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RESIDUAL VCM STABILITY IN SUSPENSION VINYL RESINS

BOTTLE VS. BAG STORAGE

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Vinyl resins produced at Texas City have routinely been sampled in polyethylene bags in the production and distribution areas and sent to the laboratory for specification and special analyses, including the analysis for residual vinyl chloride. With respect to the VCM analysis, a resin sample, upon receipt in the laboratory, is placed in a small, two ounce bottle and tightly capped to minimize the loss of VCM prior to actual dissolution of the resin in tetrahydrofuran. Recently, a recommended procedure for the "Sampling and Storage of Test Samples of Vinyl Resins for Residual VCM Analysis" was proposed by Dr. John Brezinski.¹ This procedure is at variance from the established plant procedure in that the proposed sampling of the resin is initially in 4 oz, wide-mouth glass jars which are tightly sealed with an aluminum-lined lid and electrical tape applied to the juncture of the jar and lid. Analysis of the resin is recommended to be within four hours and in no case later than twenty-four hours after sampling.

Accordingly a comparative evaluation of the recommended and current sampling procedures with respect to residual vinyl chloride in suspension resins has been made. Spot samples of several resin types were obtained from the dryers. Transfer of the samples into 4 oz, wide-mouth bottles as well as polyethylene bags was quickly made. The 4 oz bottles were tightly capped and sealed with electrical tape. The polyethylene bags were tightly knotted leaving essentially no vapor space above the resin. Samples were then withdrawn from both bottle and bag after 0, 4, 24 and 48 hours storage at room temperature and analyzed for residual VCM using the current analytical procedure, WC-326-G-1. Results of this comparative evaluation are shown in Table I.

The storage stability of suspension vinyl resins with respect to residual VCM is much greater in tightly-sealed sample bottles than in polyethylene bags. Unless resin samples taken for residual VCM analysis are initially stored in tightly-sealed bottles, the loss of VCM from the resin is virtually unchecked until resin dissolution. A graphical comparison of residual vinyl chloride analysis with storage time for each suspension resin evaluated is shown in Graphs I - III.

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The data further indicates that an average VCM loss of approximately 20% occurs in samples stored in polyethylene bags compared to 5% in bottles after a storage time of twenty-four hours (Figure IV). Vinyl resin samples are generally analyzed for residual VCM within twenty-four hours after sampling.

RECOMMENDATIONS

The routine sampling of vinyl resins by production and distribution personnel in small sample bottles for determining residual VCM would represent a drastic change to present sampling techniques particularly for shipping samples which generally are composites of 3-8 samples. The following proposed changes to current sampling procedures are offered:

1. Minimal delay in sample pick-up, compositing and storage in sample bottles is given to resin samples which are to be analyzed for residual VCM.
2. Resin samples which are specifically required for process studies involving residual VCM are initially taken in small sample bottles which are tightly capped and sealed with electrical tape.
3. The < 1 ppm VCM specification on all solvent vinyl (dry) resins has necessitated the sampling and analysis for VCM of the storage bin (after blending and prior to transfer into the final shipping container). Generally, solvent resins analyze <0.5 ppm VCM. However, in the event a solvent vinyl bin analyzed >0.8 ppm, it is recommended that the bin be resampled and initially placed in a sample bottle until reanalysis is made. The reanalysis would eliminate possible VCM loss which might have occurred from normal sampling procedures.

REFERENCE

¹Brezinski, J. J., letter to R. H. Wheeler "Sampling of Vinyl Resins for Residual Vinyl Chloride Monomer Analysis," 511, April 17, 1975.

TABLE I

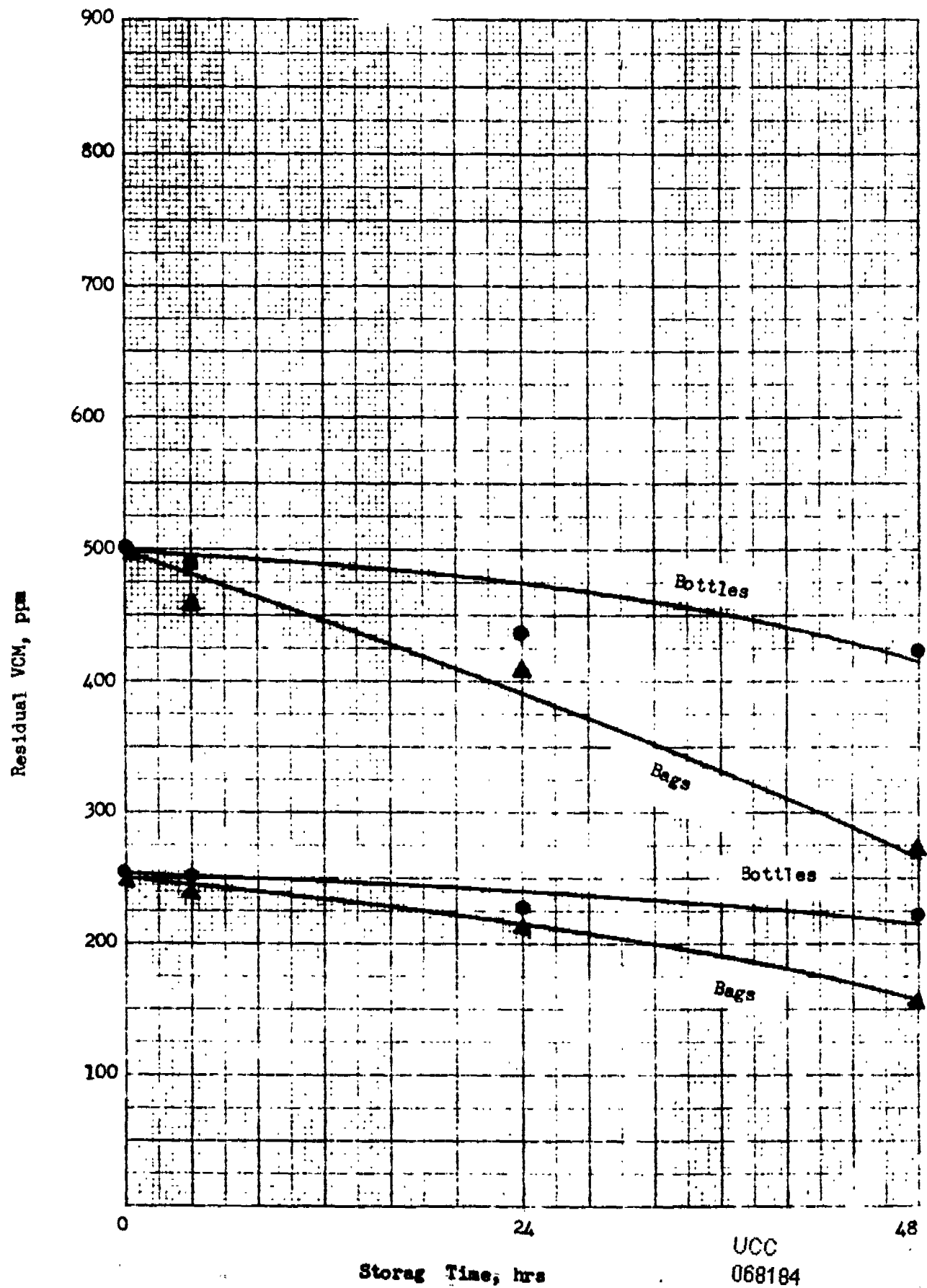
COMPARISON OF VCM STORAGE STABILITY OF SUSPENSION VINYL RESINS
BOTTLE VS. BAG SAMPLE CONTAINERS

<u>RESIN TYPE</u>	<u>VCM, ppm</u>			
<u>QSAL-7</u>	<u>No. 1</u>		<u>No. 2</u>	
	<u>Bottle</u>	<u>Bag</u>	<u>Bottle</u>	<u>Bag</u>
Initial	259	259	1049	1049
4-hours	259	227	1040	944
24-hours	250	207	1013	860
48-hours	247	130	913	769
 <u>QSQH-7</u>				
Initial	877	877	1262	1262
4-hours	853	838	1244	1104
24-hours	840	729	1240	886
48-hours	828	544	1221	654
 <u>VSJE-10</u>				
Initial	255	255	500	500
4-hours	255	242	490	461
24-hours	227	216	436	409
48-hours	224	158	426	271

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FIGURE I

Comparison of Residual VCM Stability of VSJE-10 Samples
Bottles versus Bags



14-21 10 X 10 TO 1/2 INCH 46 1432
1/2 INCH 46 1432
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FIGURE II

Comparison of Residual VCM Stability of QSAL-7 Samples
Bottles v rsus Bags

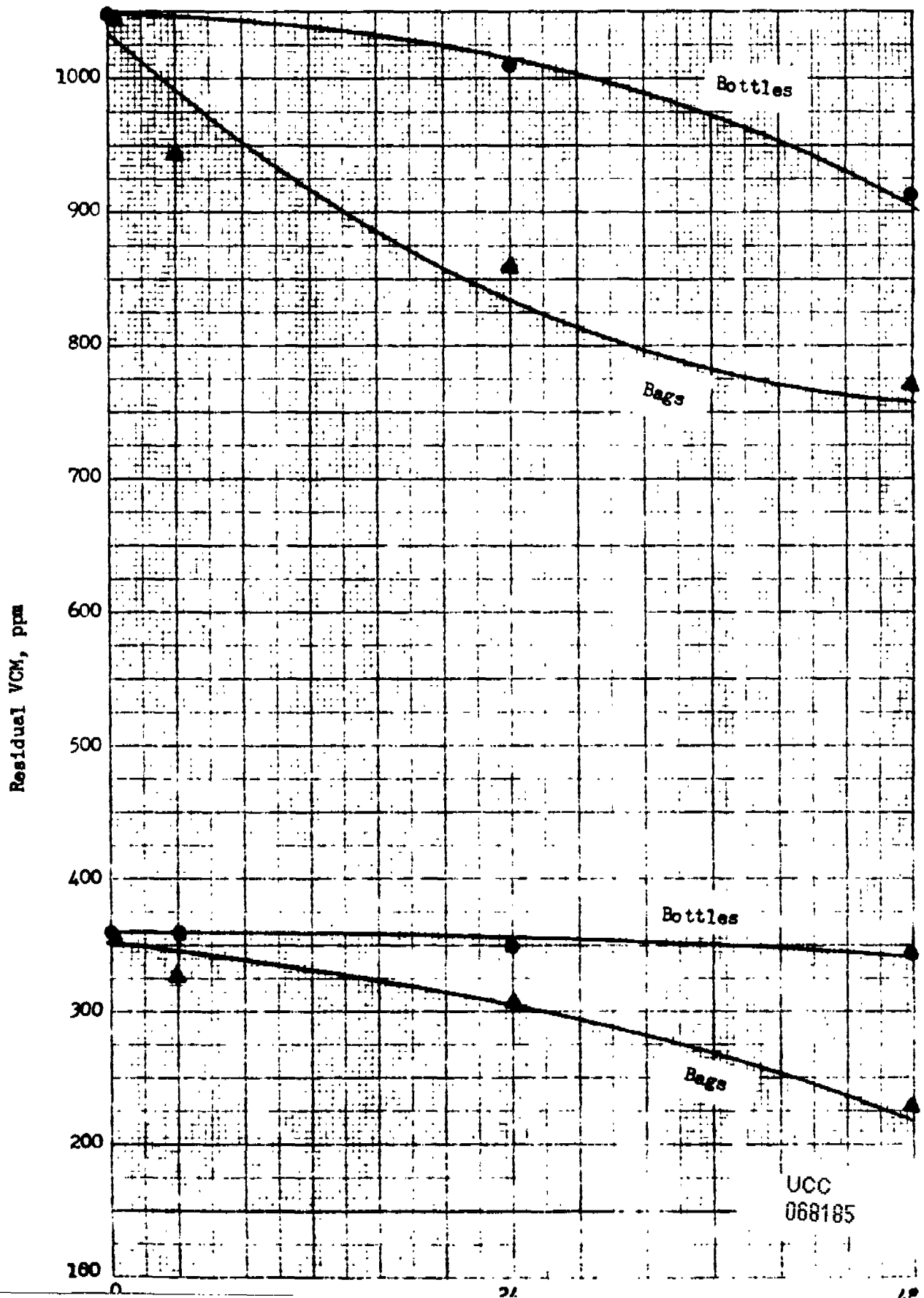
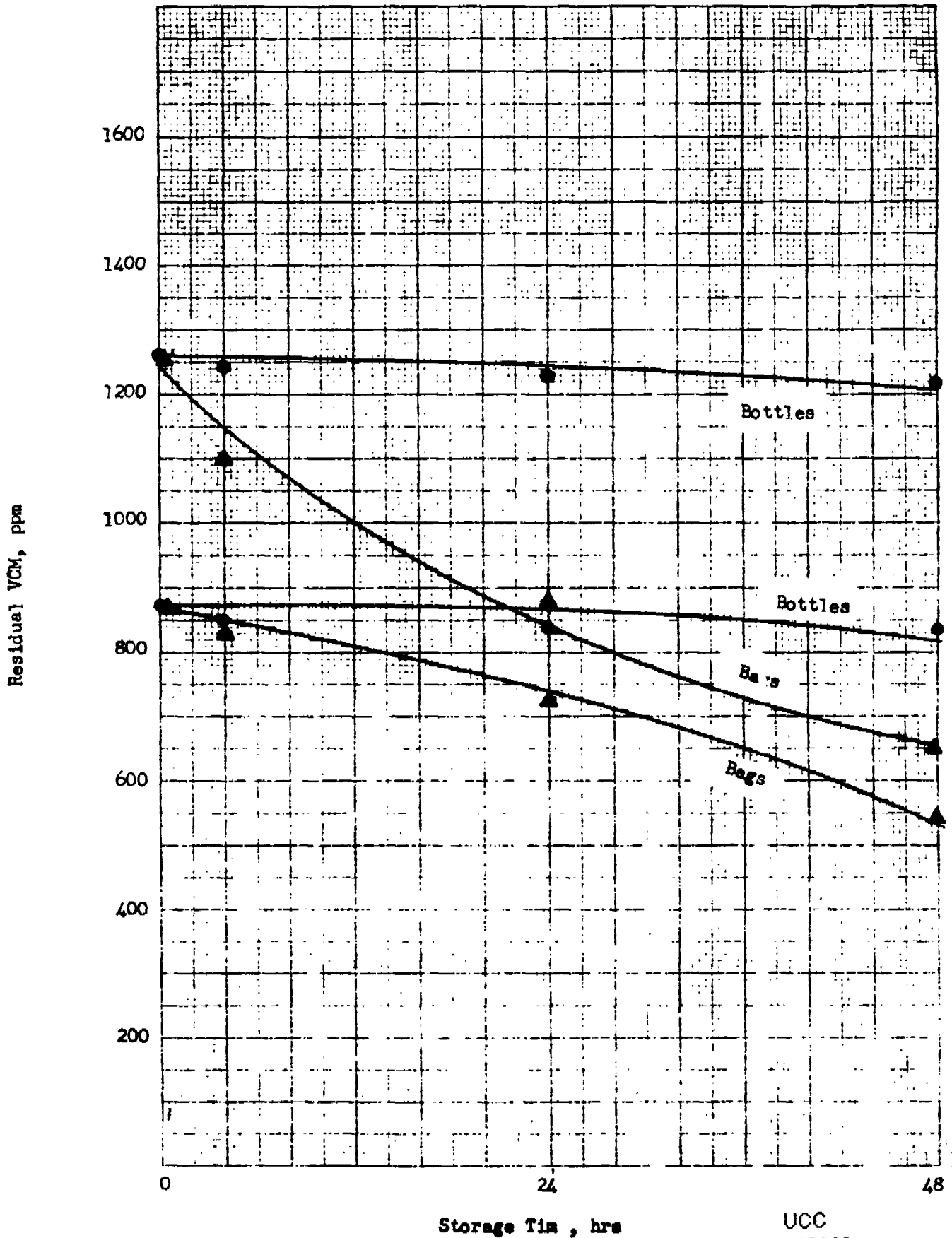


FIGURE III

Comparison of Residual VCM Stability of QSQH-7 Samples
Bottles versus Bags

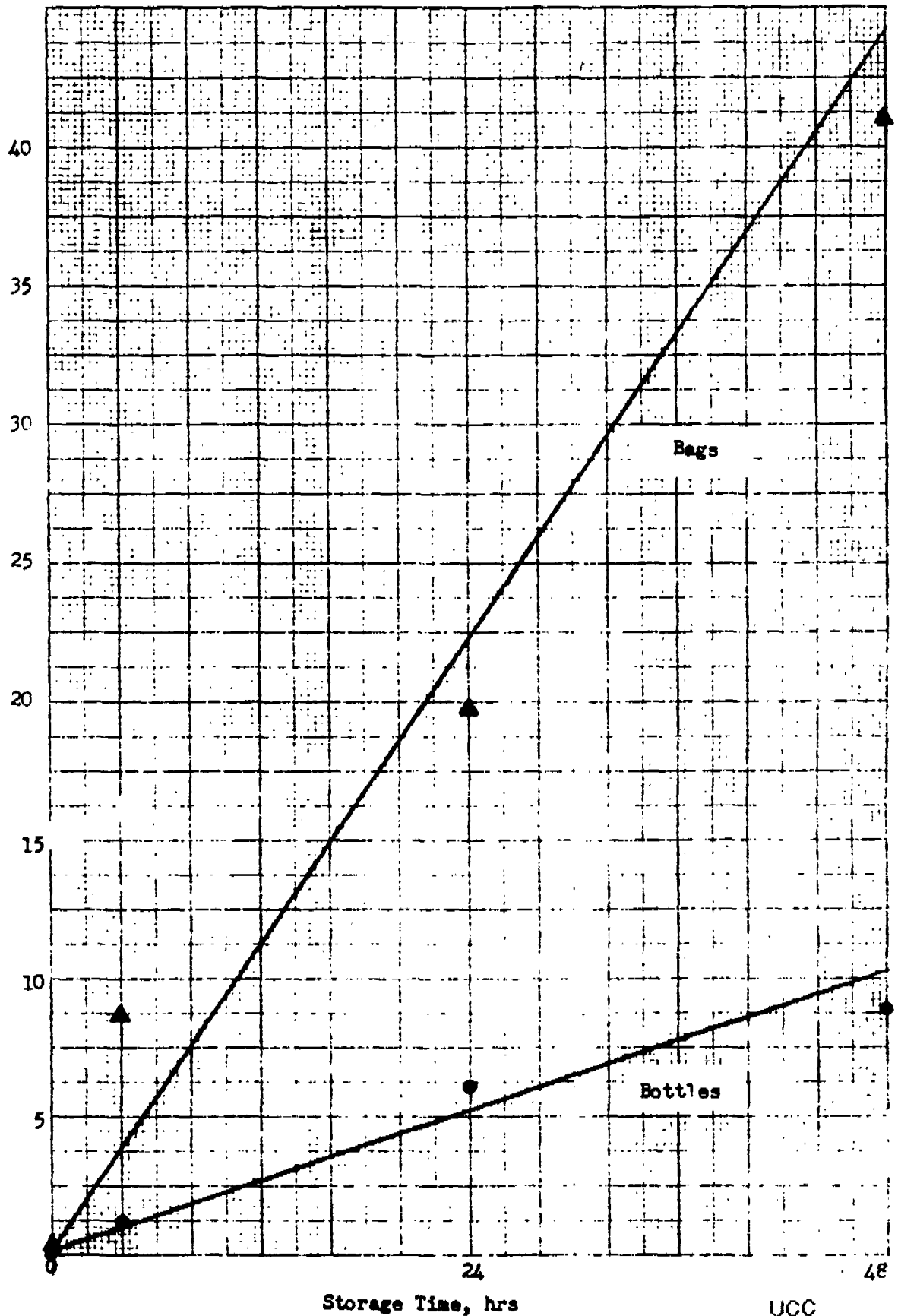


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FIGURE IV

Comparison of % Average Loss of Residual VCM in Suspension Vinyl Resins
During Storage in Bottles versus Bags

% Average Loss of Residual VCM



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