particles by means of a continuous-flow centrifuge. All these samples contained dinoflagellates, pollen grains, arthropods, and copepods, but none contained mineral matter. One sample of 200 liters was obtained from a depth of 4030 meters, 160 meters above the sea bottom, at 32°15′N, 74°45′W. This relatively small amount of sea water contained, apart from some organisms, about 0.50 g. of suspended clay, a surprisingly large quantity.

A study of the amount and type of suspended mineral matter contained in sea water at various depths should provide information concerning (i) the transportation of sediments in the ocean basins and (ii) post depositional diagenetic changes (*1*).

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Note

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Barium Xenate

Abstract. *Barium xenate was prepared by the addition of barium hydroxide to xenic acid. The resulting white amorphous precipitate was shown by elementary analysis to be* Ba_3XeO_6.

At the recent Argonne Conference on Noble Gas Compounds one of us (A.D.K.) reported (*1*) on the preparation of barium xenate. At the same meeting, sodium and heavier metal perxenates were described by J. G. Malm *et al.* (*2*). In this paper we give more detailed information on the xenate.

First of all, the properties of the noble gas compounds generally follow the expected properties of their neighbors in moving from the left to right in the same period of the periodic system. Thus, the xenon compounds are similar to the iodine compounds; the compounds of tellurium, the element just preceding iodine, show even greater similarity in view of the same even valences. Therefore, the xenon and tellurium compounds have identical formulas and probably identical geometry for the corresponding compounds. Recognition should be given, of course, to the fact that Te is more electropositive than Xe. As an example, the close similarity of XeF_6 and XeF_4 to TeF_6 and TeF_4 both in their physical and chemical properties may be described (*3*, *3a*).

Table 1. Distillation of aqueous xenic acid solution at 0°C.

Stage of test	Volume (cm³)	Concentration (mmole/cm³)	Total (mmole)	
Start	3.00	0.018_9	0.057*	
Distillate	1.50	.0097	.015	0.058
Residue	1.50	$.028_5$	.043	

* Corrected for decomposition of xenic acid at 0°C during distillation.

Xenic acid, $Xe(OH)_6$, first described by Dudley *et al.* (*4*) and Williamson and Koch (*5*), is like $Te(OH)_6$ (*3*), a very weak acid. It is a strong oxidizer and can be titrated (*4*) quantitatively by titrating I_2 with standard thiosulfate in line with the equation :

$$Xe(OH)_6 + 6\ KI + 6\ HCl \rightarrow Xe + 6\ H_2O + 3\ I_2 + 6\ KCl.$$

We have prepared xenic acid by slowly hydrolyzing XeF_4 (*6*) or xenon oxyfluorides at 0°C with water. Our yields varied between 30 to 40 mole percent, based on the XeF_4 content. A simple way of obtaining pure xenic acid solution free of HF was to agitate the solution with a slight excess (approximately 5 percent above the amount of HF determined by titration) of powdered $Ca(OH)_2$ or $CaCO_3$ at 0°C. Calcium fluoride is rapidly formed and is then filtered or centrifuged off, after the reaction is complete.

The $Xe(OH)_6$ was titrated in line with the above equation, while the free HF was titrated with standard alkali. For storage the xenic acid solution was kept in a freezer at −20° to −30°C without decomposition for weeks.

We expected that salts of xenic acid would be stable and that barium xenate would be a slightly soluble salt similar to barium tellurate. This proved to be the case. Barium xenate was prepared by adding, at 0°C, 3 moles of barium hydroxide solution to 1 mole of pure xenic acid. The reaction was complete and yields up to 100 percent were obtained provided the addition was rapid enough to avoid decomposition of the xenic acid. The composition of the very slightly soluble, white amorphous precipitate was very close to Ba_3XeO_6, regardless of whether the base was added in one or several portions. The ratio of Ba to $Xe(OH)_6$ in the precipitate was 2.97:1 when the base was added all at once; and it was 2.80, 3.17, and 2.95:1 when the base was added in three steps. The solubility of the barium xenate in water is 0.25 g per liter at 20°C.

Sodium and potassium salts of xenic acids have also been prepared in our laboratories, but they have not, as yet, been fully characterized. Aqueous solutions (7 to 8m*M*) of Na or K salts did not noticeably lose their oxidizing power at 0°C after 70 hours at *p*H 7 and *p*H 11.5. These xenates, as well as our barium xenate, are thermally stable at room temperature for at least a month. Barium xenate begins to decompose slowly at 125°C and the decomposition is complete at 250°C. To be sure of this, it was heated to 485°C and there was no further evolution of gas; the BaO residue was tested with acidic potassium iodide solution and was free from undecomposed xenate and also BaO_2, a possible product of decomposition if perxenate was present. The decomposition follows the equation:

$$Ba_3XeO_6 \rightarrow 3\ BaO + Xe\uparrow + 1.5\ O_2\uparrow$$

The salt and the BaO residue were analyzed as follows: BaO by acid titration and as $BaSO_4$; xenic acid by the I_2 liberated from KI; and Xe and O_2 by gas analysis. The results in percent by weight of two samples were as follows: BaO, 72.9, 71.75 (calculated for Ba_3XeO_6, 71.96); Xe, 19.8, 20.60 (calculated, 20.53), and O, 7.0, 7.55 (calculated, 7.51). The molar ratio of O_2/Xe in the thermal decomposition is 1.43, 1.52 ± 0.04 (calculated, 1.50). The second sample was also tested for $BaCO_3$ and found to contain none. Thus, our salt is a xenate and not a perxenate, which would require a 2.00 to 1 ratio.

The thermal stability of barium xenate and the greater stability of alkali xenates offer the interesting possibility that xenon may exist in nature, not only in the atmosphere as the free element but in hydrothermal minerals or in the waters of the ocean like the iodates (*7*).

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Surface Features of Metallic Spherules

Abstract: *Metallic spherules of variable character have been recovered from Antarctic snow. Three types were recognized from their surface features: type I, smooth, polished spherules, apparently produced by surface melting of the particles upon entry into the earth's atmosphere; type II, spherules with a corrugated surface caused by differential hardness of internal, intersecting lamellae, but modified by superimposed pits; and type III, spherules with random, circular depressions or pits apparently resulting from impact with submicroscopic particles. Spherules of types II and III were too small to have suffered abrasion by impacts in the earth's atmosphere, and it is postulated that their surfaces may have been produced by erosion in space. Preservation of these surface details would have been possible if entry into the atmosphere took place at low velocities and low-angle trajectories.*

Murray (*1*) was the first to recognize surface details on metallic spherules of extraterrestrial origin. He noted that some spherules possessed a large surface depression or "cupule," while others had a "crystalline" surface or "granular" appearance. Later, Buddhue (*2*) reported the discovery of similar metallic spherules with "a number of shallow, dimplelike depressions," but noted that otherwise their surfaces were smooth and shiny. To investigate the surface features of such particles further, we examined, under optical and electron microscopes, samples recovered from Antarctic snow.

The metallic spherules studied were selected as representative of those which occur in the snow of the southern Antarctic Peninsula region (Ellsworth Land) (*3*). Electron probe analyses of apparently identical material indicated the following elemental composition: Fe, 65 to 70 percent; Mn, 1 to 2 percent; Ti, trace; Si, trace. These results correspond to the composition of the mineral magnetite, which was found to be the primary mineralogical component by mineragraphic and x-ray tests. The spherules were microscopic in dimensions, the average size being only about 40 μ in diameter. Their surface features are at least two orders of magnitude smaller. Three types of spherules were recognized from surface features: type I, smooth, polished spherules with no apparent surface detail; type II, spherules of lower luster and a corrugated surface, comprised of two regular sets of lamellae; these lamellae intersect at approximately right angles and show pronounced relief, creating surface "ridges" and "furrows"; type III, spherules of variable luster, with randomly oriented, approximately circular depressions or "pits." In the samples examined, spherules which displayed surface features (types II and III) were on the order of three times more abundant than those which did not (type I).

Figure 1 shows an optical micrograph of a type-II spherule under high magnification. Unfortunately, it was only possible to bring a small portion of the spherule into focus. Nonetheless, the figure shows the regular pattern of intersecting lamellae characteristic of this spherule type. The relief between adjacent lamellae is clearly demonstrated by the shadow patterns.

Details of the surface microtopography of type-II and -III spherules were studied under the electron microscope by the carbon replication technique (*4*). The spherules were carefully transferred to a clean, thin, glass cover slip by a fine, sable-hair brush wetted with ethanol. They were then preshadowed *in vacuo* with platinum in two directions mutually perpendicular to one another at an angle of incidence of about 45°, and then carbon was evaporated over the specimen. The glass slide and spherules were dissolved from the replica by floating them over several changes of HF and HCl. The resulting carbon replica film was washed in distilled water and lifted onto a 200-mesh grid; electron micrographs were made of representative fields, with a Siemens Elmiskop I instrument.

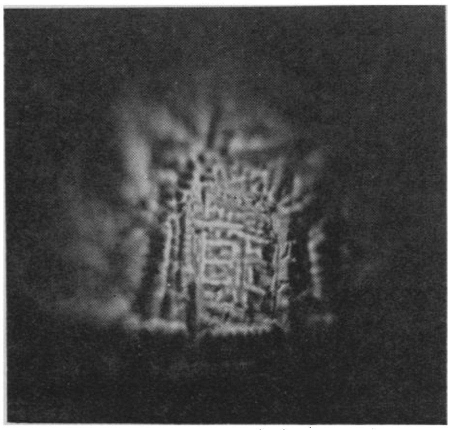

Fig. 1. Optical micrograph of type II metallic spherule from Station 496, Antarctic Peninsula Traverse. [about × 3000]

Fig. 2. Electron micrograph of type-II metallic spherule from Station 464, Antarctic Peninsula Traverse.

Figure 2 shows part of the surface structure of a type-II spherule at a magnification equivalent to visual examination of a portion of a sphere several meters in diameter. As in Fig. 1, two sets of lamellae which intersect approximately at right angles can be observed. Each lamella is about 0.5 μ in width. In addition, Fig. 2 reveals the presence of shallow, circular depressions or pits superimposed on the lamellar pattern. Polished surfaces of individual spherules revealed that mineralogical components of the spherules occurred as lamellae which oriented in at least two directions. It would appear that the regular array of surface features on type-II spherules is a manifestation of their internal lamellae; differential hardness of the lamellae comprising the spherules probably produced the surface relief.

Figure 3 shows part of a type-III spherule. Roughly circular pits with steep, ridge-margined walls and level floors are randomly scattered over its surface. Some pits appear to overlap,