X-ray Diffraction Analysis of the Corrosion Products

S. M. ALEXANDER

Department of Art, University of Texas, Austin

1. Surface appearance

Before the cleaning undertaken by the Metropolitan Museum, the surface of the figurine was covered with a fairly uniform layer of corrosion. A small area of the surface, on the back legs and the thighs, had received a preliminary cleaning before the arrival of the object at the Museum, to reveal metallic silver beneath the mineralization.

2. The corrosion products

- a. The outermost layer, covering the whole animal, was off-white in color, powdery in texture, and easily removable by light scraping. Several parts of this layer showed the pattern of woven material, whose individual fibers had been completely mineralized while retaining their original position on the object. This type of mineralization was particularly thick around the head, a fact that may be due to nothing more than a first rough brushing at the time of finding. Apparently each of the forelimbs, the head with the horns, and the vessel between the forelegs, had been wrapped separately with the fabric. The interior of the vessel also contained a moderately thick deposit of this substance. b. On the horns and the face of the animal, and on the upper section of the vessel, this whitish mineralization was in places of light green color.
- c. Beneath the outermost layer was a tough uniform purplish covering of corrosion, deposited directly on the surface of the metal, and adhering closely to it. Its texture was lumpy, even to the unaided eye, with shiny gray nodules irregularly dispersed over the surface.

- d. Several areas of layer c were covered with what appeared to be redeposited silver, which gave a metallic luster by reflected light.
- e. Small clusters of dark gray black crystals had formed on those parts of the animal that had received the preliminary cleaning.

3. The analyses

The x-ray diffraction analyses were carried out on a Norelco X-ray diffractometer, with standard 114.2 mm. diameter Debye-Scherrer powder diffraction camera. Radiation was the K alpha wavelengths of copper, with nickel filter.

The samples were mounted individually on glass rods that had previously been covered lightly with petroleum jelly, and exposed for 10 hours at 30 kv., 15 ma.

Several samples of the petroleum jelly were run separately under conditions identical with those used for running the samples from the figurine; the d-values from the jelly are duly noted.

The results of the analyses were as follows:

- a. Mineralized fiber from the right thigh: pure calcite $CaCO_3$.
- b. Green corrosion from right side of face: readings only for calcite, as above. It seems likely that the green color was due to contact with an object containing copper whose corrosion had mingled with that of the figurine, but in insufficient amount to give a definite reading on the x-ray film.
- c. The tough purplish layer: pure silver chloride AgCl.
- d. "Redeposited silver" from right arm: silver. Because the x-ray diffraction patterns for silver and gold

are similar, the sample that had been first used for the x-ray diffraction analysis was then used for spectrographic analysis. This confirmed the material as silver; no gold at all was present.

e. The crystals described under point e above were too small and of insufficient quantity for an adequate sample to be run.

X-ray diffraction readings:

a. mineralized fiber from right thigh

from right thigh			
	dues gstrom nits	intensity of line	ASTM standard
4.	13	medium	(petroleum jelly)
3.	75	medium/weak	3.84 (60) calcite
_		weak	(petroleum jelly)
3.0		very strong	3.02 (100) calcite
2		weak/medium	2.49 (60) calcite
		medium	2.28 (70) calcite
	•	medium	1.92 (90) calcite
	-	medium	1.87 (80) calcite
	-	weak	1.60 (50) calcite
Ι.,		weak	1.52 (60) calcite
c. tough purplish laye	r		
4.	13	weak	(petroleum jelly)
3.	-	very weak	3.86 (12) calcite (?)
3.		very strong	3.20 (49) silver chloride
-		weak	3.04 (100) calcite (?)
2.,		very strong	2.77 (100) silverchloride
		very strong	1.96 (50) silver chloride
		medium	1.67 (15) silver chloride
I.	6o :	medium	1.60 (15) silver chloride
I.	39	weak	1.39 (6) silver chloride
		very weak	1.28 (3) silver chloride
Ι.:		medium	1.24 (11) silver chloride
	•	medium/strong	1.13 (7) silver chloride
d. "redeposited silver" from right arm			
4.	11	weak	(petroleum jelly)
-		very weak	(petroleum jelly)

very strong

very strong

medium/strong

medium/strong

medium/weak

2.35 2.05

1.45

1.23 1.18 2.37 (100) silver

2.05 (80) silver

1.44 (80) silver

1.23 (90) silver

1.18 (50) silver