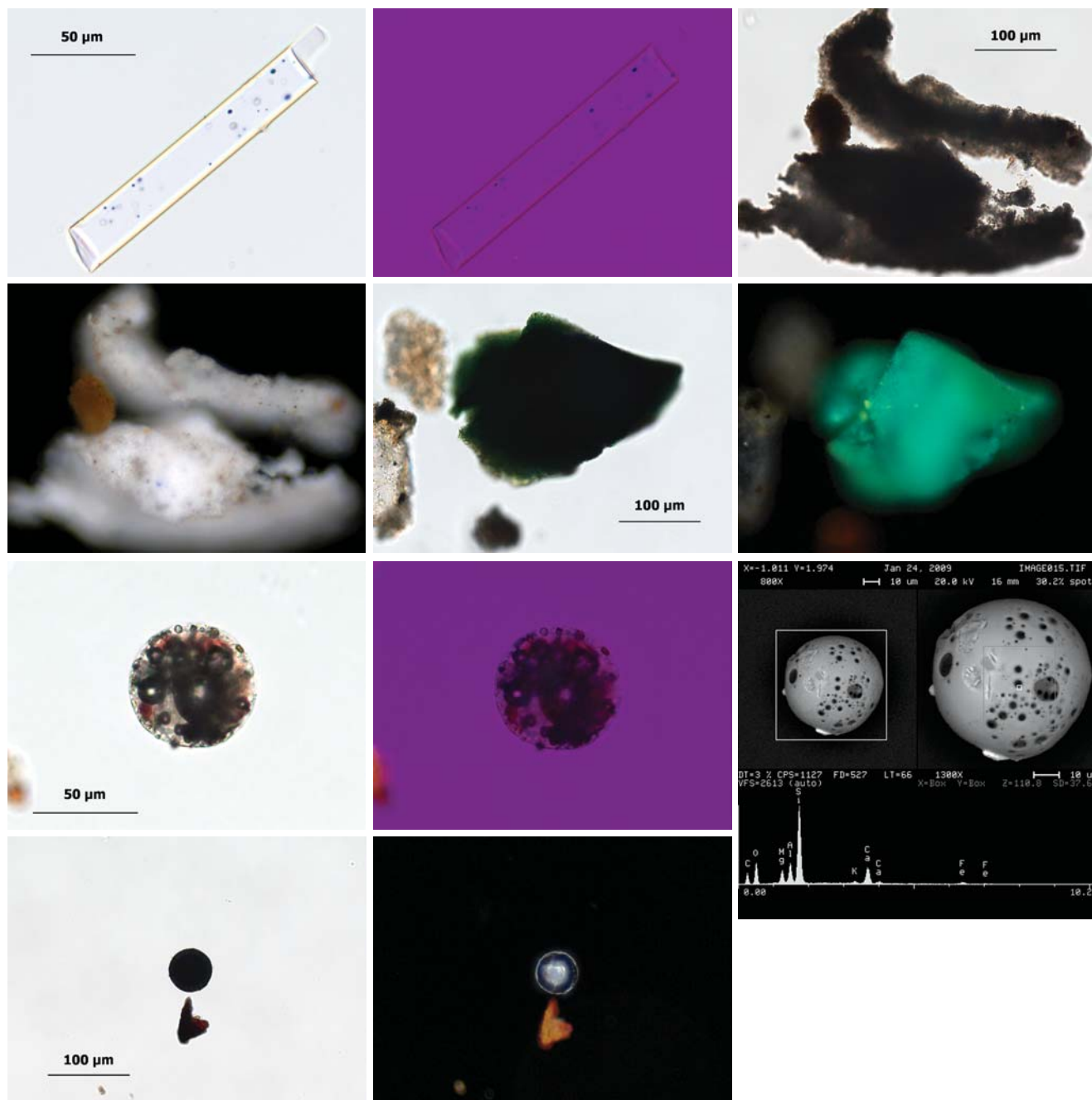


Plate 8. Anthropogenic Materials (continued)



## Plate Captions

### Plate 1. Minerals (pp 122-123)

Page 122, top to bottom: Mineral grains shown are plagioclase, epidote, quartz, monazite and tourmaline (polarizer oriented E-W on the left and N-S in the middle). Page 123, top to bottom: Mineral grains shown are titanite, hornblende (polarizer oriented E-W on the left and N-S in the middle), basaltic hornblende (polarizer oriented E-W on the left and N-S in the middle), glaucophane (polarizer oriented E-W on the left and N-S in the middle) and celestine. Mounting media are 1.540 for plagioclase and quartz, and 1.660 for all others.

### Plate 2. Foraminifera (p 124)

A *Heterohelix planata* specimen is shown, from left to right, in plane-polarized light, in crossed polars and in crossed polars with a 530 nm compensator (right). Mounting medium is 1.540.

### Plate 3. Plant Opal Phytoliths (p 124)

An unidentified plant opal phytolith is shown in plane polarized light (left) and in crossed polars with a 530 nm compensator (right). Mounting medium is 1.540.

### Plate 4. Pollen (p 124)

Top row: The pollen taxa are, from left to right, *Pinus* (diploxylon type pine), Brassicaceae (mustard family) and Apiaceae (umbel family). Bottom row: The pollen taxa are, from left to right, *Centaurea* (knapweed), Rhamnaceae (buckthorn family) and *Plantago* (plantain). Mounting medium is glycerine.

### Plate 5. Botanical Macerals (p 125)

A fragment of grass epidermis is shown, from left to right, in plane polarized light, in crossed polars and in crossed polars with a 530 nm compensator (right). Mounting medium is 1.540.

### Plate 6. Insect Parts (p 125)

An unidentified insect part is shown in plane polarized light. Mounting medium is 1.540.

### Plate 7. Starch Grains (p 125)

Top row: The images shown, from left to right, are likely a potato starch grain in plane polarized light, in crossed polars and in crossed polars with a 530 nm compensator (right). Bottom row: Images show an aggregate of likely wheat starch grains under the same conditions. Mounting medium is 1.540.

### Plate 8. Anthropogenic Materials (pp 126-127)

Page 126, top row: The images show, from left to right, a charred wood fragment, followed by two spheres of poly(styrene-4-sulfonate, Ca) by stereomicroscopy. Second row: An SEM image of one of the spheres with its EDS spectrum (left), and its FTIR spectrum (right image, top), stacked with a reference spectrum of poly(styrene-4-sulfonate, Ca) (right image, bottom). Third row: Carborundum. Fourth row: A polyester fiber. Page 127, rows 1-3: The images are of a glass fiber, a white paint fragment, a green paint fragment, a fly ash sphere shown by light microscopy, followed by an SEM image and EDS spectrum of the fly ash sphere. Fourth row: The images are of a metallic sphere (likely from a welding process) shown, from left to right, in both transmitted and reflected light.