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EXAMINATION OF PROTECTIVE COATINGS UTILIZED ON ARMY DS2 CONTAINERS

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MATERIALS TESTING AND EVALUATION BRANCH

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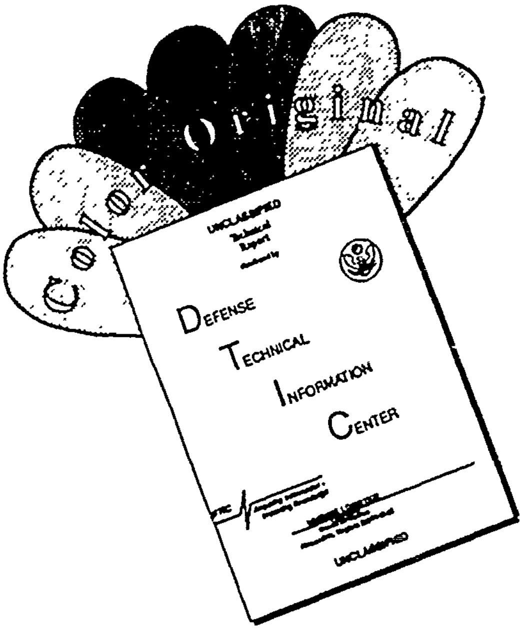

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ABSTRACT This report

The Chemical Research, Development & Engineering Center (CRDEC) requested the U.S. Army Materials Technology Laboratory (MTL) to characterize and evaluate the integrity of protective coatings used on DS2 containers and isolate the origin of the corrosion observed on the containers. Three different size containers were examined. It was determined by optical microscopy and visual examination that the corrosion was initiated from exterior surfaces, particularly in recesses, weld seams, and other areas which trapped moisture. Optical and electron mircroscopy was performed on cross-sectional specimens of the coating. Inconsistent phosphate coating coverage was revealed on the five-gallon container and the urethane topcoat thickness was below specified requirements. The epoxy primer was thicker than specified. The wash primer, epoxy primer, and topcoat thicknesses of the 14-liter container did not meet the specification. The exterior terne coating on the 1-1/3-quart container exhibited corrosion and blistering in many areas.

Several recommendations were made: design flaws conducive to corrosion should be eliminated, alternative materials (e.g. plastics, stainless steels, and aluminum) should be considered, and more stringent quality control of coating application, as well as the handling of coated containers, should be enacted. Finally, if the present coating system and material for the container are not changed, then periodic inspections should be performed to detect corrosion in the early stages.

CONTENTS

BACKGROUND

DS2 is a homogeneous solution of diethylenetriamine (69% to 71%), sodium hydroxide (1.9% to 2.1%), and ethylene glycol monomethylether (methyl cellosolve) remainder and is a standard chemical decontamination agent for the U.S. Army. Since this chemical agent poses an environmental and health hazard, great concern was expressed when it was revealed that many DS2 containers were found to be leaking while in storage at various sites. In addition, when DS2 is exposed to air, polymerization can occur which renders the agent ineffective.

DS2 is stored in steel containers which are manufactured in three different sizes: five-gallon, 14-liter, and 1-1/3-quart. The five-gallon container is utilized for bulk decontamination operations while the 14-liter container is used for the M13 Decontamination Apparatus. The 1-1/3-quart refills the MII Decontamination Apparatus. The five-gallon container had a wall thickness of 0.0245 inches, the 14-liter container was 0.0465 inches thick, and the 1-1/3 container was 0.0160 inches thick. The U.S. Army Materials Technology Laboratory (MTL) received one filled container representing each of the three sizes. The DS2 agent in the containers was disposed of in accordance with federal, state, and local regulations prior to examination. The corresponding lot numbers and dates of manufacture are listed in Table 1.

Table 1. CONTAINER IDENTIFICATION

Sample	Lot Number	Contract Number	Date of Manufacture
Five-Gallon Container	PLY87J001-001	DAAA09-86-C0587	5/87
14-Liter Container	ALL85L001-008	DAAK11-83-C-0066	12/85
1-1/3-Quart Container	45	DAAA09-81-C-2234	5/82

OBJECTIVES AND PURPOSE

The primary objective of this investigation is to characterize and evaluate the integrity of the protective coatings used on the exterior and/or interior surfaces of the DS2 containers and to isolate the origin of the corrosion observed on the surfaces of the containers in order to determine if it had occurred from the outside in or from the inside out.

The results of this investigation will be used to determine the feasibility of the current materials used to fabricate the DS2 containers and the coatings selected to protect the exterior and interior surfaces.

VISUAL INSPECTION AND LIGHT OPTICAL MICROSCOPY

Five-Gallon Container

Figure 1 shows the five-gallon container in the as-received condition. Overall, this container showed the least amount of corrosion when compared to the other two containers. Near the top, large areas of the green topcoat had been chipped away, as a result of mechanical damage, leaving behind a bright white undercoat, as depicted in Figures 2 and 3. In addition, all three macrographs reveal a dull, matte finish on approximately 66% of the surface area of the container. This was most likely the result of weathering and exposure to adverse storage conditions. Figure 4 illustrates an area near the bottom weld chime where the topcoat had also been damaged. To the left of this region, a few isolated blisters and corrosion of the base metal occurred. The only significant evidence of corrosion was observed at welded seams which trapped moisture and allowed crevice corrosion to take place. Figure 5 shows the bottom weld chime which contains an example of crevice corrosion at the welded seam (top of micrograph). The bottom edge of the container had experienced some general corrosion where the external surface coating had been worn away. Figure 6 represents the corrosion observed along the side weld seam which also served as a site for water entrapment.

14-Liter Container

Figures 7a and 7b show the 14-liter container in the as-received condition. This container consisted of various welded parts at the top and consequently numerous areas of corrosion were observed. On the top, general corrosion was prevalent on regions along the rounded surface, as shown in Figure 8. The protective coatings on these surfaces could easily be damaged during handling, exposing bare metal to the environment. Crevice corrosion occurred on both latches at regions which are welded to the container and support the handle. These latches did not have a continuous weld and consequently contained crevices where moisture could collect and cause corrosion, as revealed in Figures 9a and 9b. Corrosion was also noticed where the top containment latch came into contact with the surface of the container. General corrosion was observed on the handle itself, (refer to Figure 10) as its concave design allowed water to puddle. In addition, general corrosion occurred on the base of the container where it came in contact with the ground or another support, as Figure 11 illustrates. Here, the coating appeared to have been lifted away exposing the base metal. Overall, some of the design features of this container were conducive to corrosion.

1-1/3-Quart Container

Figure 12 is a macrograph of the 1-1/3-quart container in the as-received condition. This container appeared to be in the worst overall condition showing evidence of blistering, denting, and staining, as well as varying degrees of corrosion. This condition may be attributed to the fact that it was the oldest container under investigation. The top surface, especially near the closure, exhibited extensive general corrosion, as shown in Figure 13. The entire top of the container being recessed allowed water to collect. This cup design provided a haven for corrosion. The side seam consisted of overlapping material which formed a longitudinal gap along the exterior of the container where crevice corrosion was predominant, as shown in Figure 14. Figure 15 contains a graphic example of the extensive blistering which occurred randomly over the exterior surface. Blistering is one of the first signs of the breakdown in the protective nature of a coating. The blisters were local regions where the coating had lost adherence from the substrate and where water accumulated and accelerated deterioration. Extensive corrosion was seen beneath blisters that had been scraped off during examination. In some instances, solder spatter and other surface asperities were observed beneath the paint blisters. Surface debris such as greases, shop dirt, etc. can also lead to poor coating adhesion and cause blistering. Figure 16 shows an example of general corrosion that took place at the bottom crimp-solder where the coating had been damaged when the container rested against a supporting surface.

It is important to note that no evidence of corrosion was observed on the interior surfaces of all three containers. This was determined by performing another visual examination after the containers had been emptied and sectioned for metallographic analysis.

METALLOGRAPHIC EXAMINATION

Five-Gallon Container

Figures 17 and 18 are schematics which identify the areas where cross-sectional specimens were taken from the five-gallon container. The corresponding surfaces which were prepared for metallographic examination are also indicated. Table 2 lists the specimens that were sectioned from the container.

Table 2. METALLOGRAPHIC SPECIMENS SECTIONED FROM THE FIVE-GALLON CONTAINER

- Top Weld Chime
- Bottom Weld Chime
- Side Weld Seam
- Top Closure

The required coating system and thickness specification for the five-gallon container was outlined in Section 3.10.2 of MIL-P-51529 dated 11 December 1986 and is shown in Table 3. This is the specification that was presumably in effect during the fabrication of the five-gallon container.

Table 3. COATING SYSTEM OF THE FIVE-GALLON CONTAINER

	Coating	Thickness (mils)	Specification
1.	Phosphate, TYPE I (spray) or Wash Primer	*	TT-C-490 DOD-P-15328
2.	Epoxy Primer	0.8 - 1.4	MIL-P-51529
3.	Polyurethane Topcoat	1.8 - 2.4	MIL-P-51529

*The zinc phosphate coating is required to be a minimum of 3.2 g/m^2 when application is performed by dipping, and 1.6 g/m^2 when the coating is sprayed.

Figure 19 shows a representative cross section of the coating system of the five-gallon container taken from the side weld. This optical micrograph was exposed under polarized light in order to highlight the different coating layers.

In general, the coatings displayed good adherence and were uniform, except for the phosphate layer which appeared to be quite discontinuous. Figure 20 further illustrates this point as it shows a thick phosphate layer, in contrast to Figure 19 which revealed only trace amounts of this coating. Figure 20 represents an area sectioned from the side weld where the topcoat was nonuniform and thinner than the specification permitted. Figure 21 shows a cross section of the top weld which did not contain a topcoat layer. As previously described, within the Visual Inspection section of this report, areas of the topcoat had been damaged contributing to the nonuniformity of the topcoat. Regions where the closure contacted the top of the container were especially susceptible to this type of damage.

Figure 22 represents the variation observed in the thickness of coatings located on surfaces that were sloped and uneven or within welded areas. This photograph reveals the exterior coatings on the bottom weld of the five-gallon container.

The variation in coating thickness near welds or seams was also observed on the remaining two containers examined. Welds and seams either contained an excessive coating thickness or an inadequate coating depending upon the slope and the geometry of the area being coated.

14-Liter Container

Figures 23 and 24 are schematics which identify the areas where cross-sectional specimens were taken from the 14-liter container. The corresponding surfaces which were prepared for metallographic examination are also indicated. Table 4 lists the specimens that were sectioned from the container.

Table 4. METALLOGRAPHIC SPECIMENS
TAKEN FROM THE 14-LITER CONTAINER

- Top of Container
- Bottom Weld Chime
- Side A
- Side B
- Side C
- Side D Weld Seam

The required coating system and thickness specifications for the 14-liter container was outlined on U.S. Army Armament, Munitions and Chemical Command Research, Development and Engineering Center (AMCCOM), Engineering Drawing Number E5-51-528, dated 5 February 1982, and is shown in Table 5. The additional specifications listed include the description, characteristics, usage, and quality assurance provisions of the coating system. The particular 14-liter container under investigation was designated to have a Chemical Agent Resistant Coating (CARC) which consisted of an epoxy primer and a urethane topcoat. A wash primer was applied on the substrate in preparation for the CARC system.

Figure 25 shows a representative cross section of the coating system of the 14-liter container taken from Side B. The use of polarized light was effective in distinguishing the various coating layers. The wash primer was best examined utilizing electron microscopy since it was only about 0.0002 inches in thickness and required analysis at higher magnification. It appears as a fine dark line in the photograph. The epoxy primer layer is red in appearance while the urethane topcoat is green.

Table 5. COATING SYSTEM OF THE 14-LITER CONTAINER

	Coating	Thickness (mils)	Specification
1.	Wash Primer	0.2 - 0.5	DOD-P-15328
2.	Prime Coat	0.8 - 1.4	MIL-P-53022 or MIL-P-53030
3.	Topcoat	1.8 - 2.4	MIL-C-46168 or MIL-C-53039

In Figure 25 the epoxy primer is approximately 0.6 mils in thickness while the topcoat averages about 1.8 mils which conformed to the lower range of the specified requirements. This trend was observed throughout most of the specimens examined. The urethane coating was also nonuniform at the top and bottom of the container and the epoxy primer was very discontinuous in these same areas, as Figure 26 reveals. The epoxy primer appeared spotty beneath the green topcoat instead of as a continuous layer. The photograph clearly shows the green urethane coating in intimate contact with the wash primer on the substrate and the absence of a red epoxy primer coating on most of the specimen.

The poor condition of the epoxy primer may have been the result of improper handling of the container between coating applications which lead to damage or nonuniform coverage. The specimens representing the sides of the container all displayed uniform but slightly thinner than specified epoxy primer coatings and topcoats.

1-1/3-Quart Container

Figures 27 and 28 are schematics which identify the areas where cross-sectional specimens were taken from the 1-1/3-quart container. The corresponding surfaces which were prepared for metallographic examination are also indicated. Table 6 lists the specimens that were sectioned from the container.

Table 6. METALLOGRAPHIC SPECIMENS
FROM THE 1-1/3-QUART CONTAINERS

- Top crimp solder
- Bottom crimp solder
- Side solder
- Top closure

The required coating system for the 1-1/3-quart container was outlined on AMCCOM, Engineering Drawing Number B5-51-382, and is shown in Table 7. The 1-1/3-quart container was designated to be fabricated from material conforming to ASTM A 623 "Tin Mill Products, General Requirements" which consists of a low carbon steel that is dipped into a molten mixture of lead and tin. The exterior surface of the container was subsequently coated with an alkyd paint after fabrication.

Table 7. COATING SYSTEM OF THE
1-1/3-QUART CONTAINER*

	Coating
1.	Interior and Exterior Terne
2.	Exterior Alkyd Topcoat

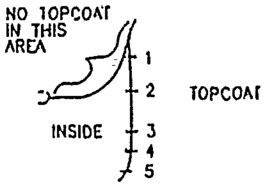
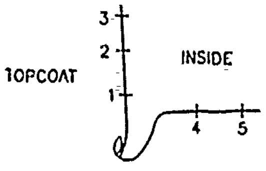
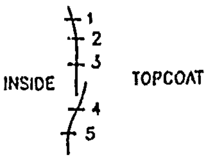
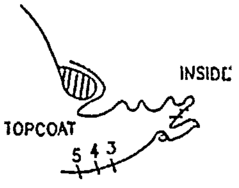
*NOTE: No required thickness was specified for the alkyd coating on the 1-1/3-quart container engineering drawings or on MIL-P-51529.

Figure 29 shows a representative cross section of the interior terne coating taken from the top weld of the 1-1/3-quart container. Polarized light was once again utilized to highlight the various coating layers. The terne coating appears as a bright "sparkling" green layer while the steel substrate has a bluish tint. Figure 30 shows the terne coating and the alkyd topcoat which is characterized as the thicker pale green layer. The topcoat is approximately 1.5 mils in this photograph and the area examined represents the bottom weld in which the alkyd coating varied significantly in thickness along the length of the specimen. Figure 31 shows a variation in alkyd coating thickness near the bottom weld of 1-1/3-quart container. Figure 32 shows a cross section of the coatings located at the top weld where a void was found in the alkyd topcoat.

COATING THICKNESS MEASUREMENTS

Each cross-sectional specimen taken from the three containers was examined in order to measure the various coating layers. Measurements were performed from optical micrographs or from electron micrographs when the coatings were very thin; i.e., zinc phosphate and wash primer. Two measurements were taken of each sample within 2 mm of visible corrosion when possible; some areas of the containers examined showed no evidence of corrosion. The zinc phosphate coating was weighed in accordance with DOD-P-16232 when applicable (see Zinc Phosphate Weight Determination section). The only container examined that was zinc phosphated was the five-gallon container. The measurements of the various coating layers on each of the three containers is listed in Tables 8 through 10.

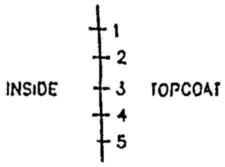
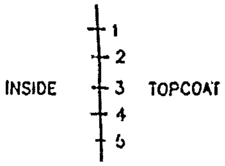
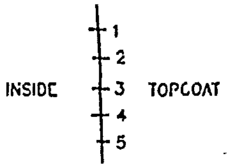
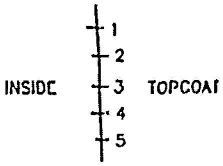
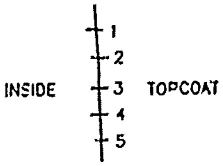
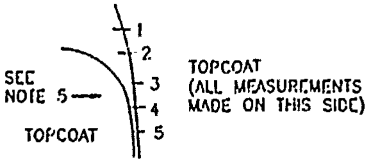
Table 8. FIVE-GALLON DS2 CONTAINER COATING THICKNESS MEASUREMENTS

Sample	Location	Expoxy Primer Undercoat (mils)	Urethane Topcoat (mils)	Notes	Metallographic Sample
Top Weld	1	2.1	1.6	1,2	
	2	2.3	2.3		
	3	2.0	2.5		
	4	1.9	1.8		
	5	2.1	2.3		
Average		2.1	2.1		
Bottom Weld	1	2.3	1.9*		
	2	2.4	2.0		
	3	2.1	1.9		
	4	2.1	1.8*		
	5	3.0	1.3		
Average		2.4	1.8		
Side Weld	1	2.1	1.4		
	2	1.9	1.3		
	3	2.0	1.5*		
	4	2.1	1.4*		
	5	2.1	2.1		
Average		2.1	1.5		
Closure	1	3.3	2.4		
	2	3.0	2.1		
	3	3.1	1.0		
	4	3.6	0.9		
	5	2.9	0.6		
Average		3.2	1.4		
Total Average		2.4	1.4		
Requirement per MIL-P-51529		0.8 - 1.4 mils	1.8 - 2.4 mils		

Key to Notes:
1. Poor topcoat
2. No topcoat or undercoat in some areas

* Readings measured within 2 mm of visible corrosion.

Table 9. 14-LITER DS2 CONTAINER COATING THICKNESS MEASUREMENTS

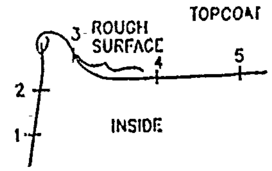
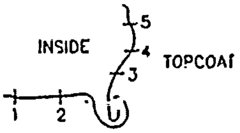
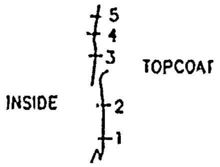
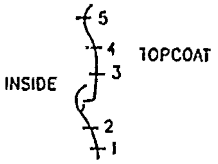
Sample	Location	Wash Primer (mils)	Epoxy Primer Undercoat (mils)	Urethane Topcoat (mils)	Notes	Metallographic Sample
Side A	1	0.1	0.5	0.7*	1,2	
	2	0.1	0.4	0.8		
	3	0.1	0.4	0.4		
	4	0.1	0.5	0.6		
	5	0.1	0.3	0.6*		
Average		0.1	0.4	0.6		
Side B	1	0.2	0.5	1.3	3	
	2	0.2	0.4	1.9		
	3	0.2	0.6	2.3		
	4	0.2	0.7	2.4		
	5	0.1	0.5	1.1		
Average		0.2	0.5	1.8		
Side C	1	0.1	0.4	0.9		
	2	0.1	0.5	0.9		
	3	0.1	0.4	0.7		
	4	0.1	0.3	1.0		
	5	0.1	0.4	1.0		
Average		0.1	0.4	0.9		
Side D (Weld)	1	0.1	0.5	0.8	1,3,4	
	2	0.1	0.4	0.8		
	3	0.1	0.4	0.6		
	4	0.2	0.4	0.8		
	5	0.1	0.4	0.7		
Average		0.1	0.4	0.7		
Top	1	0.2	0.0	1.3*	2,5	
	2	0.2	0.0	1.4		
	3	0.1	0.0	1.3		
	4	0.1	0.0	1.8		
	5	0.1	0.0	1.4*		
Average		0.1	0.0	1.4		
Bottom Weld	1	0.2	0.7	1.1	5	
	2	0.2	0.6	1.1		
	3	0.1	0.6	1.2		
	4	0.1	0.8	1.6		
	5	0.2	0.8	2.1		
Average		0.2	0.7	1.4		
Total Average		0.1	0.5	1.1	6	
Requirement per Eng. Dwg. E5-51-528		0.2 - 0.5 mils	1.8 - 2.4 mils	1.8 - 2.4 mils		

Key to Notes:

1. Poor Topcoat
2. Poor undercoat
3. Inconsistent topcoat
4. Some of topcoat runs into lip
5. Undercoat in beads, not measurable
6. Excluding 'top' data set

* Readings measured within 2 mm of visible corrosion.

Table 10. 1-1/3-QUART DS2 CONTAINER COATING THICKNESS MEASUREMENTS

Sample	Location	Interior Terne (mils)	Exterior Terne (mils)	Topcoat (mils)	Notes	Metallographic Sample
Top Solder	1	0.3	0.2	1.3	1	
	2	0.2	0.1	1.1*		
	3	0.1	0.2	1.4*		
	4	0.1	0.4	2.2		
	5	0.3	0.2	1.6		
Average		0.2	0.2	1.5		
Bottom Solder	1	0.1	0.2	0.9	2	
	2	0.1	0.3	1.0*		
	3	0.1	0.3	1.4*		
	4	0.1	0.4	1.8		
	5	0.2	0.2	1.1		
Average		0.1	0.3	1.2		
Side Solder	1	0.8	0.6	1.8	1,3	
	2	0.3	0.2	2.2		
	3	0.1	0.1	1.4*		
	4	0.4	0.1	1.6*		
	5	0.6	0.1	1.4		
Average		0.4	0.1	1.7		
Closure	1	0.3	0.0	2.9	1,2,4	
	2	0.2	0.0	1.8*		
	3	0.3	0.0	2.9*		
	4	0.1	0.0	2.6		
	5	0.1	0.0	1.5		
Average		0.2	0.0	2.3		
Total Average		0.2	0.1	1.7		

Key to Notes:
1. Poor terne coating on exterior surface
2. Poor terne coating on interior surface
3. No topcoat on some areas
4. Topcoat broken up/damaged

*Readings measured within 2 mm of visible corrosion.

CHARACTERIZATION OF THE COATING SYSTEMS

Energy dispersive spectroscopy (EDS) and infrared spectroscopy were utilized to characterize the surface coatings located on all three containers. Metallographic samples and actual chips of the coating were placed in the scanning electron microscope (SEM) and analyzed by EDS techniques. When performing infrared spectroscopy, samples of the coating were chipped off, pulverized, and pressed in a KBr matrix. Absorption spectra were obtained from a Perkin-Elmer Model 1700 Fourier Transform Infrared Spectrometer.

Five-Gallon Container

Figure 33 shows an SEM micrograph of the coatings located on the bottom weld of the five-gallon container. Although the epoxy primer and urethane topcoat are not as readily distinguishable as in previous optical photographs, the phosphate layer exhibits good contrast. Figure 34 reveals the phosphate layer at higher magnification and represents area "a", as identified in Figure 33. Figure 35 is the resulting spectrum of the zinc phosphate coating which shows high zinc and phosphorus peaks while the iron peak represents the steel substrate. Figures 36 and 37 are the spectrum obtained from analysis of the epoxy primer and urethane topcoat, respectively. Titanium dioxide is a constituent added to the primer as a white coloring agent while chromium, silicon, and oxygen represent common pigments of the topcoat. Figure 38 is the infrared spectrum collected from the topcoat which is indicative of a urethane.

14-Liter Container

Figure 39 shows an SEM micrograph of the coatings located on Side C of the 14-liter container. The photograph identifies the various layers. Figure 40 is the EDS spectrum of the wash primer showing high peaks of phosphorus, zinc, and chromium, as well as iron which represents the steel substrate. Figures 41 and 42 contain the spectrum of the epoxy primer and topcoat, respectively. Magnesium, silicon, and iron are contained in pigments added to the epoxy primer for rust inhibitive properties and color. Figure 43 is the infrared spectrum of the topcoat which is indicative of a polyurethane.

1-1/3-Quart Container

Figure 44 is an SEM micrograph of the exterior coatings located on the top weld of the 1-1/3-quart container. The black region separating the alkyd coating from the terne coating is an interface where there is no adhesion. The photograph identifies the terne coating and the alkyd topcoat. Figure 45 shows the interior terne coating observed on the top weld cross section. Both interior and exterior terne coatings were uniform and displayed good adhesion to the steel substrate. Figures 46 and 47 reveal the resulting spectrum obtained from the exterior and interior terne coating, respectively. The only major peaks detected consisted of lead and tin which are the primary elemental constituents of the coating. Figure 48 is the resulting spectrum of the alkyd coating present on the 1-1/3-quart container. Figure 49 represents the infrared spectrum of the same coating which indicates that the coating was an alkyd.

ZINC PHOSPHATE WEIGHT DETERMINATION

The zinc phosphate coating located on the five-gallon container was measured in accordance with the requirements established in DOD-P-16232 (Section 4.8.4.2). A total of three separate test panels were sectioned from the container in areas where no corrosion was present and a relatively uniform topcoat was observed. A commercial paint stripper (Park's) was utilized to remove the topcoat and epoxy primer before the zinc phosphate coating could be measured. The paint stripper contained methylene chloride, methanol, and toluol. These compounds are organic and should not have attacked the zinc phosphate layer.

Upon removal of the supplementary treatment, each sample was weighed to the nearest tenth of a milligram. The zinc phosphate was subsequently removed in a 50 g/l chromic acid solution at 74°C (165°F). The samples were immersed for 15 minutes at this temperature, removed, rinsed in clean running water, dried, and reweighed. This procedure was repeated twice (for a total of three strippings) before the weight became constant. The calculation of the zinc phosphate coating weight for each sample is as follows:

ZINC PHOSPHATE WEIGHT CALCULATIONS

	Sample 1	Sample 2	Sample 3
Total Surface Area:	$= 1.45\ in^2$	$= 1.45\ in^2$	$= 14.53\ in^2$
	$= 9.37 \times 10^{-4} m^2$	$= 9.37 \times 10^{-4} m^2$	$= 9.38 \times 10^{-3} m^2$
Initial weight	= 3.7937 g	= 3.8201 g	= 38.5655 g
Weight after 1st stripping	= 3.7913 g	= 3.8175 g	= 38.5408 g
Weight after 2nd stripping	= 3.7909 g	= 3.8170 g	= 38.5408 g
Weight after 3rd stripping	= 3.7909 g	= 3.8169 g	= 38.5408 g

The zinc phosphate weight was calculated as outlined in DOD-P-16232, where:

$$\text{Weight}(g/m^2) = \frac{(\text{Initial weight in grams} - \text{final weight in grams})}{\text{total area in square meters}}$$

Sample 1 Zinc Phosphate Weight:

$$\frac{3.7937\ g - 3.7909\ g}{9.37 \times 10^{-4} m^2} = 2.99\ g/m^2$$

Sample 2 Zinc Phosphate Weight:

$$\frac{3.8201\ g - 3.8169\ g}{9.37 \times 10^{-4} m^2} = 3.41\ g/m^2$$

Sample 3 Zinc Phosphate Weight:

$$\frac{38.5655\ g - 38.169\ g}{9.38 \times 10^{-3} m^2} = 2.63\ g/m^2$$

$$\text{AVERAGE WEIGHT} = 3.01\ g/m^2$$

The governing specification for the zinc phosphate coating procedure for the five-gallon container TT-C-490 states that the weight of the coating (Section 3.5.2) shall be 1.6 g/m^2 for spray application and 3.2 g/m^2 for dip application.

SUMMARY OF FINDINGS

The corrosion observed on all three containers examined initiated from exterior surfaces. No evidence of corrosion was found on the interior surfaces of the containers. The most severely corroded areas were those located at welds, soldered seams, and recesses which provided a suitable haven for corrosion.

Five-Gallon Container

The zinc phosphate coating weight was slightly lower than specified and showed some evidence of inconsistent coverage. The epoxy primer exceeded the thickness requirements by approximately 1 mil while the urethane topcoat measured just below the minimal thickness specification. This container displayed the least amount of corrosion of those examined which may have been attributable in part to the thick epoxy primer layer which was very adherent and relatively uniform. The primer and topcoat did show inconsistency near welds and seams and also on surfaces that were sloped and uneven. In addition, these two coatings narrowed down to a very thin film in regions near welds and seams and it was very common to observe very little or almost no coverage in these areas.

14-Liter Container

The wash primer coating measured less than minimum thickness requirements. Also, the epoxy primer did not meet minimal thickness specifications and was nonuniform in various regions, especially at the top and bottom of the container. Overall, however, the primer and topcoat were uniform and displayed good adherence. The urethane topcoat was slightly lower in thickness than required but to a lesser degree than the two previous coatings discussed. Crevice corrosion was prevalent within welded seams and at the top latches and handle. General corrosion occurred at contact areas at the bottom and at rounded surfaces.

1-1/3-Quart Container

The interior and exterior terne coating showed some inconsistency near the bottom of the container and at solder joints. The thickness of the alkyd topcoat was nonuniform in sloped regions and at soldered seams. Since lead and tin provide cathodic action for the steel container, once this coating has been damaged and the steel becomes exposed to an aggressive environment, corrosion can occur rapidly. In contrast to sacrificial metallic coatings, such a cadmium or zinc, lead does not offer itself up as a sacrificial anode during the corrosion process. As a result, the 1-1/3-quart container showed extensive blistering and general corrosion, especially at the top which was recessed and allowed water to puddle.

DISCUSSION

Carbon steels are very corrosive in humid and marine environments and most cost-effective protective coating systems will eventually break down or become damaged allowing corrosion to take place and, thus, limiting the useful service life of the container. This creates a hazardous situation when leakage of the DS2 agent occurs and also renders the DS2 in the container useless. Carbon steels could be utilized to fabricate the containers "in principle" if properly manufactured, coated, handled, and stored but in reality it has been clearly demonstrated that under typical conditions corrosion has taken place during storage. In addition, it has been revealed that many of the design features of the container made them more susceptible to corrosion.

These include welded/soldered seams, spot welds, and recessed areas. Therefore, it has been proposed to utilize alternative materials to fabricate the DS2 containers.

RECOMMENDATIONS

Carbon steel could continue to be utilized to fabricate the DS2 containers if the following features are incorporated:

- Elimination of poor design features which promote corrosion,
- Ensurance of coating integrity and adequacy during all stages of surface preparation, application, and handling with particular attention to the protection of all joints,
- Periodic inspection of containers in storage by visual examination for early detection of possible corrosion of containers and leakage of the DS2 agent.

More corrosion resistant metals such as stainless steel could also be utilized. However, if the poor design features of the container are not eliminated or improved, some of the same problems may occur again, such as observed within welded/soldered seams and on surfaces that allowed water to puddle.

A study previously performed for CRDEC* indicated viable alternatives to steel containers. Associated costs of welding, soldering, and coating could be eliminated. A molded plastic or fiberglass composite container could be designed to facilitate stacking and handling. The most applicable plastics would be either polyethylene with an internal fluorocarbon barrier or polyetheretherketone (a semicrystalline aromatic thermoplastic). The fiberglass composite would consist of a polymeric matrix reinforced with various types of fibers. Further laboratory testing would need to be conducted to determine which alternative is the most suitable for the application.

Consideration of aluminum is also worthwhile. Attack by DS2 is not a problem as long as water is excluded.

ACKNOWLEDGMENTS

The authors would like to thank Jack Mullin and Andy Zani for their metallographic work, Dan Snoha for specimen machining, Bradley M. Taber, III for electron microscopy, and Jim Sloan for infrared analysis.

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*STARK, DAVID C., and MENGEL, R. WILLIAM. *DS2 Container and Weatherproofing Study*. CRDEC-CR-091, December 1990.

Figure 1. Five-gallon container in the as-received condition. Reduced 70%

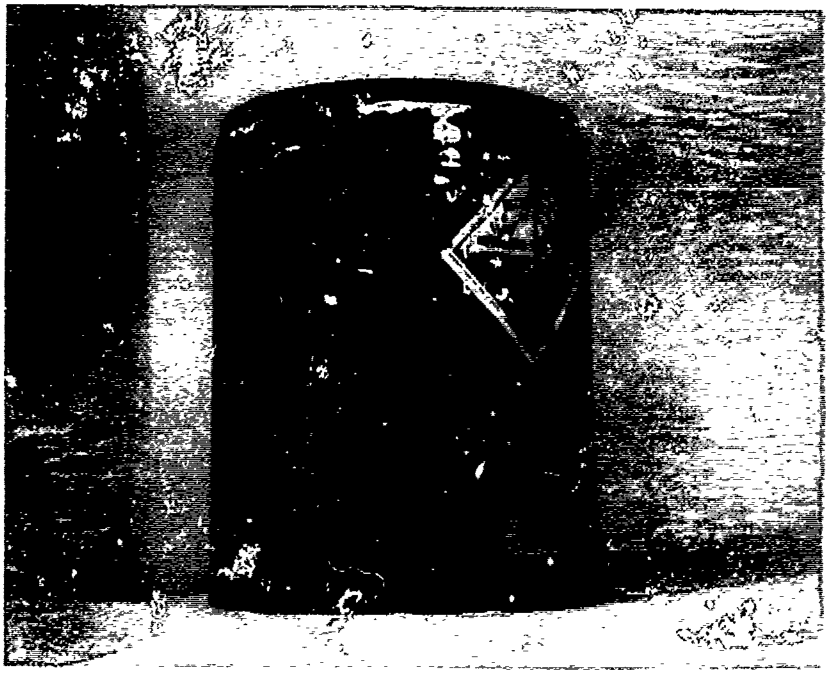


Figure 2. Prior mechanical damage to five-gallon container revealing bright white undercoat. Reduced 85%

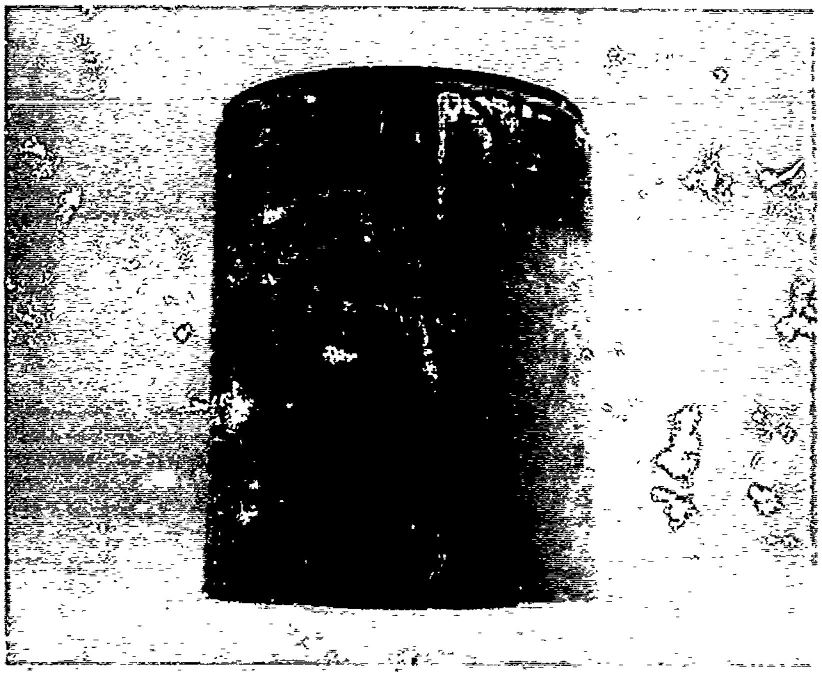


Figure 3. Another view of prior mechanical damage to five-gallon container revealing bright white undercoat. Reduced 85%

Figure 4. Area of five-gallon container near bottom weld chime illustrating topcoat damage. Mag. 1X

Figure 5. Bottom weld chime of five-gallon container showing signs of crevice corrosion (refer to top of micrograph). Mag. 7.5X

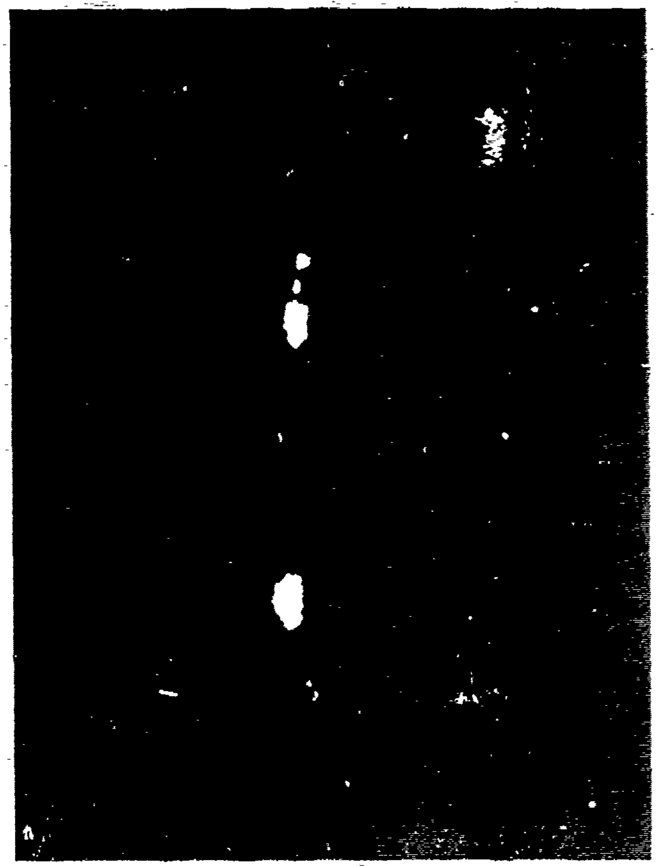


Figure 6. Corrosion observed along the side weld seam of five-gallon container. Mag. 7.5X

Top view. Reduced 85%

Side view. Reduced 70%

Figure 7. 14-liter container in the as-received condition.

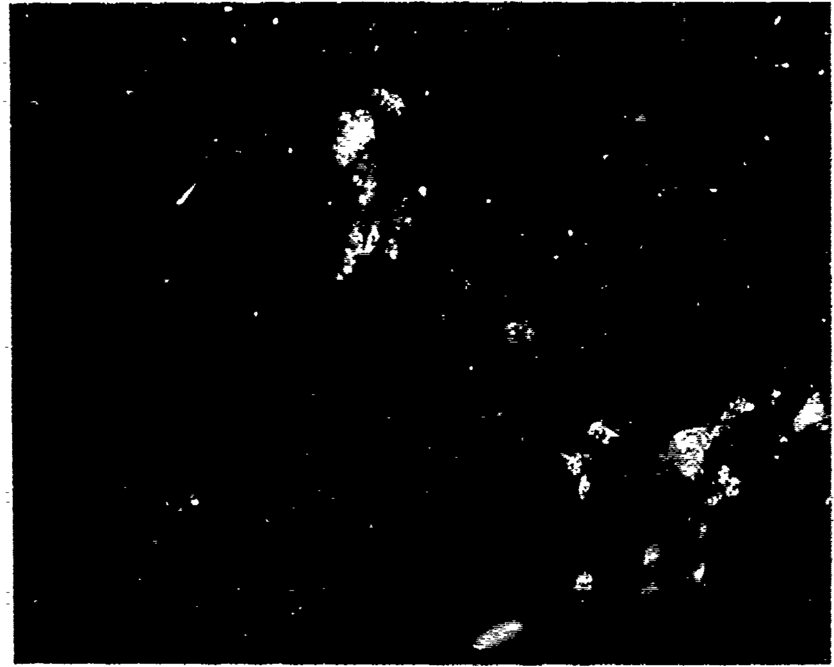


Figure 8. General corrosion prevalent on the top of the 14-liter container. Mag. 7.5X

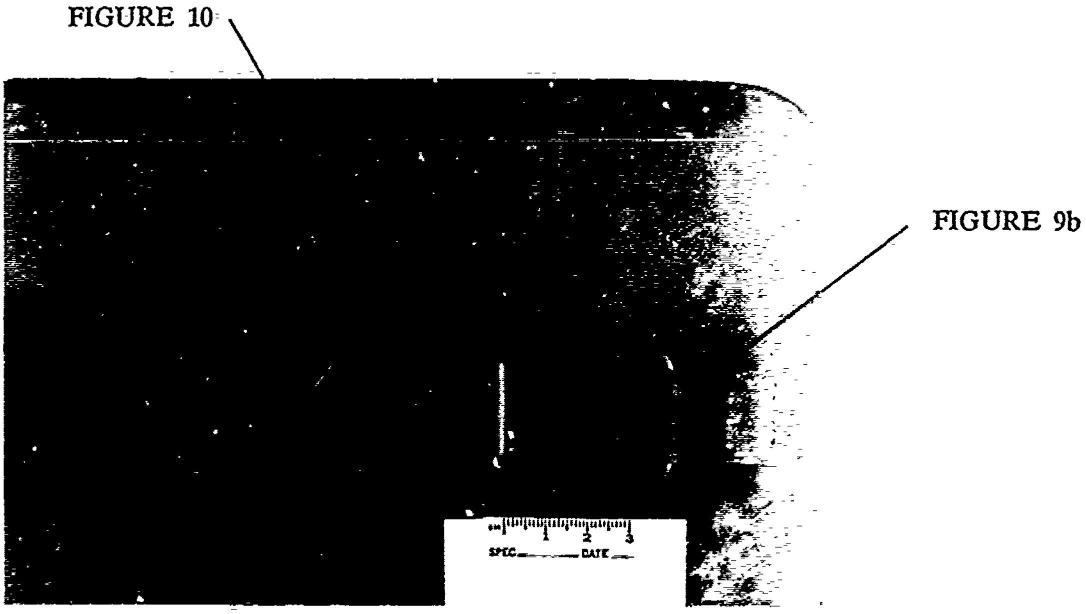


Figure 9a. Crevice corrosion on latch of 14-liter container. Reduced 25%

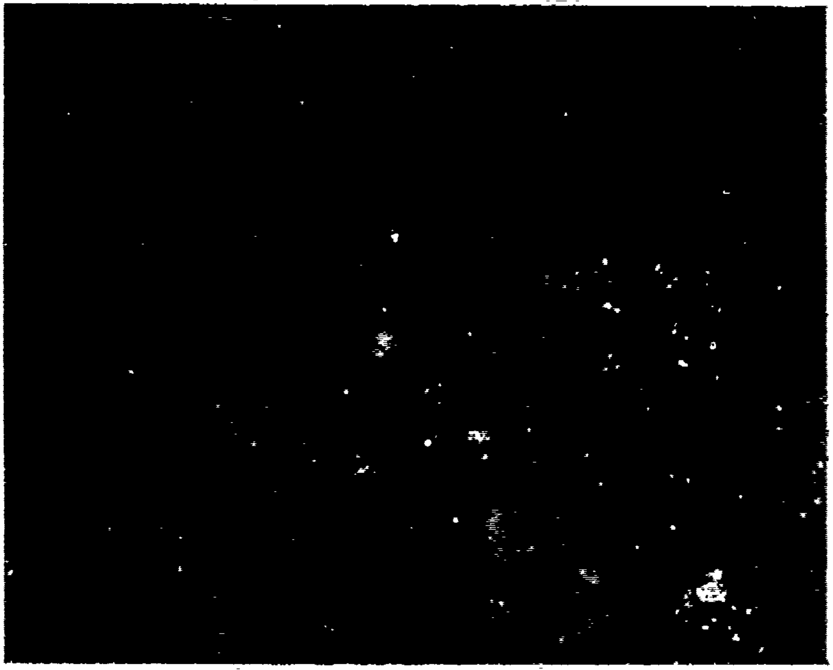


Figure 9b. Magnified view of crevice corrosion on latch of 14-liter container. Mag. 7.5X

Figure 10. General corrosion on handle of 14-liter container. Mag. 7.5X

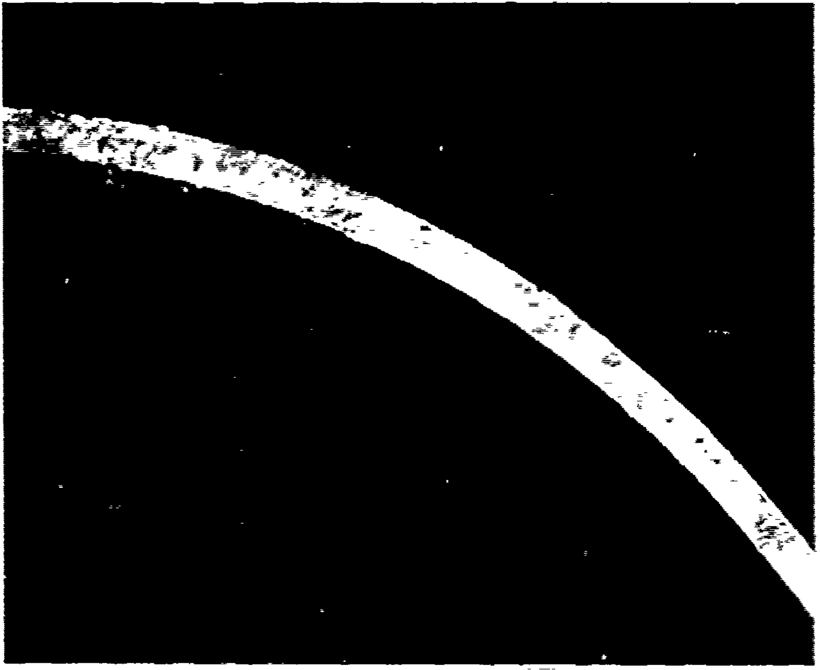


Figure 11. General corrosion at the base of the 14-liter container. Mag. 7.5X

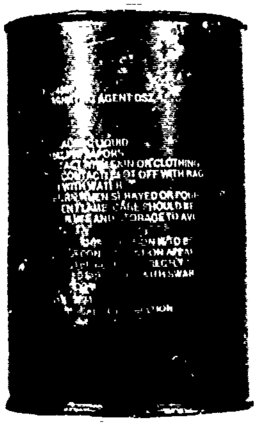


Figure 12. 1-1/3-quart container in the as-received condition. Reduced 70%

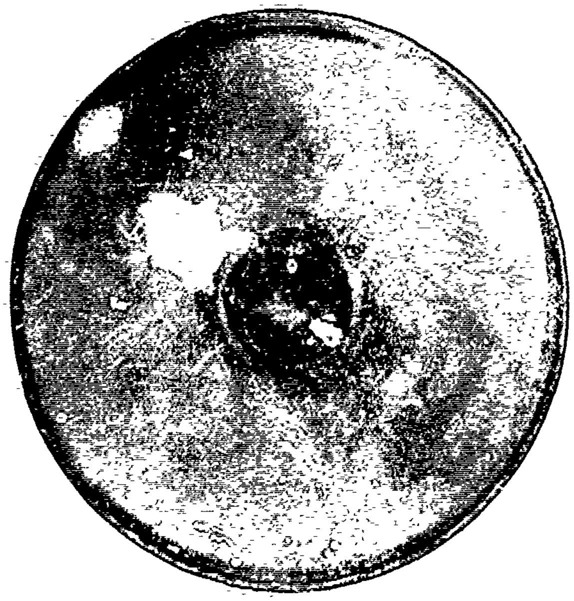


Figure 13. Extensive general corrosion along the top surface of the 1-1/3-quart container. Reduced 20%

Figure 14. Crevice corrosion along side seam of 1-1/3-quart container. Mag. 15X

Figure 15. Example of extensive blistering prevalent on exterior surface of 1-1/3-quart container. Mag. 7.5X

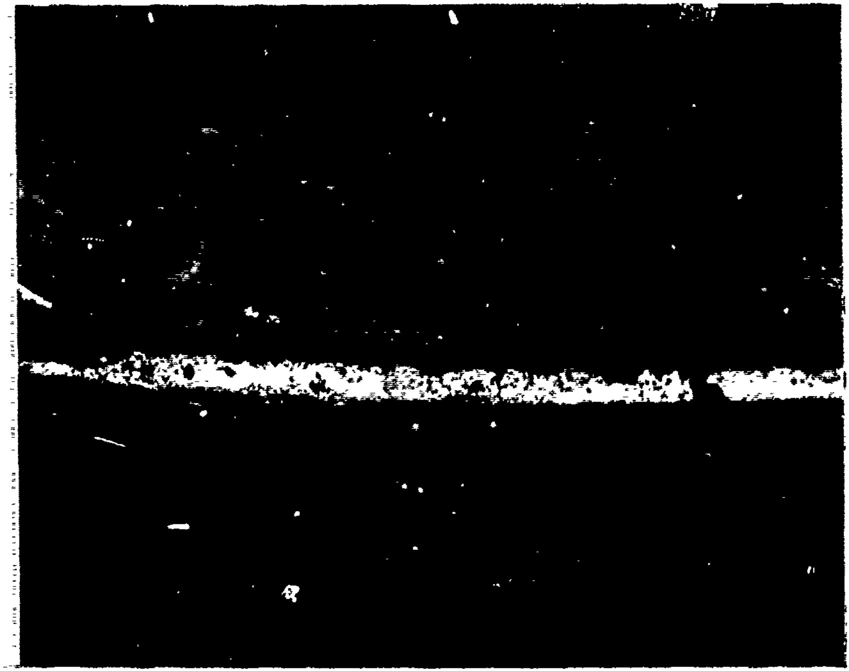


Figure 16. General corrosion present at the bottom crimp-solder of 1-1/3-quart container. Mag. 15X

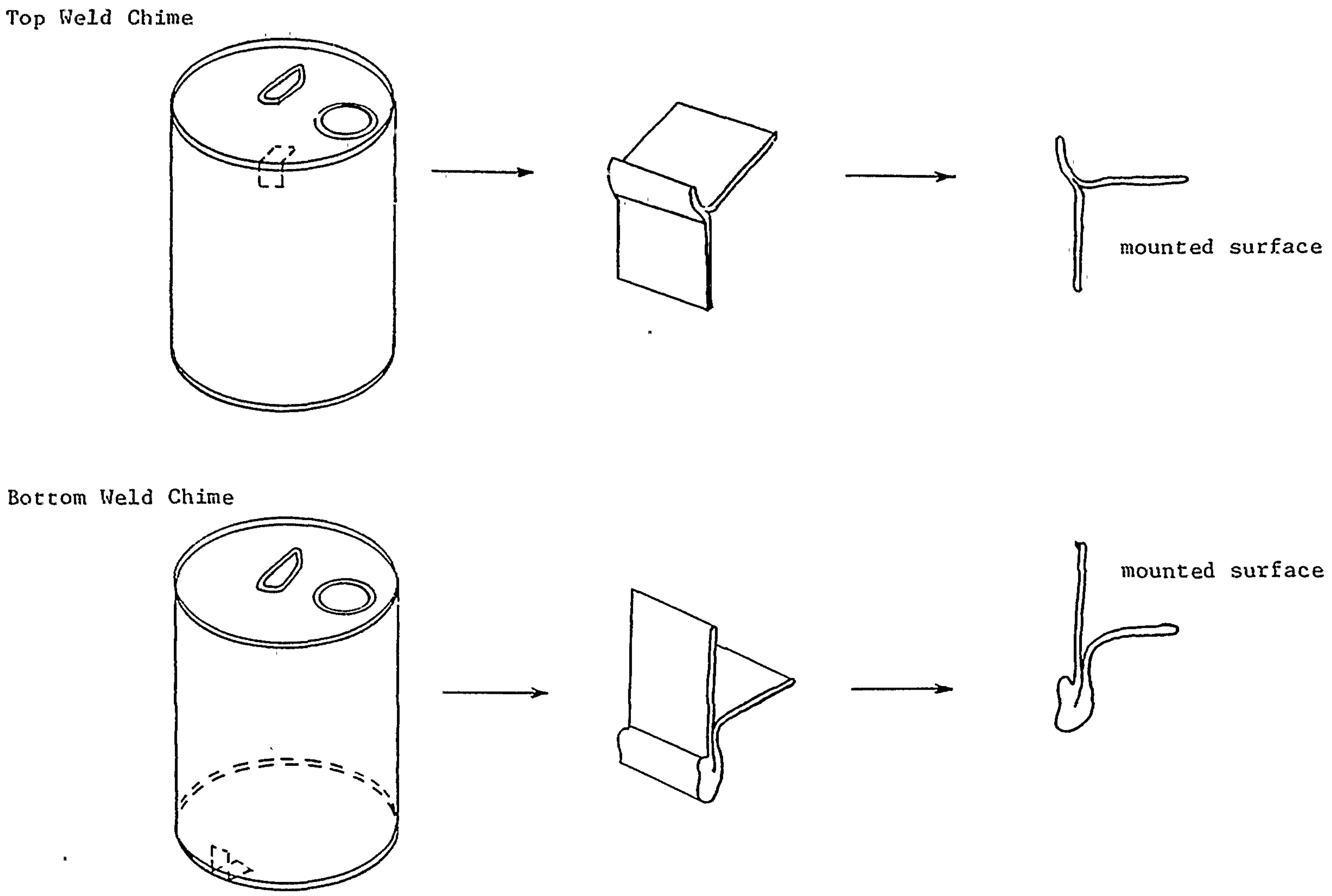


Figure 17. Metallographic cross sections of five-gallon container.

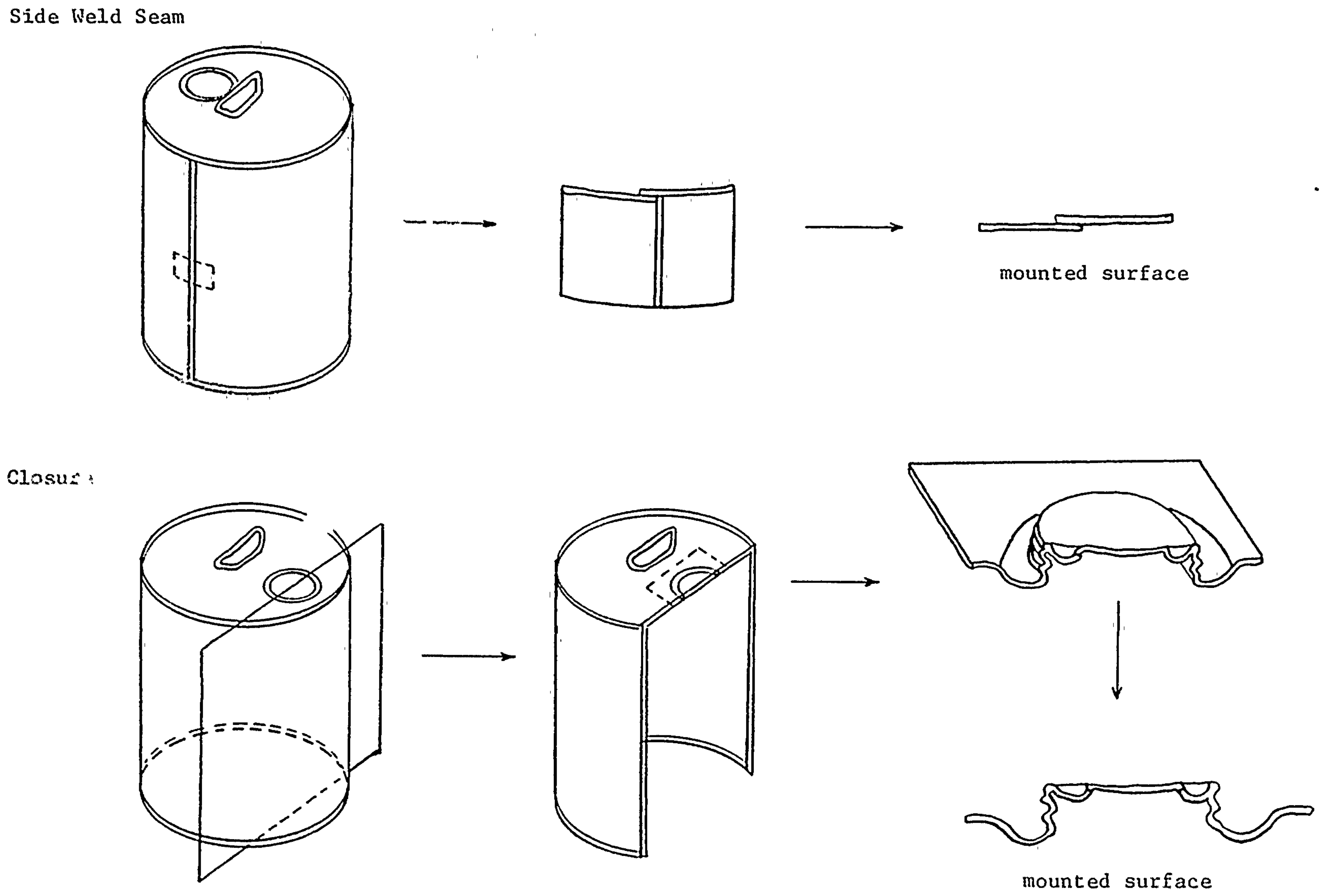


Figure 18. Metallographic cross sections of five-gallon container.

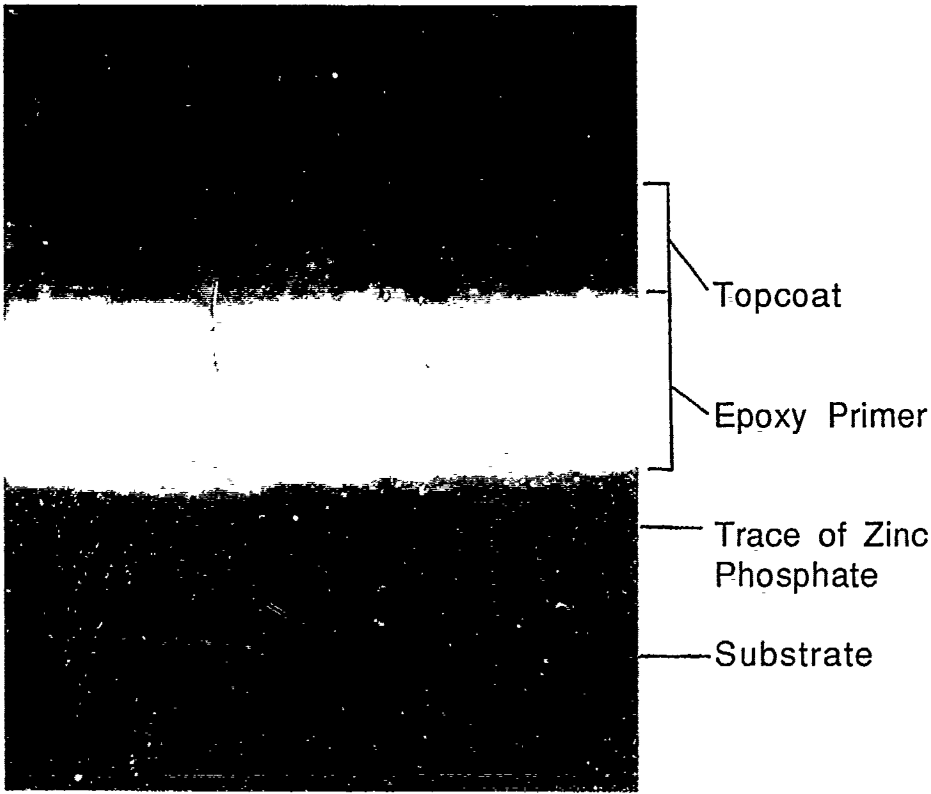


Figure 19. Representative cross section of the coating system of the five-gallon container taken from the side weld area. Mag. 500X

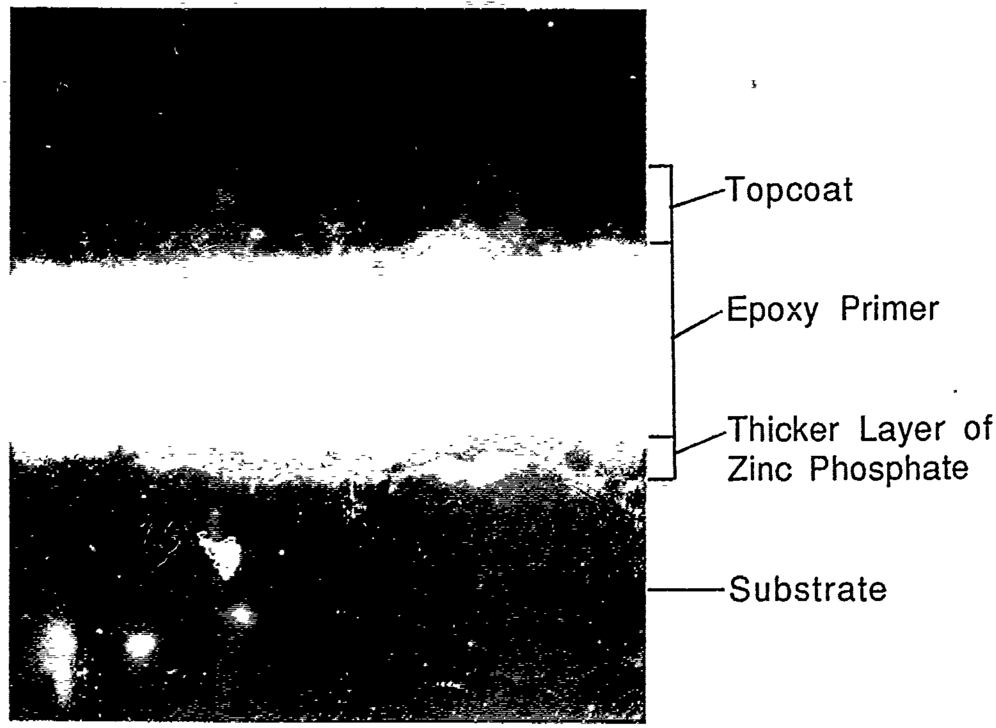


Figure 20. Representative cross section of the coating system of the five-gallon container taken from the side weld area, showing a thicker zinc phosphate coating. Mag. 500X

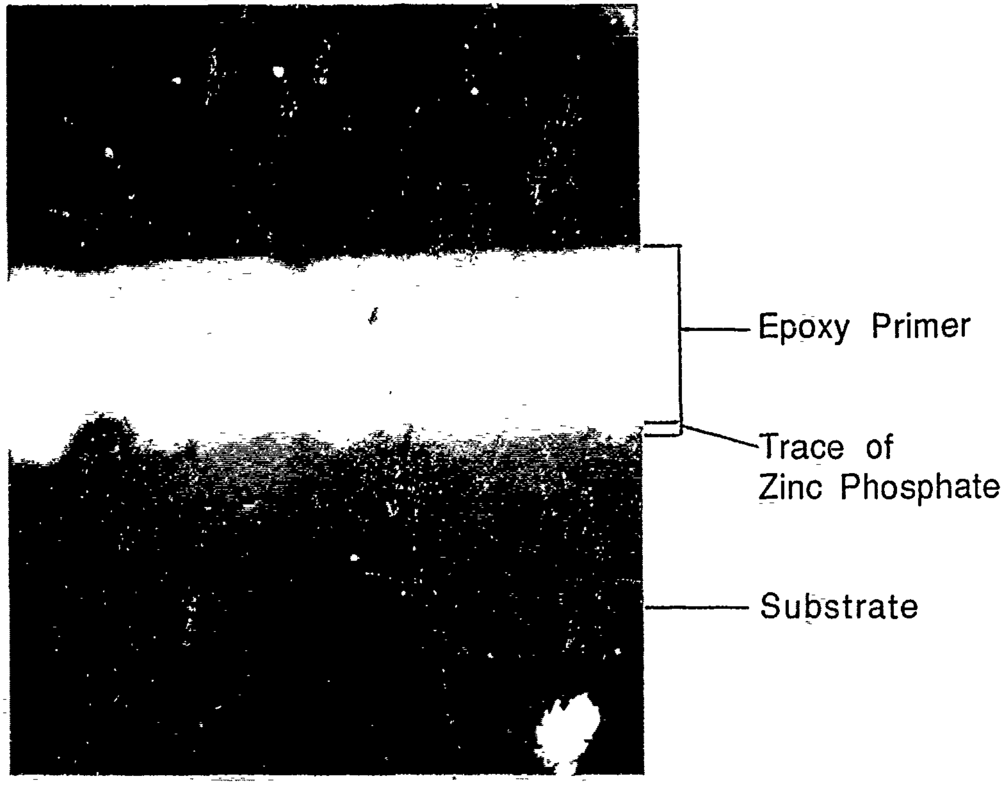


Figure 21. Cross section of the top weld of the five-gallon container indicating the nonexistence of a topcoat layer. Mag. 500X

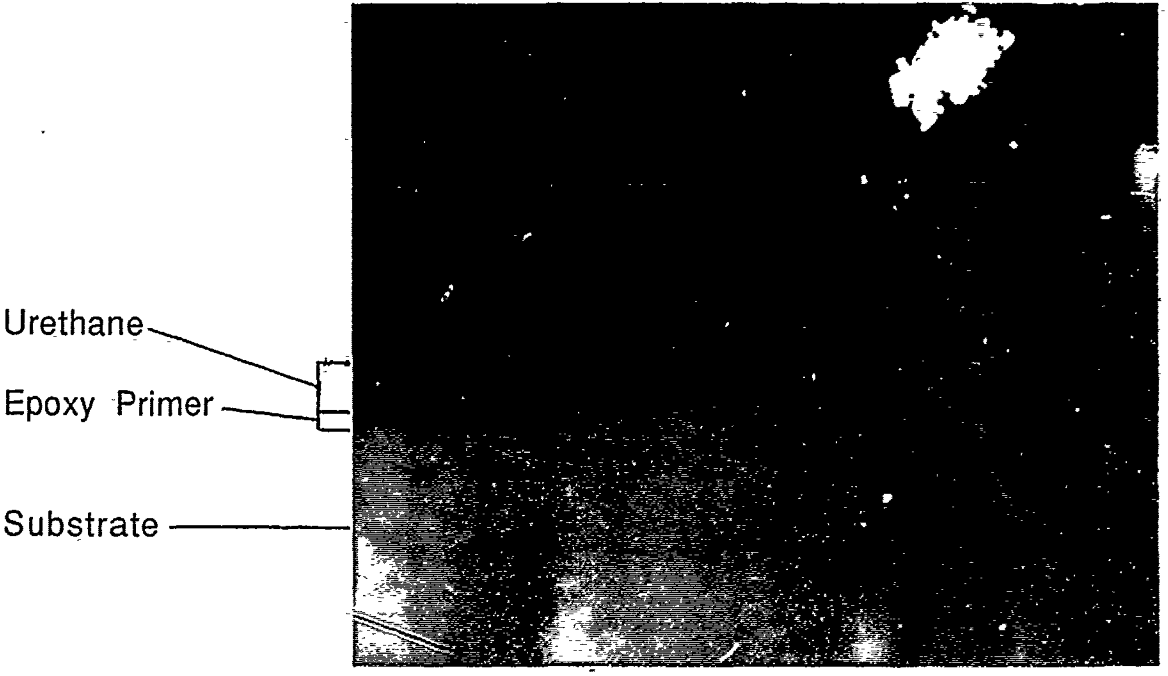


Figure 22. Variation in coating thickness as shown at the bottom weld of the five-gallon container. Mag. 70X

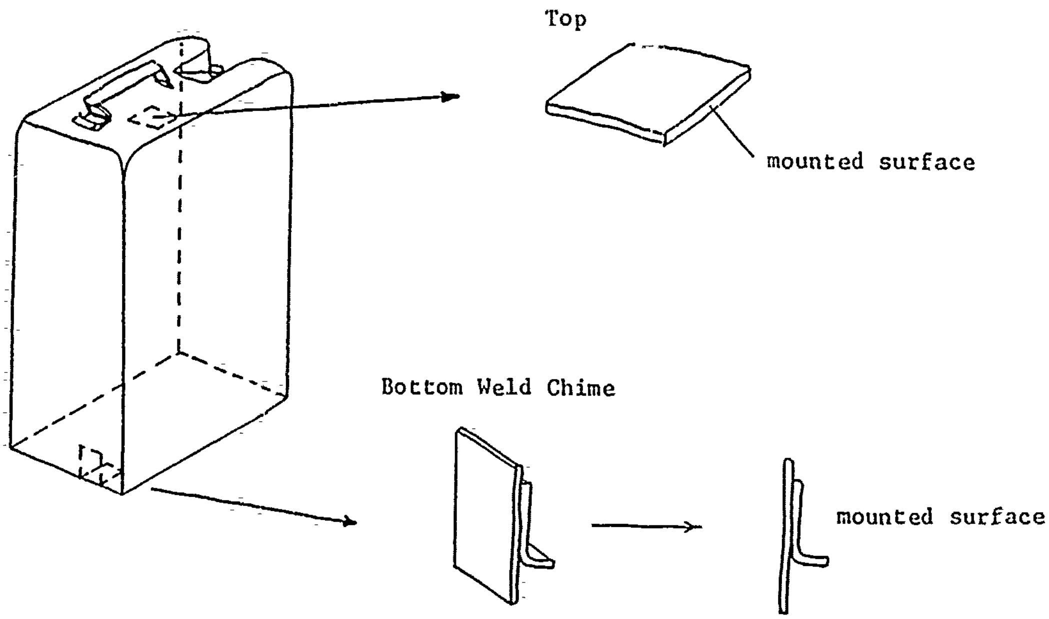


Figure 23. Metallographic cross sections of 14-liter container.

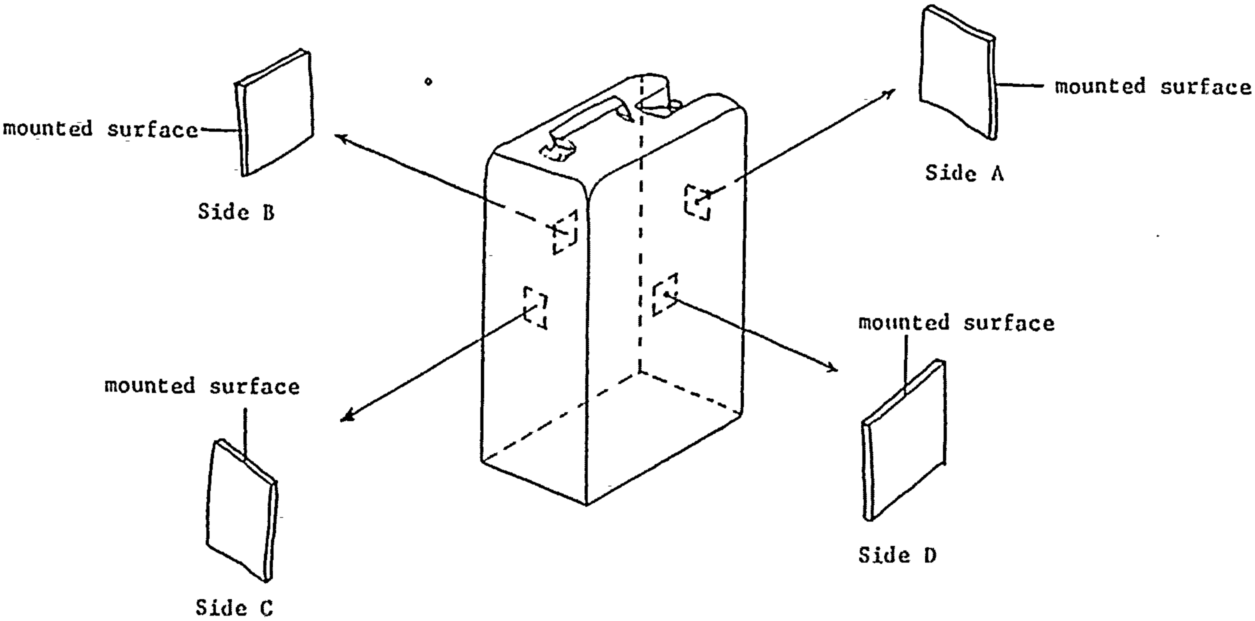


Figure 24. Metallographic cross sections of 14-liter container.

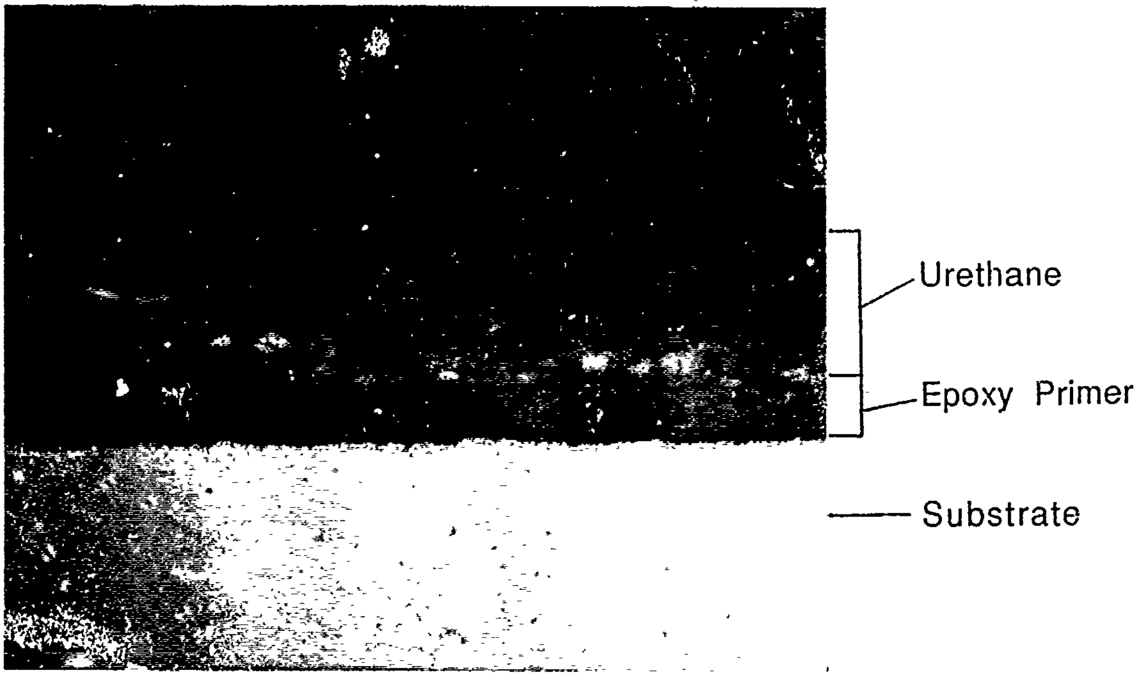


Figure 25. Representative cross section of the coating system of the 14-liter container taken from side B. Mag. 500X

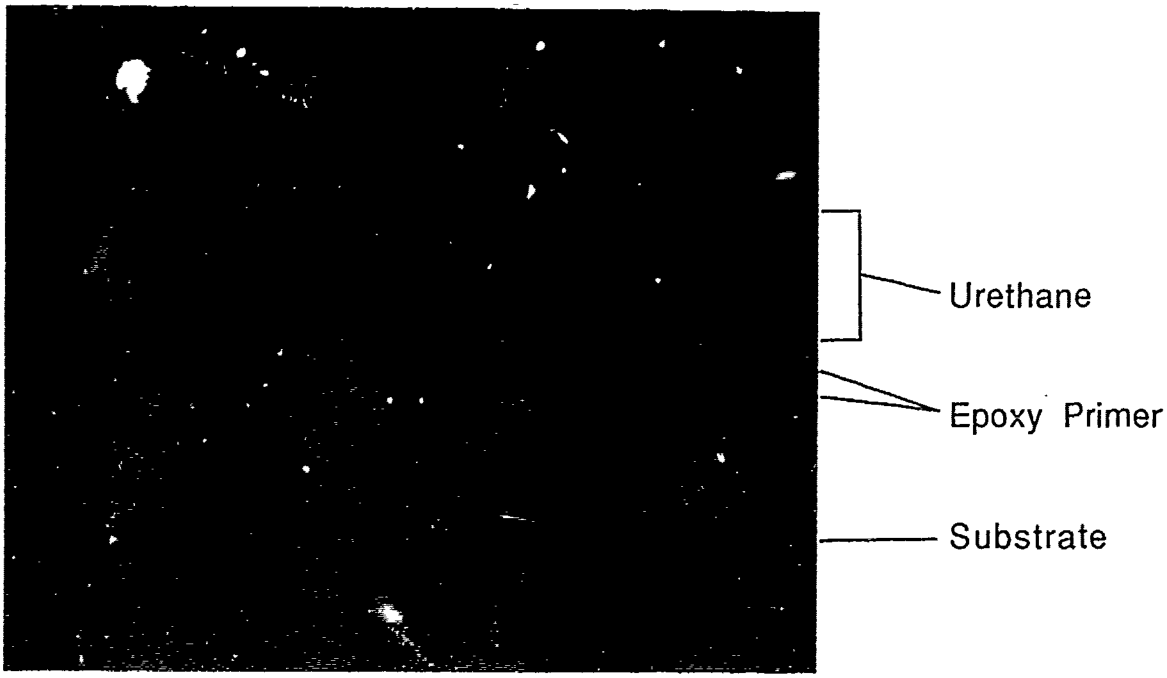


Figure 26. Nonuniform urethane and discontinuous epoxy primer observed at the top of the 14-liter container. Mag. 500X

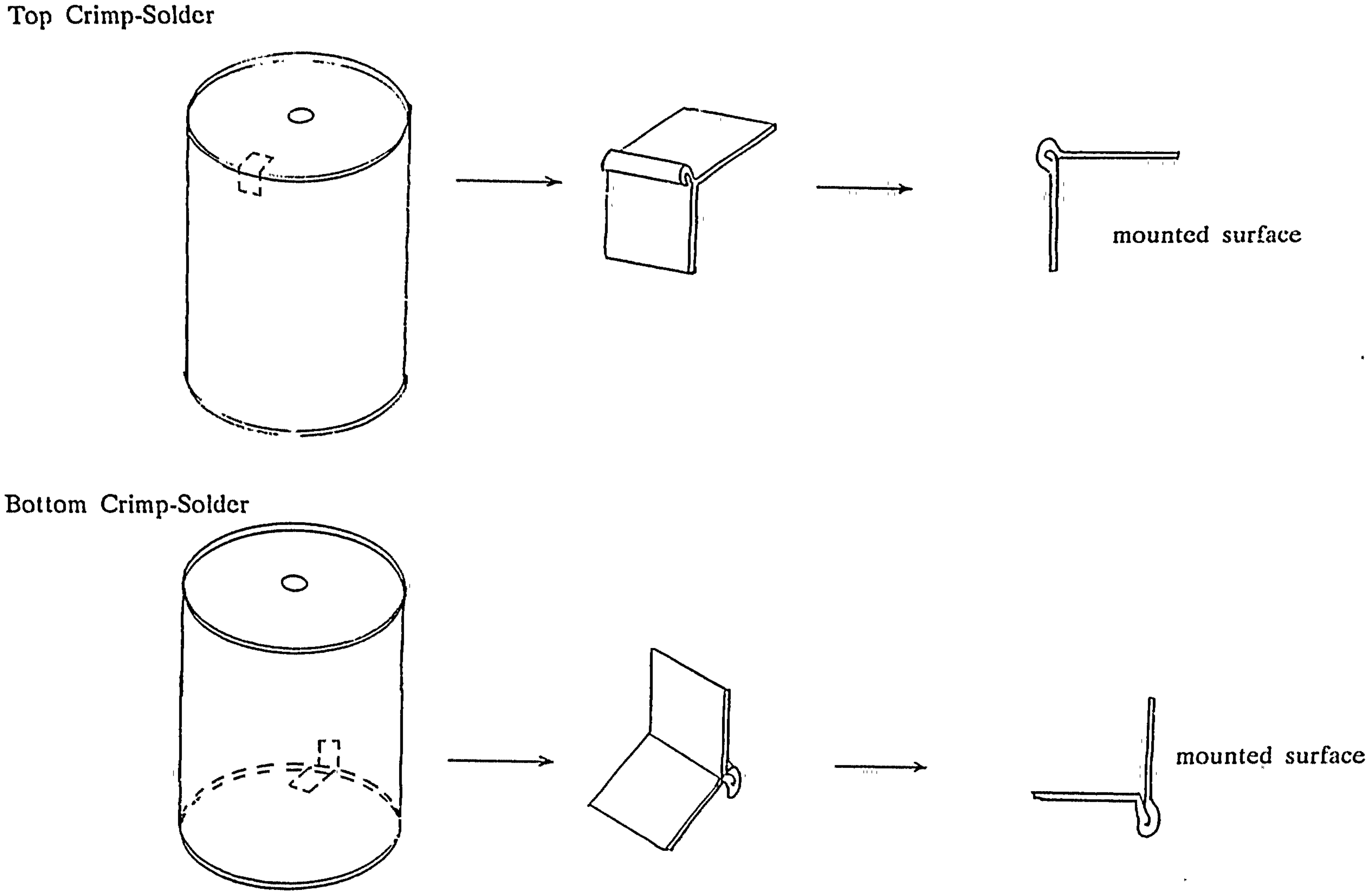


Figure 27. Metallographic cross sections of 1-1/3-quart container.

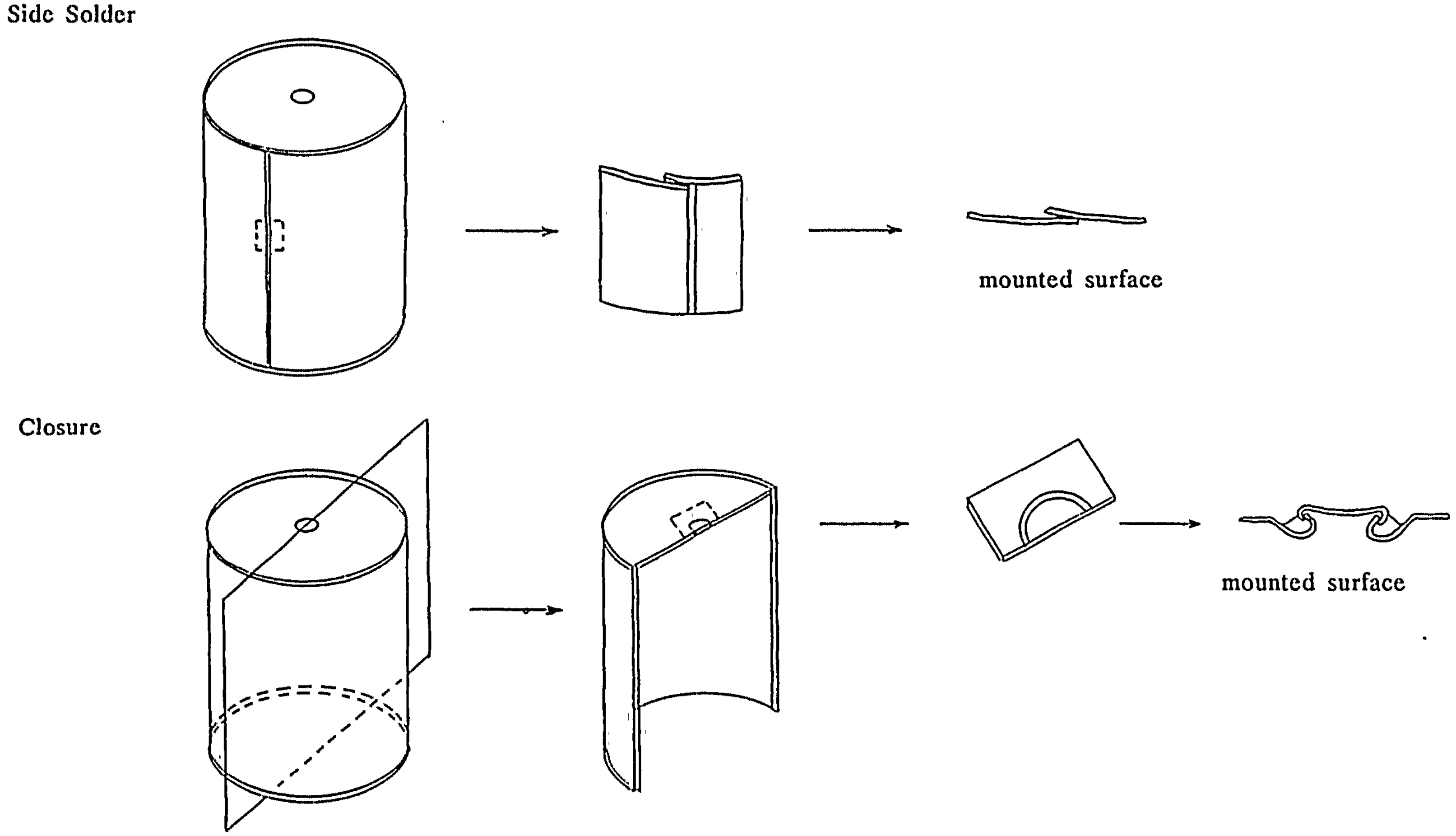


Figure 28. Metallographic cross sections of 1-1/3-quart container.

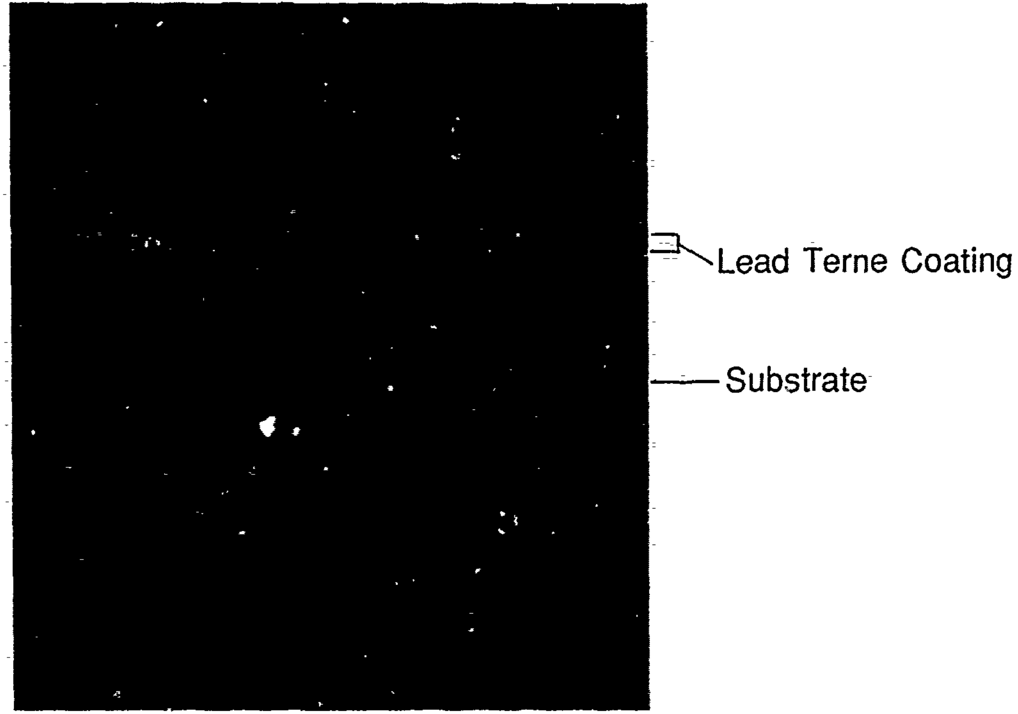


Figure 29. Representative cross section of the internal lead terne coating taken from the top weld of the 1-1/3-quart container. Mag. 500X

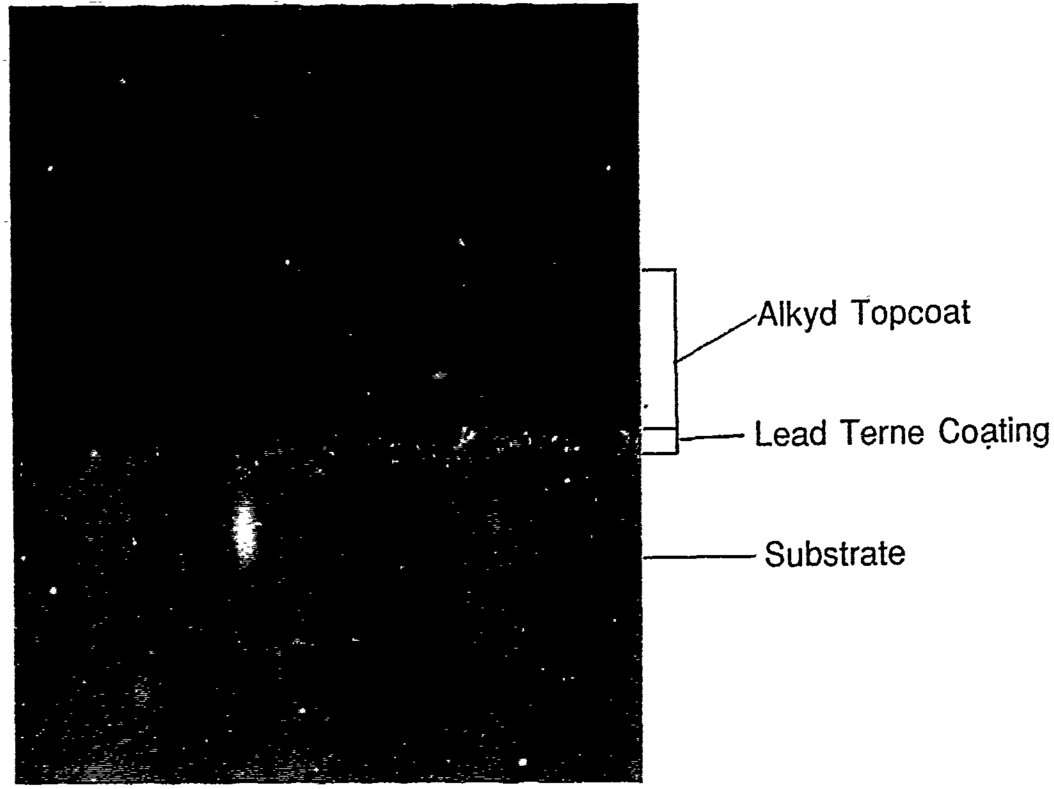


Figure 30. The lead terne coating and the alkyd topcoat taken at the bottom weld of the 1-1/3-quart container. Mag. 500X

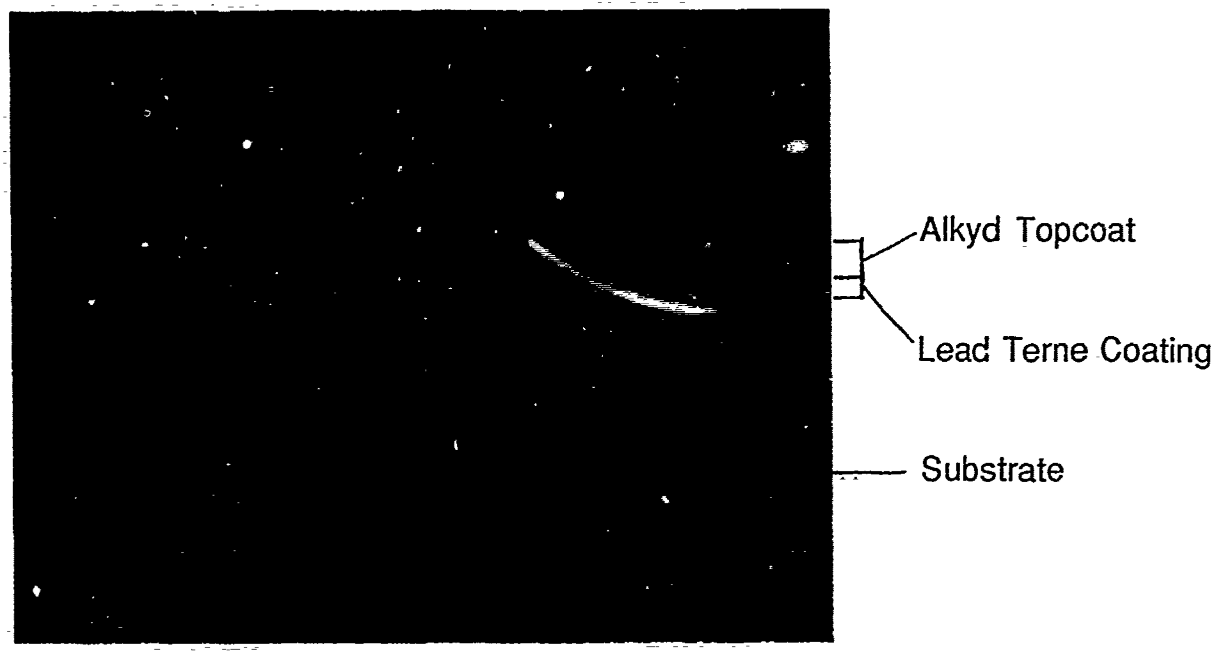


Figure 31. Variation in alkyd coating thickness near bottom weld of 1-1/3-quart container. Mag. 500X

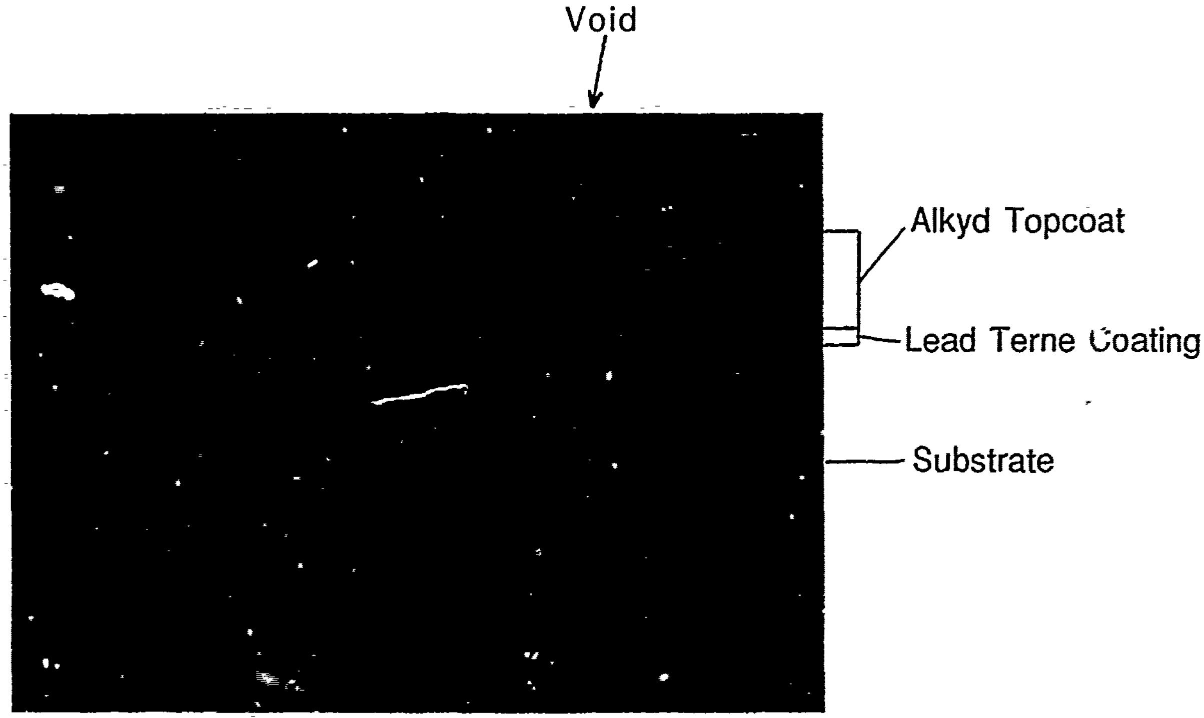


Figure 32. Void observed in the alkyd topcoat of the 1-1/3-quart container. Mag. 500X

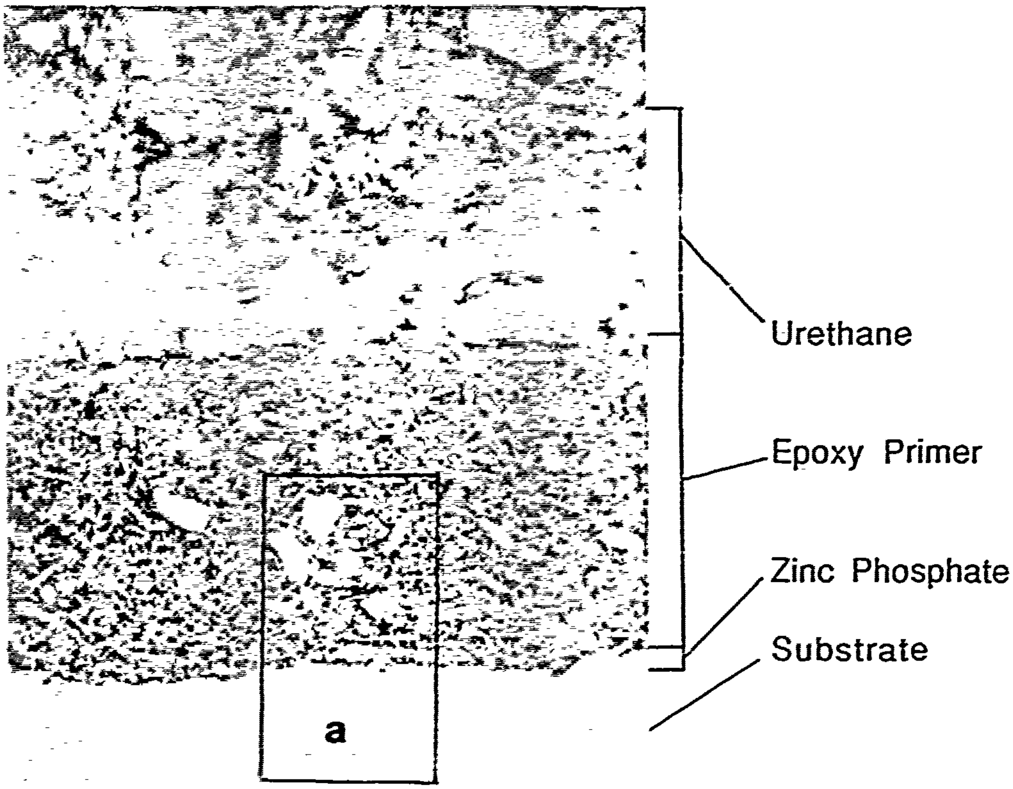


Figure 33. SEM micrograph of the five-gallon container coating system. Mag. 750X

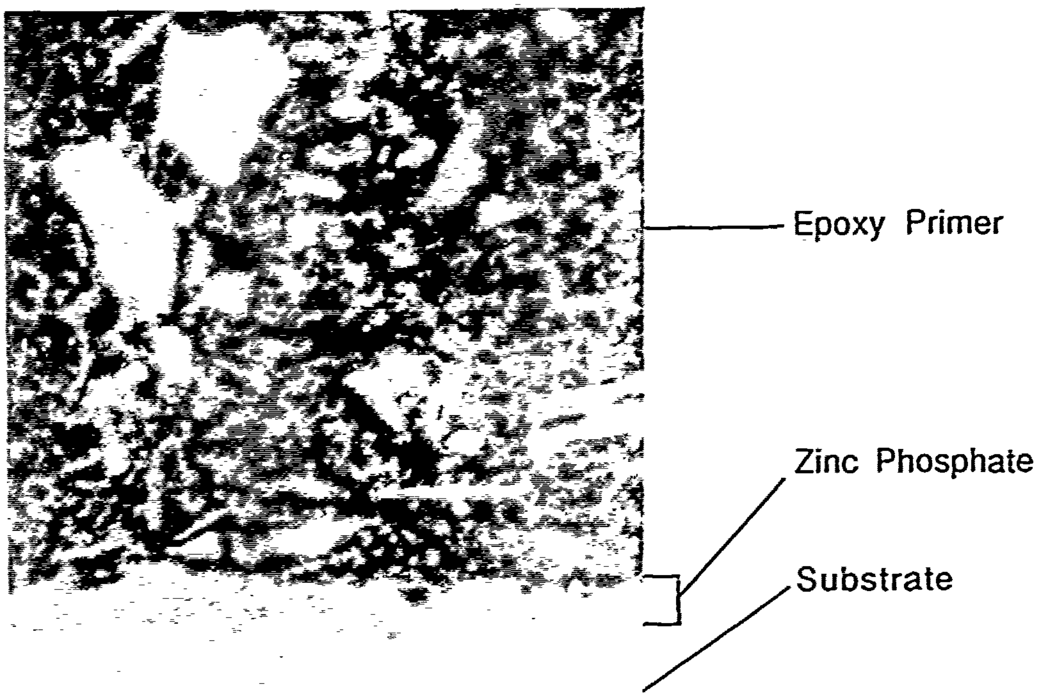


Figure 34. Zinc phosphate coating present on five-gallon container. Mag. 2500X

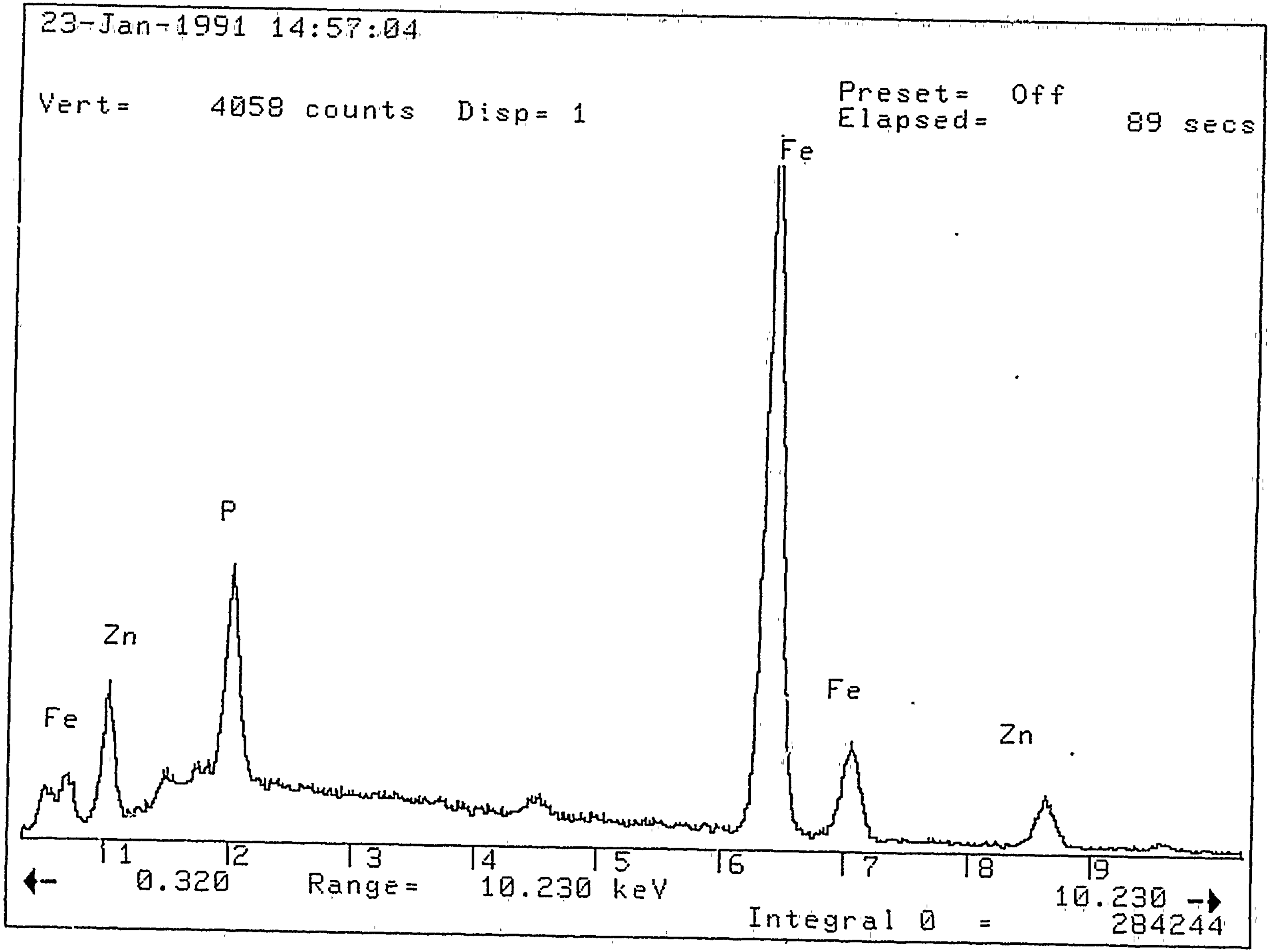


Figure 35. Energy Dispersive Spectroscopy of the exterior base metal of the five-gallon container. The zinc and phosphorous detected indicate the presence of a zinc phosphate pretreatment.

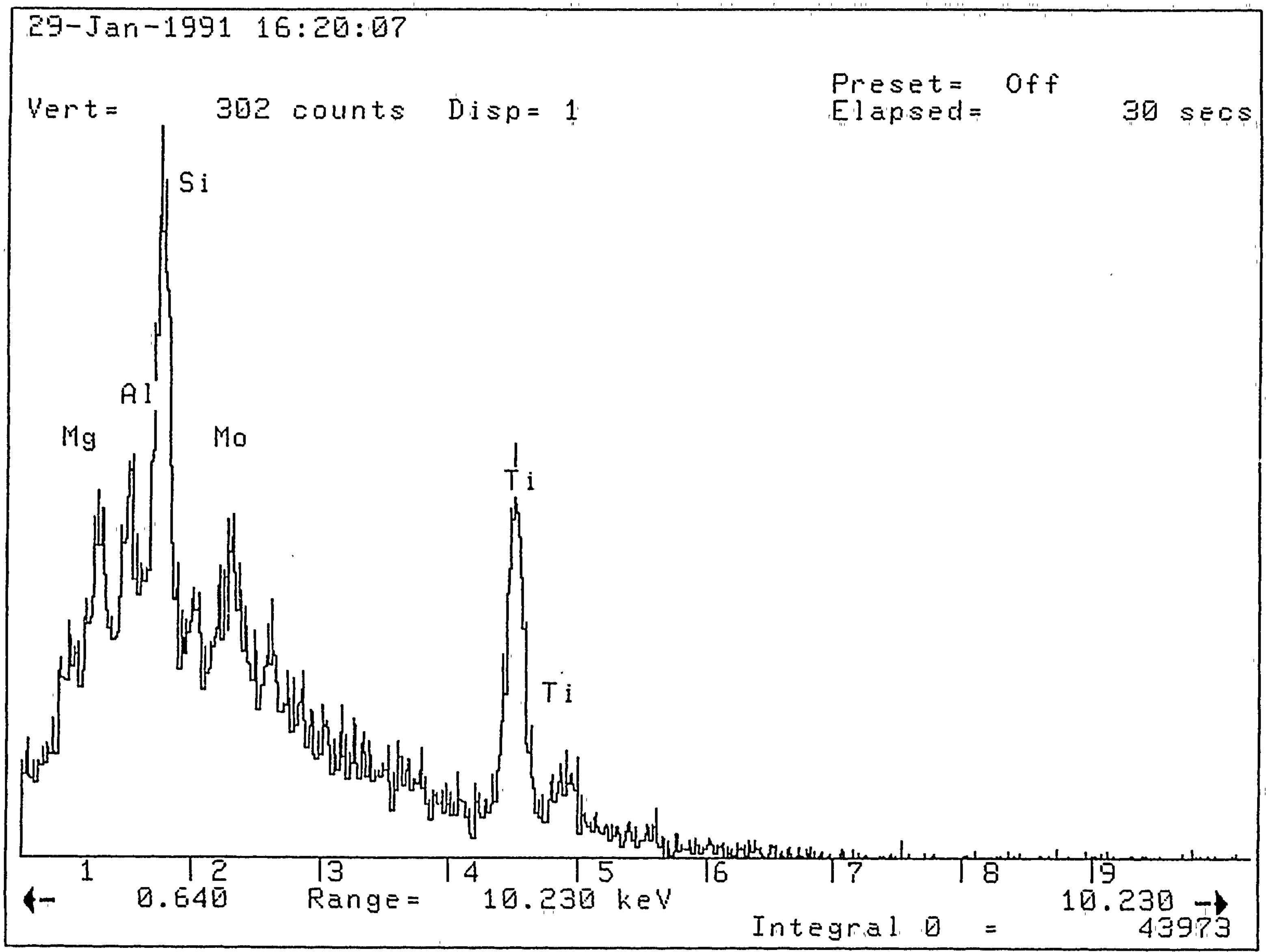


Figure 36. Energy dispersive spectroscopy of the epoxy primer of the five-gallon container. Silicon, titanium, aluminum, magnesium, and molybdenum are constituents of pigments added to the epoxy primer.

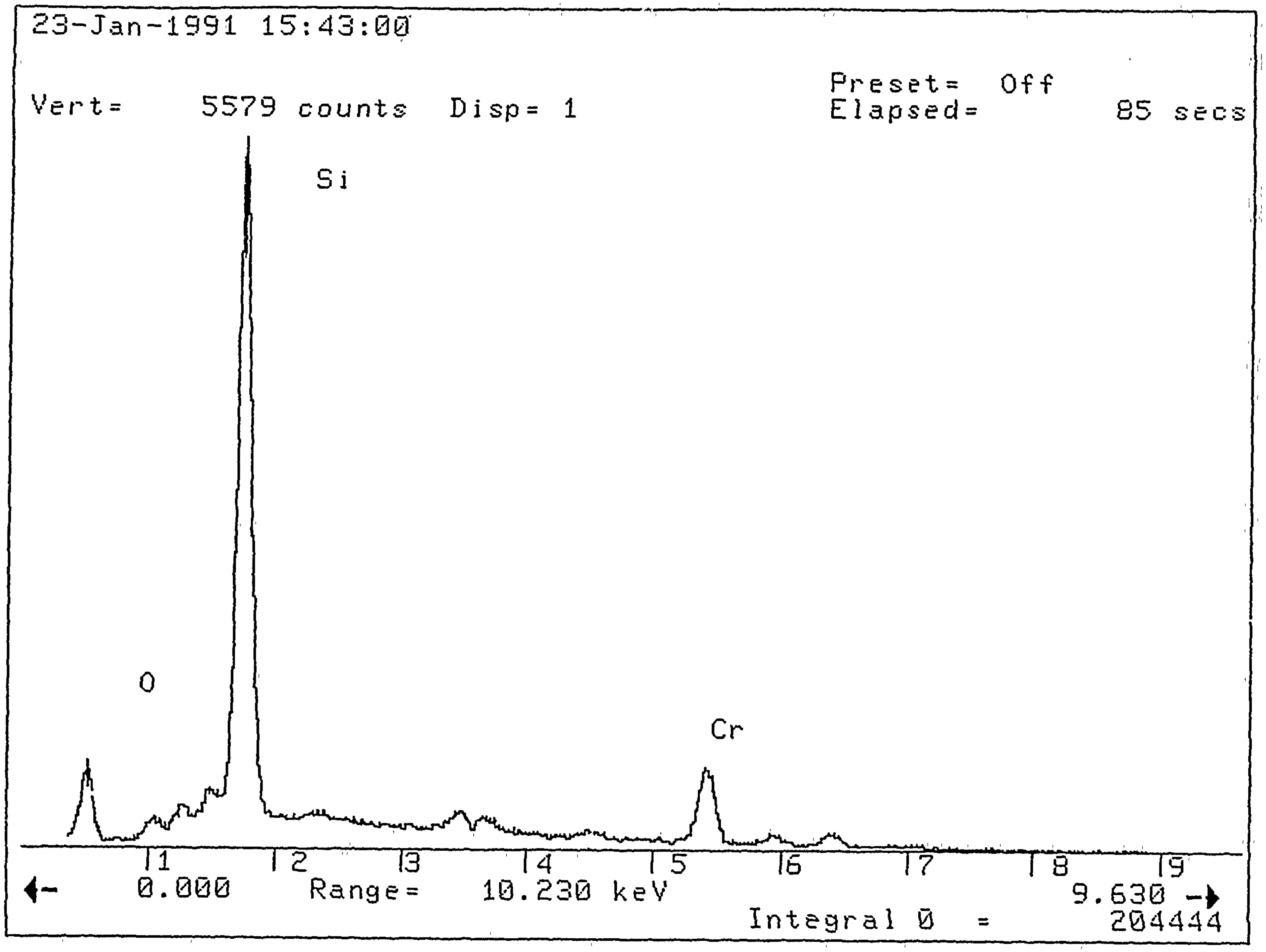


Figure 37. Energy dispersive spectroscopy of the topcoat of the five-gallon container. Silicon, chromium, and oxygen were detected. Each are constituents of pigments added to the topcoat.

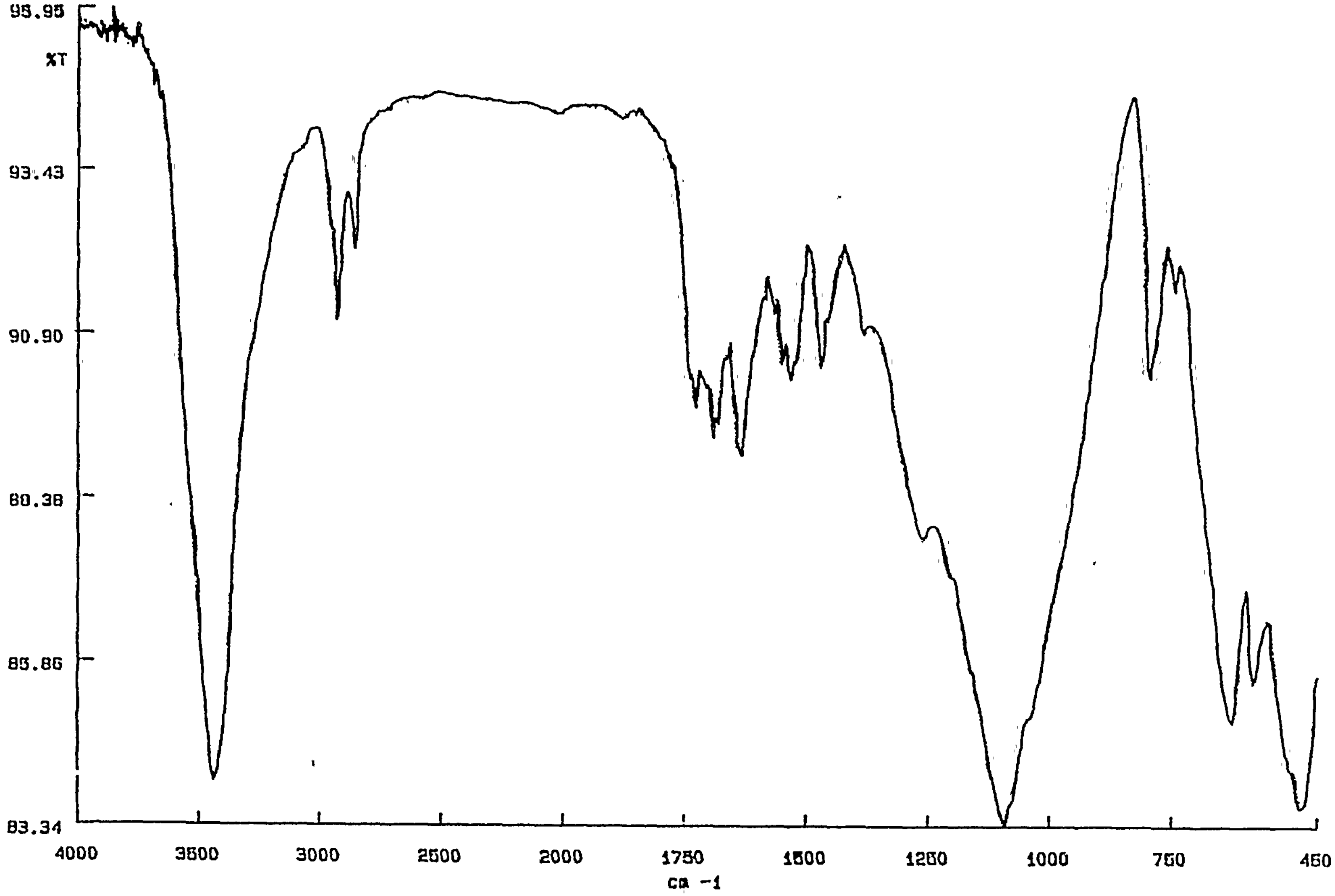


Figure 38. Infrared spectra collected from topcoat of five-gallon container indicative of urethane.

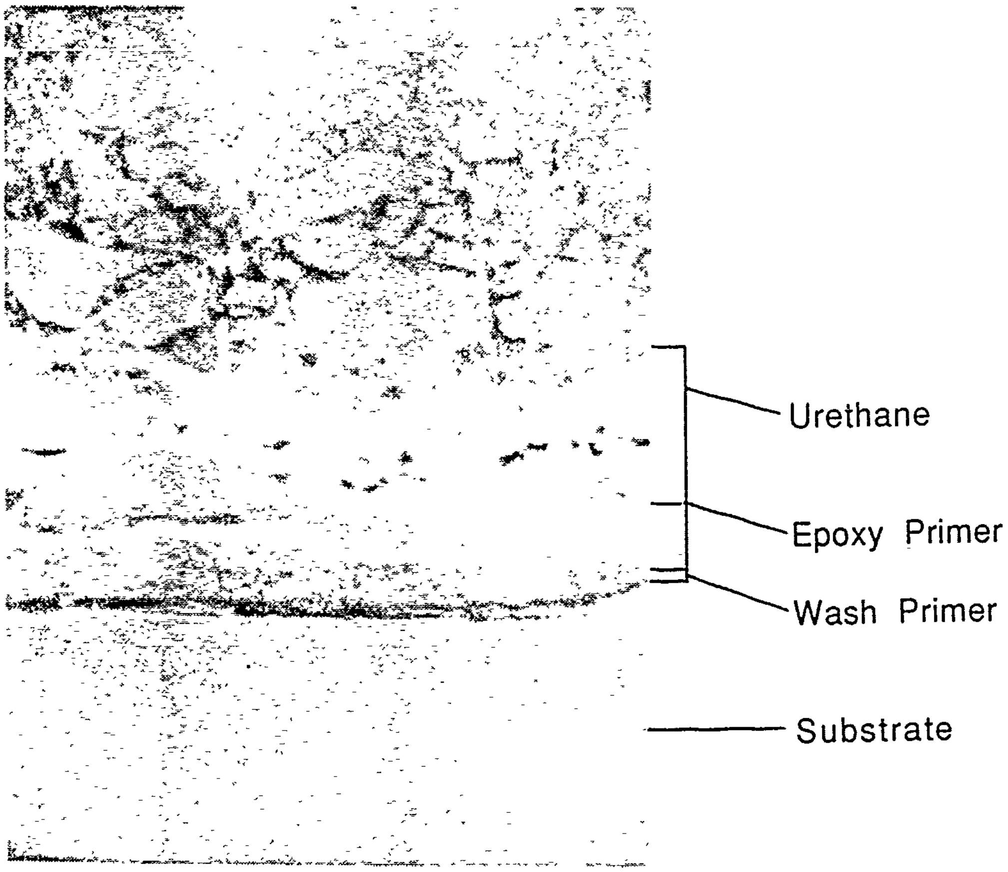


Figure 39. SEM micrograph of the coating system present on the 14-liter container. Mag. 1000X

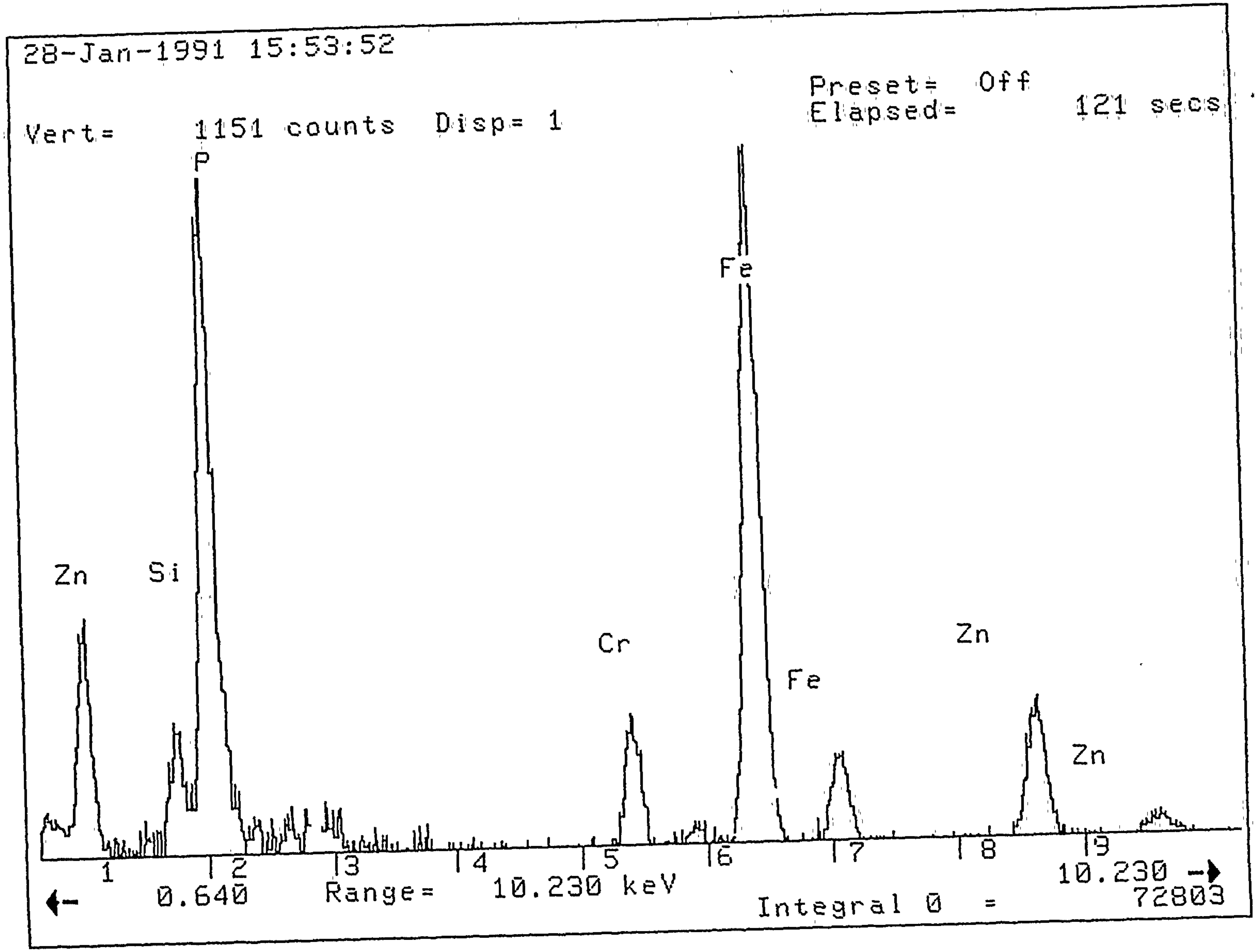


Figure 40. Energy dispersive spectroscopy of the base coating upon the exterior surface of the 14-liter container. The zinc, chromium, and phosphorous indicate the presence of a wash primer. The silicon is most likely evidence of the epoxy primer and the iron indicative of the underlying metal substrate.

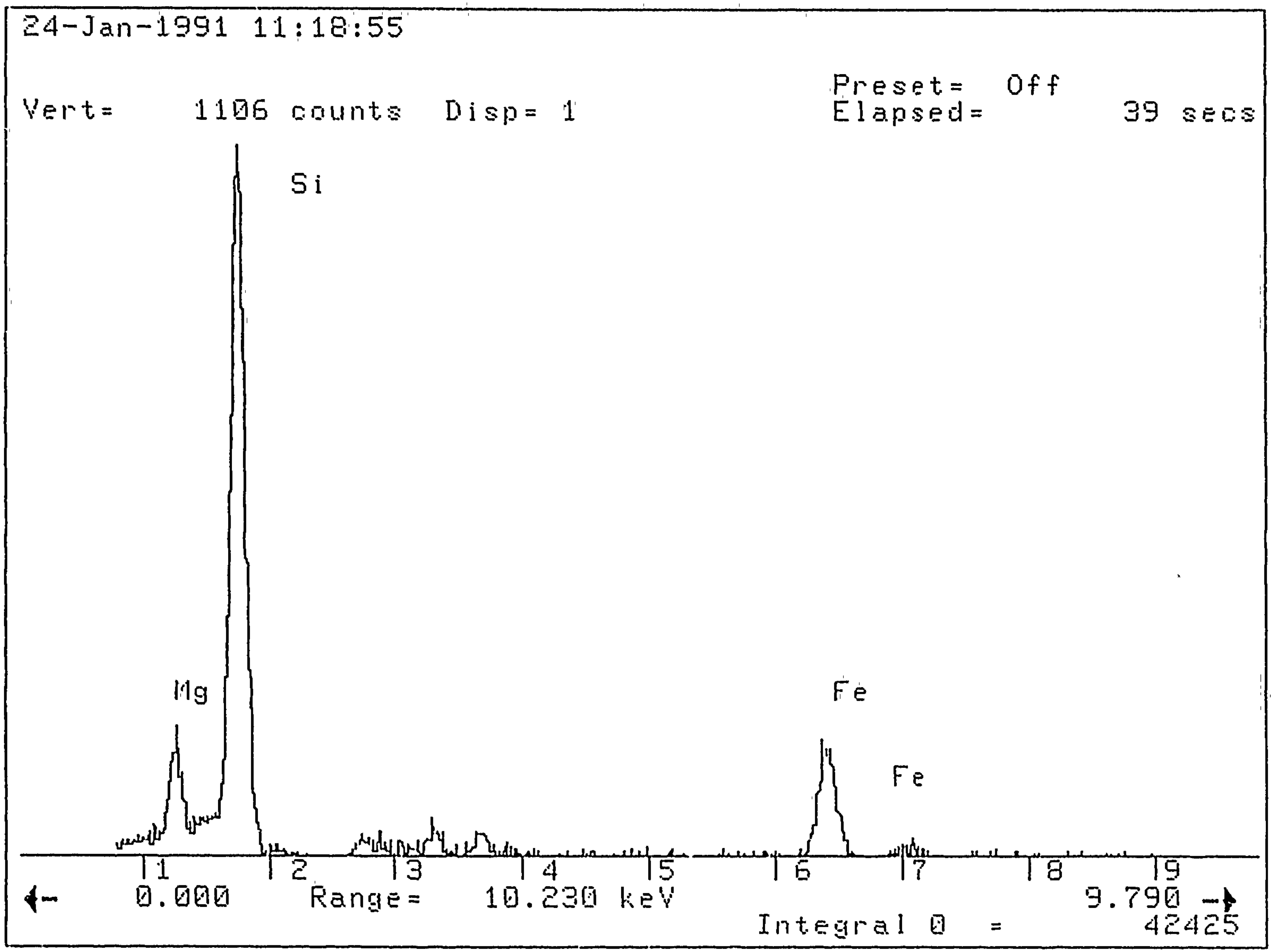


Figure 41. Energy dispersive spectroscopy of the epoxy primer on the exterior of the 14-liter container. Silicon, iron, and magnesium are all constituents of pigments added to the epoxy primer.

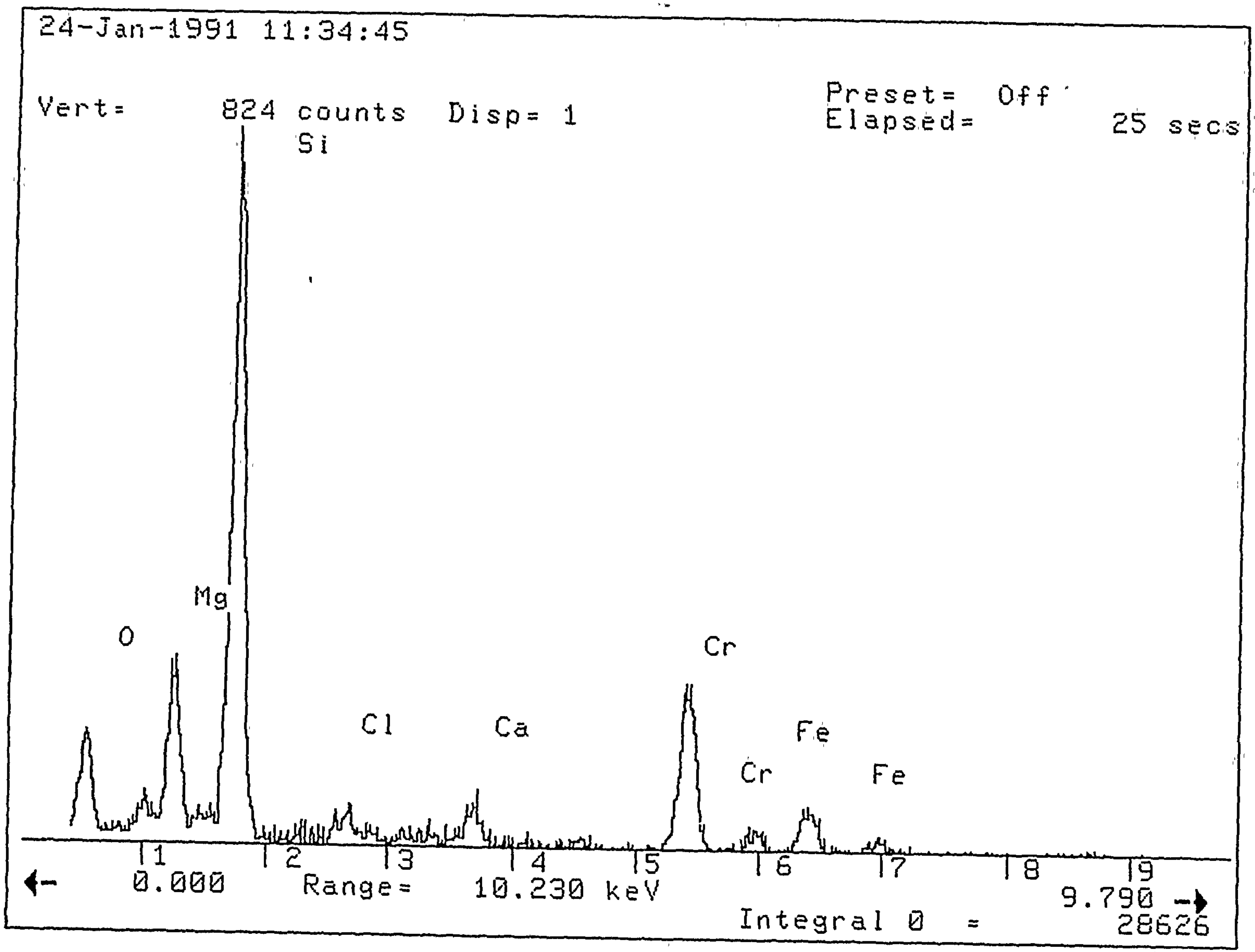


Figure 42. Energy dispersive spectroscopy of the *exterior topcoat* of the 14-liter container. Silicon, magnesium, oxygen, chromium, calcium, and iron are constituents of the pigments added to the topcoat. The presence of chlorine could indicate a contaminated sample surface.

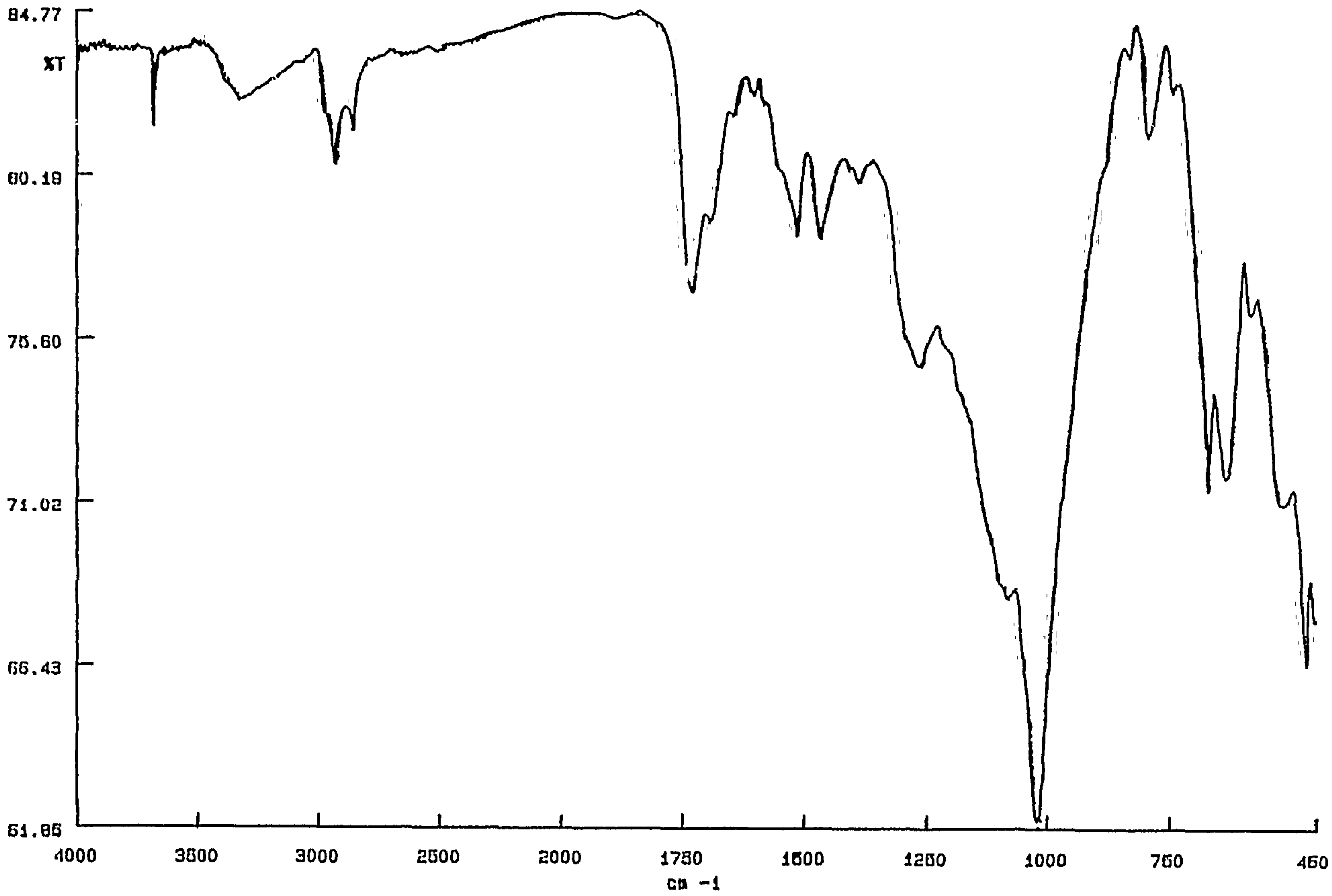


Figure 43. Infrared spectra collected from topcoat of 14-liter container indicative of polyurethane.

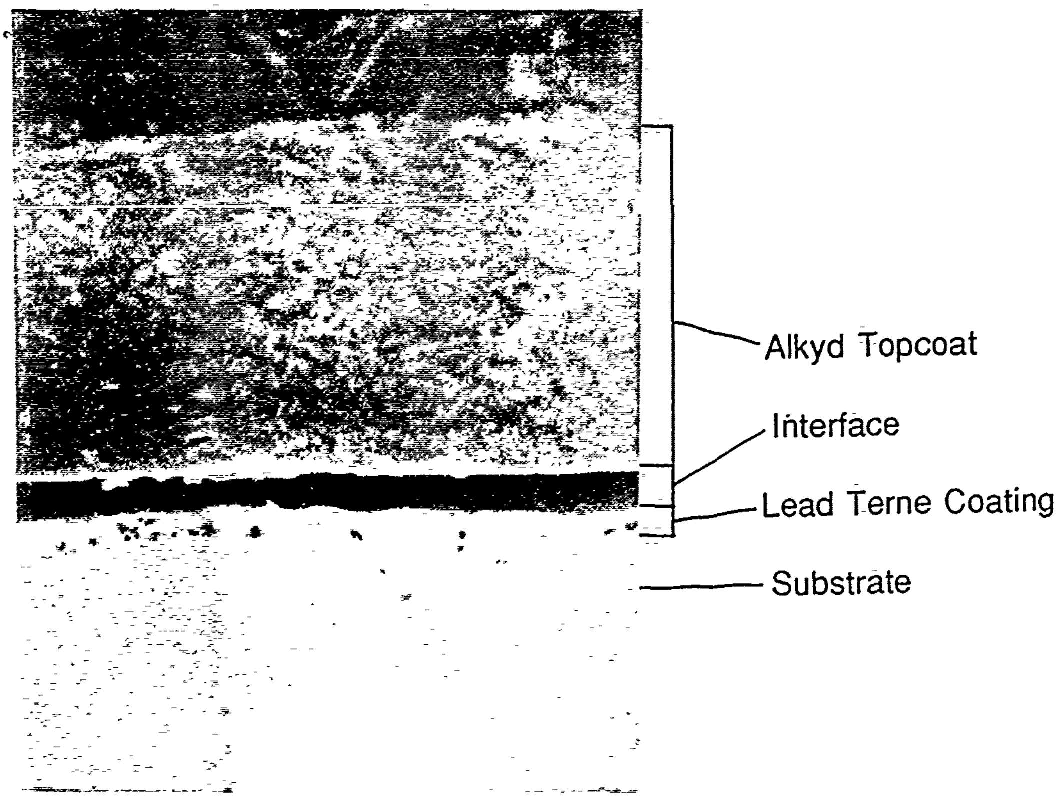


Figure 44. SEM micrograph of exterior coatings present on the 1-1/3 container. Mag. 750X

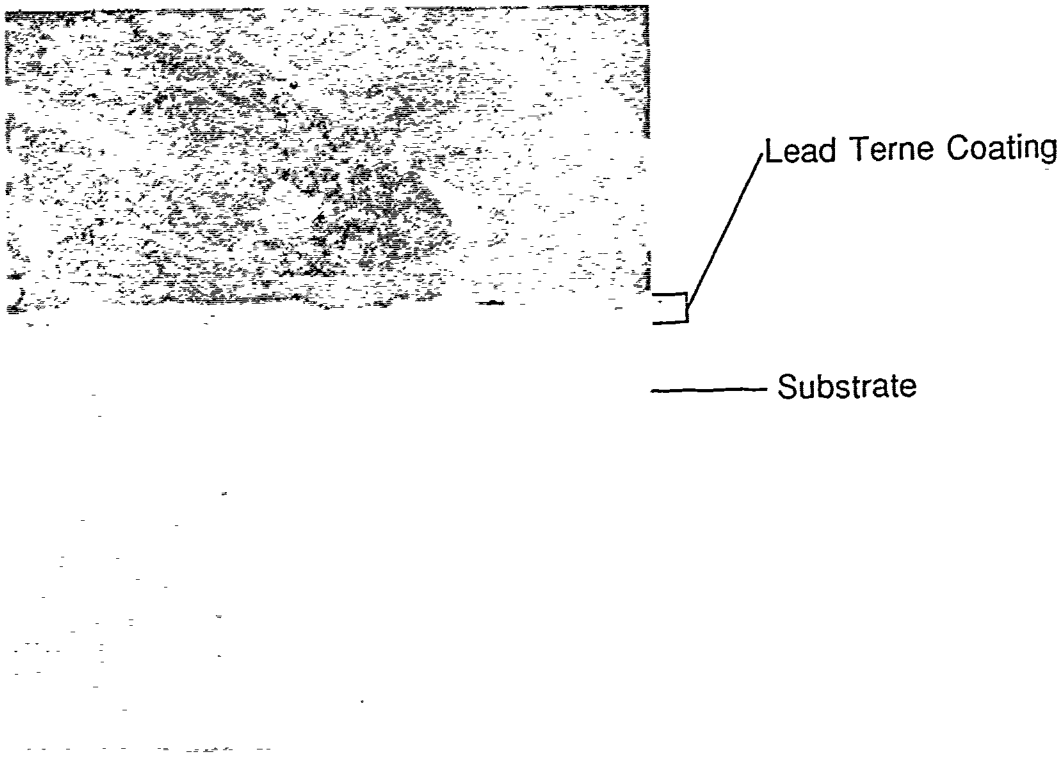


Figure 45. SEM micrograph of interior coating present on the 1-1/3 container. Mag. 750X

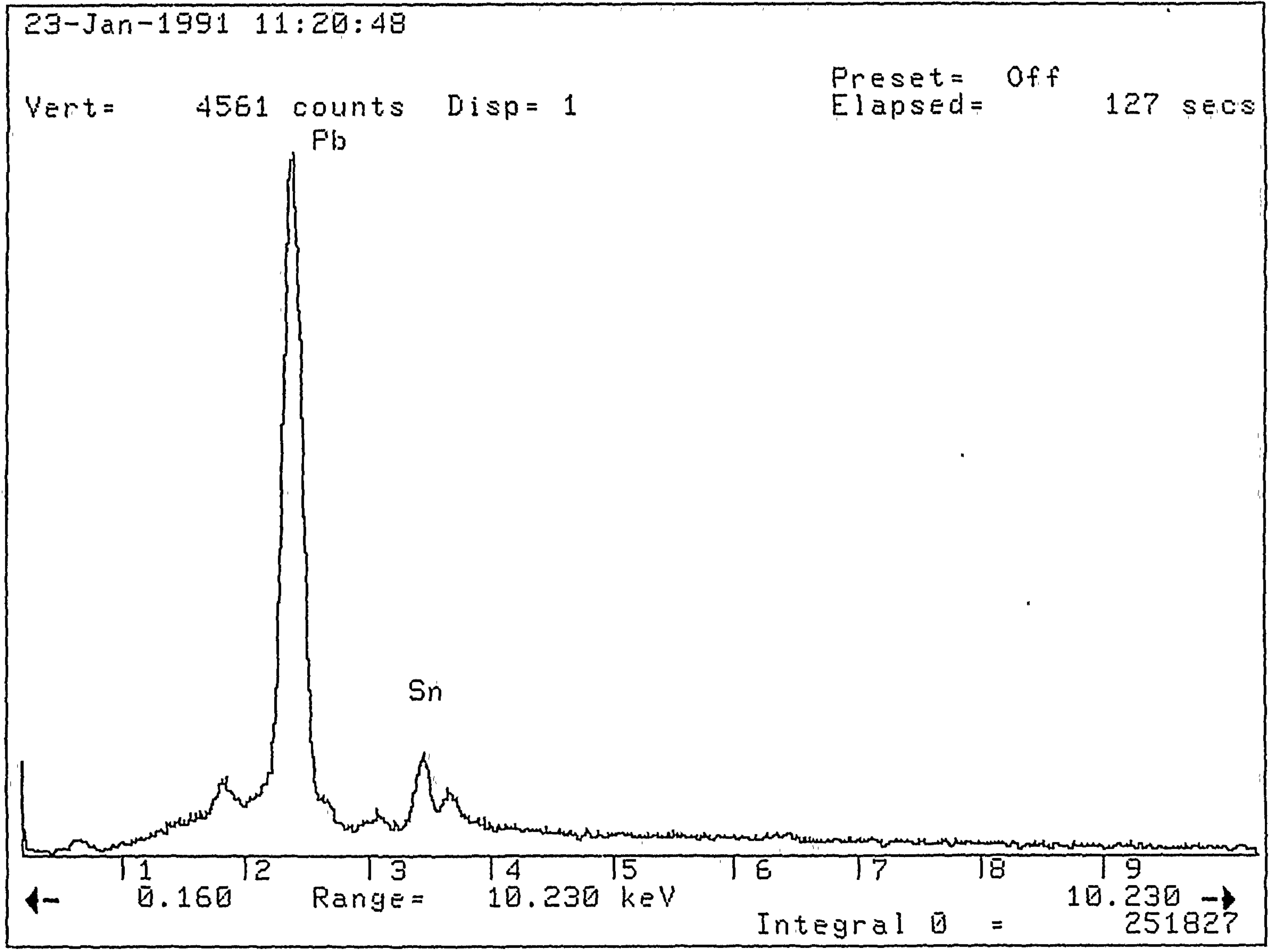
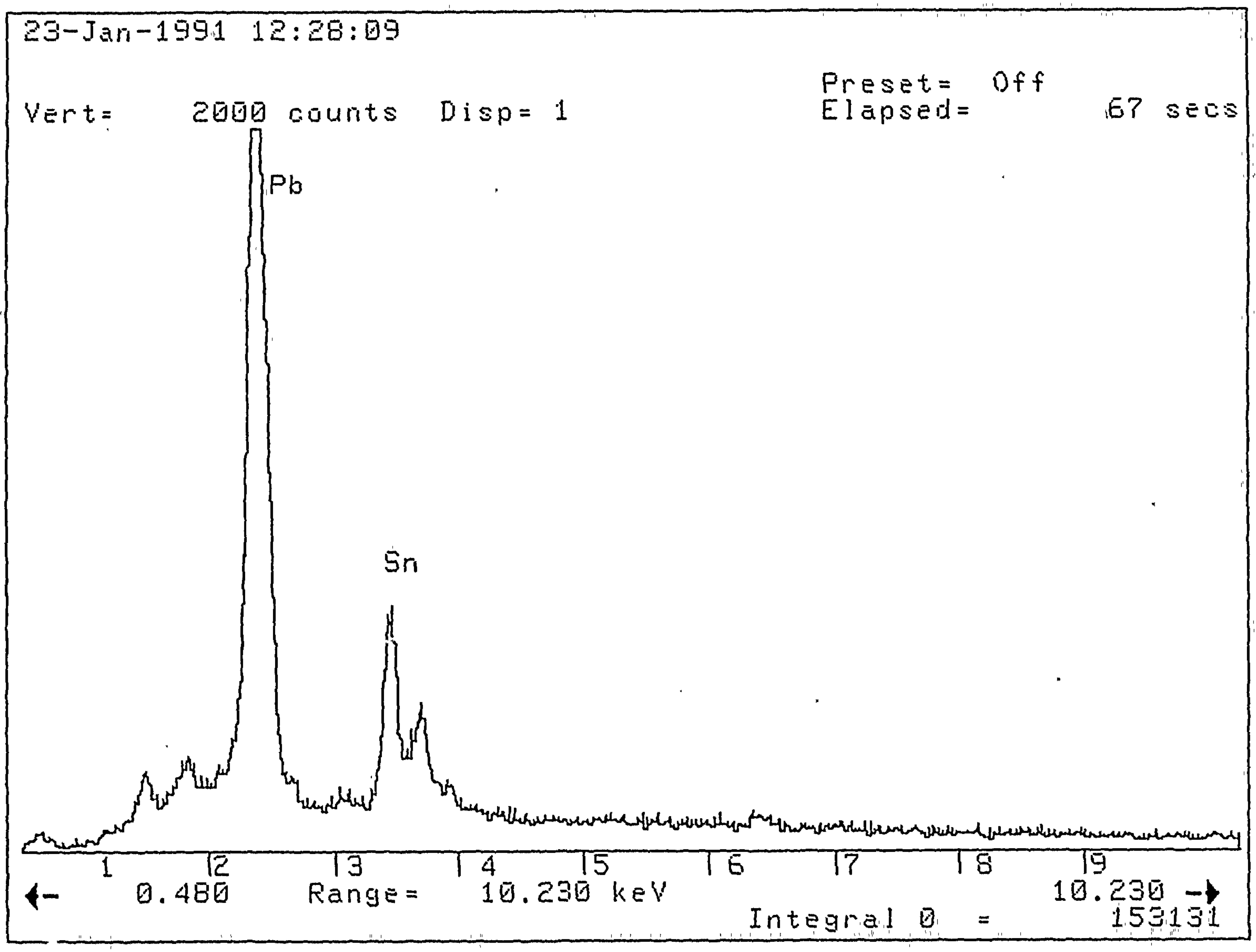


Figure 46. *Energy dispersive spectroscopy of the undercoat present on the exterior of the 1-1/3-quart container. . The main elemental constituents of lead and tin indicate a terne coating.*



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23-Jan-1991 12:28:09
                                              Preset=  Off
Vert=     2000 counts  Disp= 1                Elapsed=       67 secs

←-    0.480     Range=   10.230 keV                       10.230 -→
                                        Integral 0   =       153131
```

Figure 47. Energy dispersive spectroscopy of the inertior of the 1-1/3-quart container. The main elemental constituents of lead and tin indicate a terne coating.

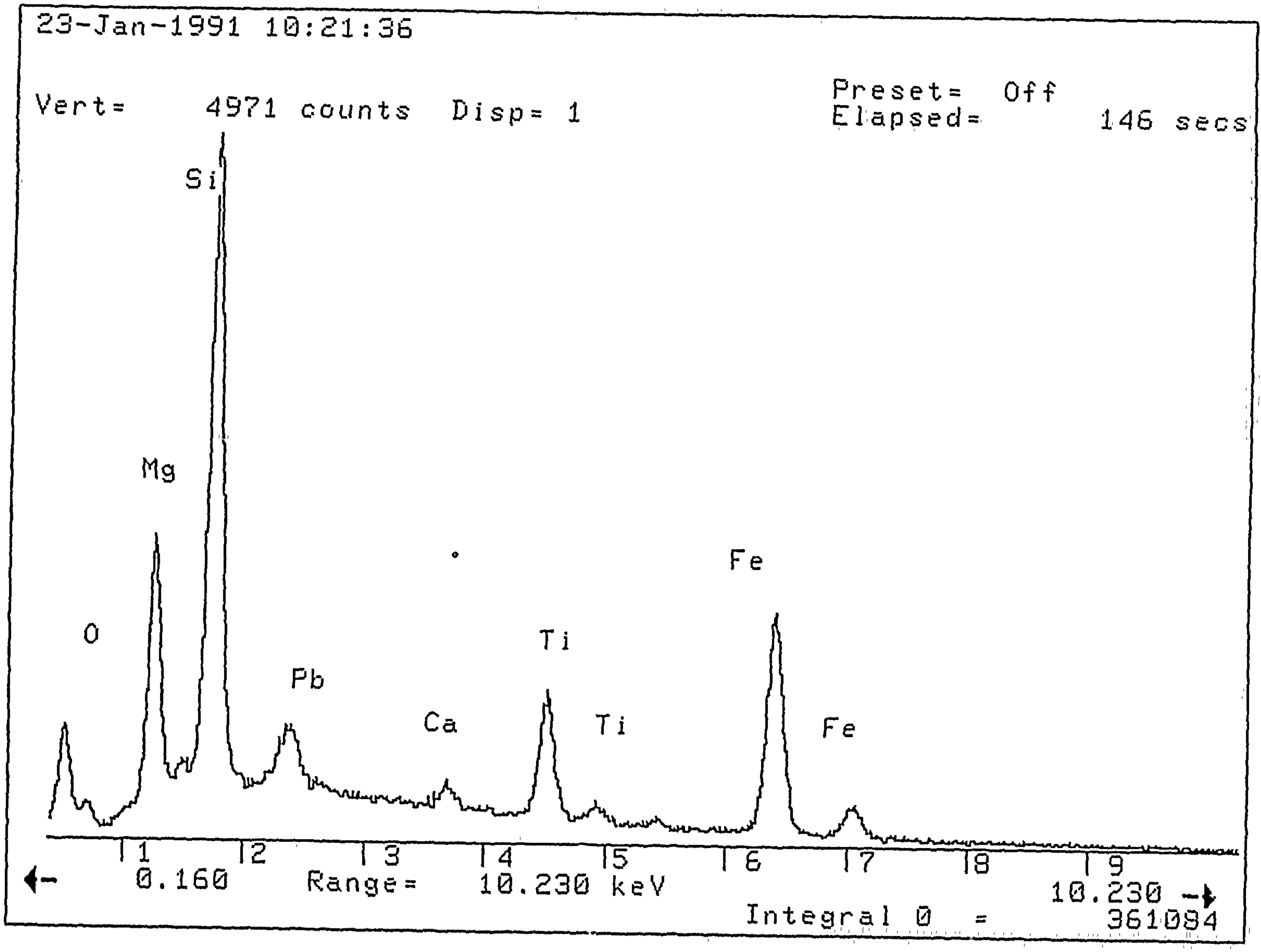

Figure 48. Energy dispersive spectroscopy of the alkyd coating present on the exterior of the 1-1/3-quart container. These elements are constituents of pigments added to the alkyd coating.

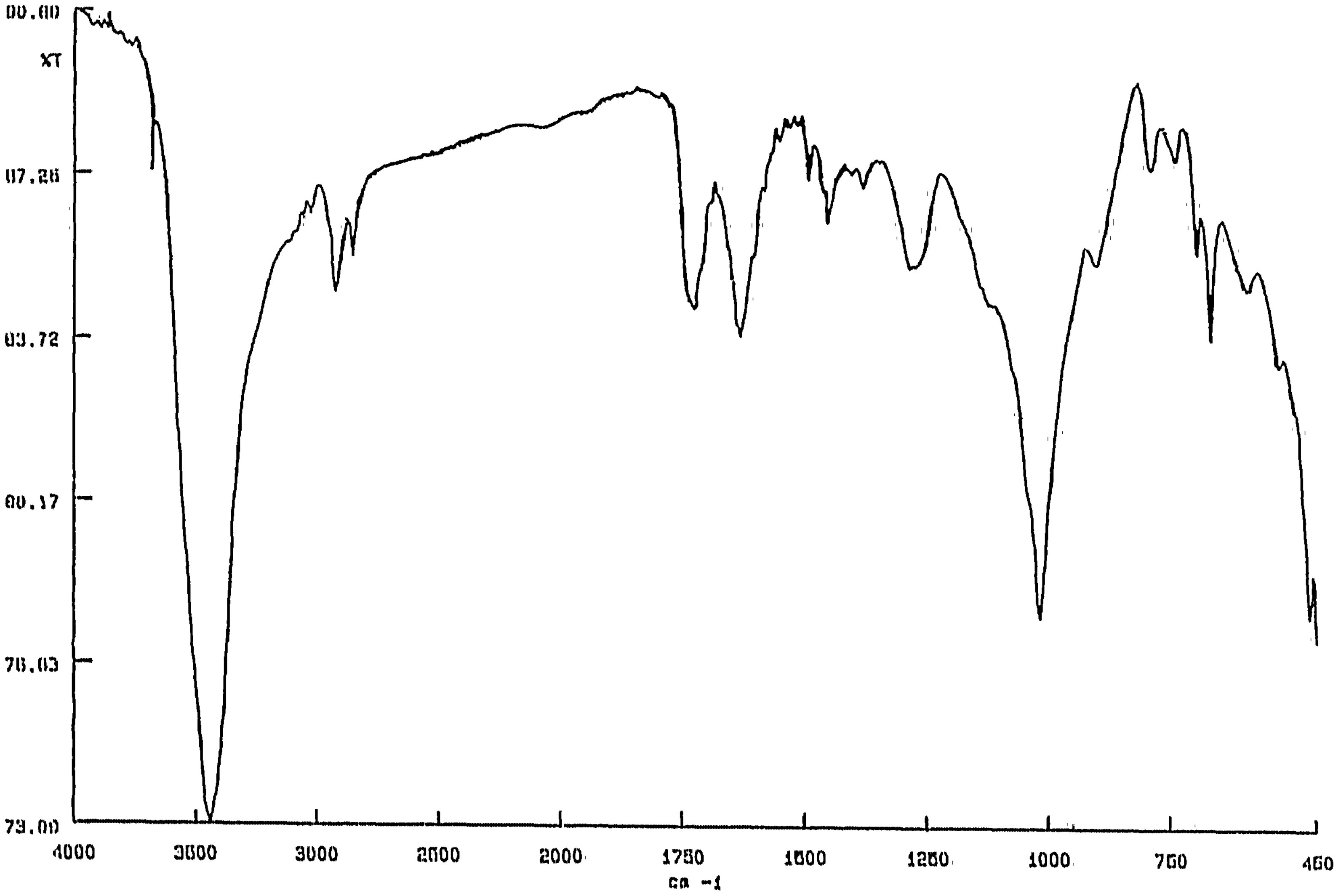

Figure 49. Infrared spectra collected from topcoat of 1-1/3-quart container indicative of an alkyd coating.

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ON ARMY DS2 CONTAINERS-
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Catherine M. Zoller, and Saul Isserow

Technical Report MTL TR 91-24, June 1991, 67 pp-
illus-tables

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The Chemical Research, Development & Engineering Center (CRDEC) requested the U.S. Army Materials Technology Laboratory (MTL) to characterize and evaluate the integrity of protective coatings used on DS2 containers and isolate the origin of the corrosion observed on the containers. Three different size containers were examined. It was determined by optical microscopy and visual examination that the corrosion was initiated from exterior surfaces, particularly in recesses, weld seams, and other areas which trapped moisture. Optical and electron mircroscopy was performed on cross-sectional specimens of the coating. Inconsistent phosphate coating coverage was revealed on the five-gallon container and the urethane topcoat thickness was below specified requirements. The epoxy primer was thicker than specified. The wash primer, epoxy primer, and topcoat thicknesses of the 14-liter container did not meet the specification. The exterior terne coating on the 1-1/3-quart container exhibited corrosion and blistering in many areas. Several recommendations were made: design flaws conducive to corrosion should be eliminated, alternative materials (e.g. plastics, stainless steels, and aluminum) should be considered, and more stringent quality control of coating application, as well as the handlingn of coated containers, should be enacted. Finally, if the present coating system and material for the container are not changed, then periodic inspections should be performed to detect corrosion in the early stages.

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