

nick Wheeler
What is the best way to
approach this issue?

Russ Davidson
4158

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Dick Lasher

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DATE: February 2, 1978

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SUPERVISOR: C. N. Merriam

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R. N. WHEELER, JR.

SUMMARY

Since mid-1977 there have been reports of restrictions being placed on the usage of vinyl coatings for the interior of potable water tanks in some states. The restrictions, originating in certain regulatory agencies, were said to be due to fear of vinyl chloride monomer (VCM) contamination of the water.

Attempts to revert those restrictions yielded a compromise by which individual coatings submitted to governmental laboratories could be qualified. This unprecedented demand would place an added burden to our customers, already laden with regulatory paperwork, and would likely hurt our participation in that market.

Three elements are presented here to demonstrate the folly of those regulations:

- 1) Recent work done by a Scandinavian researcher, demonstrating theoretically and experimentally that no VCM is retained in vinyl coatings, even under exaggerated conditions.

- 2) Calculations of hypothetical VCM concentration of VCM in a water tank coated with a vinyl system, assuming the most unfavorable set of circumstances (and disregarding point 1) results in a theoretical concentration of about 0.12 parts per trillion.

- 3) Vinyl resins have been approved and used for years in contact with ingestibles, without requiring individual coating qualification. If vinyls are compounded with other approved materials, individual coating qualification for the use in question appears unwarranted.

It is suggested that appropriate Union Carbide functions utilize these facts to obtain a permanent withdrawal of the restrictions.

restrictions.

Research and Development Department
Chemicals and Plastics
Union Carbide Corporation
Bound Brook, New Jersey

UCC
029862

INTRODUCTION

In mid-1977 it came to our attention, through Mr. W. Richter, Manager of Technical Service of the Mobil Chemical Corporation, that there were restrictions being placed upon the use of vinyl coatings in the interior of potable water tanks in the State of Virginia. The restrictions were reportedly originated at the regional office of the EPA. This information was relayed to Mr. R. J. Martell, who has responsibility for Maintenance Coatings liaison with governmental agencies.

Negotiations by Mr. Martell with the regional EPA officers resulted in a compromise, whereby vinyl coatings would be allowed, if they were cleared by the EPA laboratory in Cincinnati. However, since EPA laboratories are not geared to handle such requests from the general public, individual paint manufacturers would have to request their state or municipal customer to in turn submit a request for testing and clearance from the EPA laboratory. Such roundabout procedure is certain to lead to interminable delays at best, and at worst an eventual terminal blow to the use of vinyls in this market.

At the recent Annual Meeting of the Steel Structures Painting Council in Pittsburgh, we learned from representatives of the Pennsbury Coatings Co. that a similar ban is now in effect in the State of Delaware. Evidently, the news of the restriction in Virginia traveled faster than those of the compromised modification. This incident should be a warning of the potential that exists for uncontrolled snowballing of misinformation. The situation is serious, and requires swift and determined action at high levels to prevent further damage.

This report discusses some recent data which are helpful in clarifying the issues involved, hopefully leading to a more rational treatment of the subject.

Article by C. M. Hansen

At about the time that we first heard of the Virginia case, there was an important paper published in the respected Journal of the Oil and Colour Chemists' Association, published in England. The significance of this paper, a copy of which is included in this report, is enhanced by the fact that the author is an internationally respected authority in the field of coatings.

The Summary of the article is so brief and precise that it is worth including it here:

"There is no measurable retention of vinyl chloride in coatings formed from solutions. Calculations have shown that this is not anticipated and measurements in practical and exaggerated situations have demonstrated no measurable (<<1ppm) retained vinyl chloride".

The paper, which is short and very readable, comprises two major portions:

- 1) A review of the author's earlier work on solvent evaporation from drying vinyl films, and
- 2) Application of the author's conclusion to the problem of VCM retention.

It is important to realize that these two phases were conducted with different vinyl products. In the first phase, our VYHH was used, and the product is cited by name in several occasions. The second phase was carried out with a commercially available paint, made with a vinyl resin of undisclosed origin, having 16 ppm VCM.

Because of the obvious linkage that the casual reader may see between VYHH and the resin in the commercial paint, Mr. Hansen was contacted for a clarification. The reply, shown in the attached letter, dispells any connection.

At any rate, Mr. Hansen reports in the following sections the extremely important finding that no detectable VCM was encountered even in cases where the paint was seeded with 4,000 ppm VCM. In the subsequent conclusions, he states that:

"The dry film will never contain significant amounts of VCM, because the relative volatility will be high regardless of the solvent used".

Hypothetical VCM Concentration in a Water Tank

Union Carbide vinyl resins are certified having less than 0.2 ppm vinyl chloride monomer. It is an interesting exercise to find out the effect that 0.2 ppm in the resin would have in a typical water tank situation.

To that effect, we have selected arbitrarily Vinyl System No. 4 of the American Water Works Association, which is based on the Department of the Interior (Bureau of Reclamation) Specification VR-3. Union Carbide's starting Formula Suggestion to meet that Specification is VP-3010.

For our calculations, we have taken the case of a one-million gallon water sphere. The following assumptions have been made:

- 1) The tank is painted with AWWA System No. 4.
- 2) The paint formula used is VP-3010.
- 3) The vinyl resin contains 0.2 ppm VCM.
- 4) None of that VCM evaporates during drying.
(This, of course, has been disproven by Hansen)
- 5) All the VCM contained in the vinyl resin migrates at once in a single filling of the tank.

Calculations:

Volume of sphere:

$$V = \frac{4}{3} \pi r^3 = 4.189r^3$$

$$r = \sqrt[3]{\frac{V}{4.189}}$$

$$1,000,000 \text{ gallons} \approx 133,690 \text{ ft.}^3$$

$$r = \sqrt[3]{\frac{133,690 \text{ ft.}^3}{4.189}} \approx 32 \text{ ft.}$$

Surface of sphere:

$$\begin{aligned} S &= 4\pi r^2 = 12.57r^2 \\ &= 12.57 \times (32 \text{ ft.})^2 \approx 13,000 \text{ ft.}^2 \end{aligned}$$

1-mil coverage of VP-3010 : approx. 330 ft.²/gal.
 for 6 mils dry, coverage $\approx$ 55 ft.²/gal.
 for 13,000 ft.², approx. 236 gals. of paint needed.
 Paint (VP-3010) has approx. 1.4 lb. vinyl resin per gallon.
 $\therefore$ 236 gals. paint have $\sim$ 330 lb. $\sim$ 150kg. vinyl resin.

If vinyl has 0.2 ppm VCM = 0.2 mg/Kg

$\therefore$ 150 Kg x 0.2 mg/Kg = 30 mg. VCM

1,000,000 gals. = 8,330,000 lbs. water $\approx$ 3,778,500 Kg =

= 3,778,500,000,000 mg.

$\frac{3,778,500,000,000 \text{ mg. water}}{30 \text{ mg. VCM}} \approx 0.000,000,000,000,12$

= 0.12 part per trillion

Conclusion: Under the most unfavorable set of conditions (already negated by Hansen's work) the maximum concentration possible of VCM in water would be approximately one-tenth of a part per trillion.

Clearance of Coatings for Use in Water Tanks

A complicating factor in our efforts to obtain withdrawal of the restrictions is the fact that jurisdiction on this area is still ill-defined, split between EPA and FDA.

In his contacts with the EPA administrator whose decision started this problem, Mr. Martell obtained the allowance to use vinyl coatings, provided that they were submitted to the EPA laboratory in Cincinnati for testing. However, since there is no mechanism established to enable a private company to initiate this process, a paint manufacturer wishing to have his system accepted would have to persuade his customer (in this case a municipal or state agency) to, in turn, request approval from the EPA laboratory. Supporting data would have to be channeled through the same route.

It is easy to imagine that it would be hard to motivate an agency to initiate action with another. Furthermore, the follow-up and flow of information would be slow, inefficient, and prone to misunderstandings and snags. All this would amount to time and money for our customers, already taxed by the flood of regulatory paperwork.

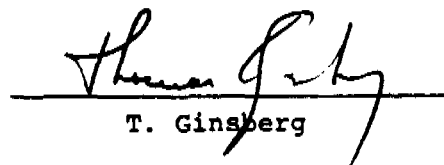
This procedure does not appear warranted. The Food and Drug Administration, which for many years has monitored the quality of coatings in contact with food, does not have such a coating qualification procedure. All that is required is that the coating manufacturer certifies that the ingredients used are FDA-approved for food contact. If this is good enough for cans, where the contact time between foodstuff and coating may be several years, it is hard to justify a more stringent control where the contact time is much shorter.

It appears that the situation is favorable to urge the EPA to adopt time-proven FDA-type compliance guidelines. This would save considerable time and effort to our customers thereby ensuring the uninterrupted use of vinyl coatings in this important application.

CONCLUSIONS

- (1) Recent work by a coatings researcher has demonstrated theoretically and practically that no residual vinyl chloride monomer remains in a film, even under exaggerated conditions.
- (2) Vinyl chloride monomer concentration calculations under the most adverse conditions demonstrate that the maximum could amount to about .1 parts per trillion.
- (3) Compliance procedures used by FDA for years in the safeguard of the public health have never contained qualification procedures for individual coatings. Adoption of such qualification by EPA appear unwarranted.

It is hoped that the data presented here will be useful in an energetic and sustained campaign to halt further spreading of damaging misinformation.


T. Ginsberg

TG:ld

Attachment: Appendix

FLASH

As this report was being typed, it was learned that the problem has appeared now in Wisconsin.

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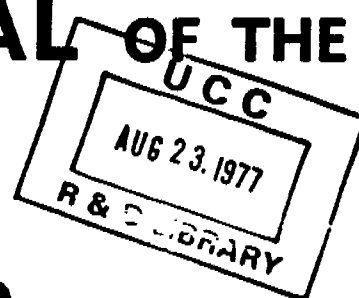
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Vinyl chloride retention in coatings formed from solutions

C. M. Hansen

Measurement and use of surface tension data in film-forming polymers

F. Ewans-Ebele and H. P. Schreiber

The microanalysis of copper oxide based marine antifouling paints in the scanning electron microscope

R. J. Bird

Non-conventional anticorrosive primers for steel

D. E. A. Williams-Wynn

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Details on page 270



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Transactions and Communications

Vinyl chloride retention in coatings formed from solutions*

By C. M. Hansen

Scandinavian Paint and Printing Ink Research Institute, Odensegade 14, 2100 København Ø, Denmark

Summary

There is no measurable retention of vinyl chloride in coatings formed from solutions. Calculations have shown that this is not anticipated and measurements in practical and exaggerated situa-

tions have demonstrated no measurable (< 1 ppm) retained vinyl chloride.

Keywords

Types and classes of coatings and allied products

ink
printing ink
foil coating

Raw materials for coatings binders (resins, etc.)

polyvinyl resin

raw materials used in manufacture or synthesis of ingredients for coatings

polyvinyl chloride

Properties, characteristics and conditions primarily associated with:

materials in general

evaporation rate

dried or cured films

solvent retention

La retention de chlorure de vinyle par revêtements à partir de solutions vinyliques

Résumé

Les revêtements à partir des solutions vinyliques ne retiennent pas de chlorure de vinyle en quantité dosable. Les calculs ont indiqué que ce n'est pas ce que l'on doit s'attendre, mais les

mesures dans la pratique et sous les conditions exagérées ont démontré que la quantité de chlorure de vinyle n'atteint pas à la limite dosable (< 1 ppm.)

Zurückhaltung von Vinylchlorid in aus Lösungen gebildeten beschichtungen

Zusammenfassung

In aus Lösungen gebildeten Filmen ist keine messbare Zurückhaltung von Vinylchlorid feststellbar. Berechnungen haben gezeigt, dass dies nicht von vornherein anzunehmen ist, und in praktisch

möglichen und übertriebenen Situationen vorgenommene Messungen haben keine messbare Zurückhaltung (< 1 pro Mio.) von Vinylchlorid gezeigt.

Introduction

Refs. 1-5

Various environmental authorities have focussed their attention on the presence of vinyl chloride monomer (VCM) in food packaging materials. This study was undertaken to explore the amount of retained vinyl chloride which might be anticipated in coatings and inks.

known that the relative evaporation rate² is a useful practical tool (which correlates with vapour pressure for example) for understanding first stage phenomena. Indeed, extensive studies with a vinyl chloride copolymer (VYHH-Union Carbide) were used to help confirm this fact⁴. The relative evaporation rate compares the rate of evaporation for a given solvent with that of *n*-butyl acetate which has arbitrarily been assigned a value of 100.

Film formation and the evaporation process

Films are formed from polymer solutions by the process of solvent evaporation. This evaporation has been shown to occur in two stages^{1,3}. These are controlled by air surface resistance and internal diffusion resistance, respectively.

The first stage of the evaporation process is influenced by such factors as solvent vapour pressure, air velocity, and solvent diffusion through a thin laminar boundary layer. It is

The second phase of solvent evaporation occurs over a much longer period of time which is usually called solvent retention. The loss of solvent is controlled by internal diffusion resistance within the film. Transport to the air surface is retarded by polymer molecular segments being required to move to create sufficient space for the solvent molecules to move. Consequently, larger and more bulky molecules move (diffuse) more slowly than smaller or more linear ones and tend to be retained for longer periods of time. Polymer to solvent "bonding" is not the significant retarding effect in this phase as evidenced by an extensive study of this phenomenon which also included numerous data for the

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same vinyl chloride copolymer mentioned above⁴. Polymers with lower glass transition temperatures can be expected to retain less solvent (or monomer) because of greater internal mobility. Poly(vinyl chloride) (PVC) is generally plasticized or modified by copolymerization to allow its use in a solution coating. Such coatings can be expected to retain less solvent or monomer than rigid poly(vinyl chloride), for example.

It might also be noted that the mathematics of diffusion predict a dependence of solvent loss on the square of the film thickness. This means that if a drying film is compared with one of twice its film thickness (at longer times), the thicker film will require four times as long to reach the same level of solvent (or monomer) content as the thinner film. Thus thicker films can inherently be expected to retain more solvent or monomer than thinner films. These relations have also been confirmed experimentally in the experiments mentioned above.

Estimates of vinyl chloride loss during the first phase

Refs. 3, 5-7

The first phase evaporation phenomena are closely related to what in the chemical engineering literature is termed "differential distillation" (distilling solvents in a batch process, for example). The polymer may influence the process to some degree, as will the activity coefficients for the mixtures involved. In a first approximation, however, these will not be considered.

The differential distillation process is described by McCabe and Smith⁸. In the simplest case, mathematical relations are derived for estimating the compositional changes in the liquid film as a function of material evaporated.

The equation concerned is:

$$\left(\frac{N_B}{N_{B0}}\right) = \left(\frac{N_A}{N_{A0}}\right)^{\alpha_{BA}} \quad (1)$$

where:

N_B = amount of B(VCM) present at a given stage in the evaporation process

N_{B0} = amount of B originally present

N_A = amount of A present at a given stage in the evaporation process

N_{A0} = amount of A originally present

α_{BA} = relative volatility of B to A

The relative volatility can be estimated by a simple ratio of the evaporation rates, but this value is not available for vinyl chloride. In the example below the relative volatility was, therefore, estimated by a ratio of the vapour pressures at room temperature^{9,7}.

$$\alpha_{BA} \approx \left(\frac{P_B}{P_A}\right) \quad (2)$$

Example:

Assuming a typical lacquer formulation having the following composition:

Vinyl copolymer 25g
Methyl ethyl ketone 75g
Vinyl chloride monomer variable

The above mentioned experiments with VYHH have demonstrated that the end of the first phase is associated with a solvent composition of about 0.2g solvent/g VYHH². Thus, the first phase represents a reduction of the volatile content of the lacquer from

3g/g VYHH to 1/15 of this value before entering into the second phase. α_{BA} at 20°C is approximately 2650/71.5 = 37.06 from equation 2.

Substituting these values into equation 1 gives:

$$\frac{N_{VCM}}{N_{VCM0}} = \left(\frac{1}{15}\right)^{37.06} = 2.59 \times 10^{-44}$$

Thus it is clear that theoretically there should be essentially no VCM remaining at the end of the first stage of the evaporation process.

For the sake of including all factors, the data on solvent retention for VYHH cited above indicate that a further reduction of the solvent and/or monomer content present at the start of the diffusion controlled phase will occur to the extent of a factor somewhat greater than two within a reasonable time (hours). Should the temperature be increased above room temperature, the mobility of the polymer chain segments will be increased with an accompanying increase in the diffusion coefficients of the solvent/monomer molecules. A greater reduction in the amount of retained volatile material will result.

Experimental

Ref. 8-11

The analyses for VCM were carried out using a gas chromatograph. For liquid samples with a large VCM content the solvent or lacquer solution was injected directly into the gas chromatograph. When the VCM content was small, the analysis was done by the head space method^{8,12}. Here the principle is to place the sample in a suitable container, such that a gaseous sample can be removed with a syringe through a membrane without otherwise disturbing the contents. The volatile contents establish an equilibrium in the container and a calibration of the VCM signal by the method of addition allows analysis of the VCM content in the sample itself.

Experimental results—films

A commercially available lacquer with the composition described in the example above was used for these studies. This lacquer was found to have 16ppm VCM based on the nonvolatile portion of the formulation. Films were made in Petri dishes. These were made at an exaggerated film thicknesses of 140 to 180 microns. After 66 hours of drying at room temperature (according to the film thickness squared rule this corresponds to 1.6 minutes for the normal film thickness of 3 microns for this product) no measurable (< 1ppm) VCM could be detected in the film.

It might also be noted that measurable VCM could not be found in films applied in the manner the product is customarily used *ie* in a film thickness of 3 microns on aluminium foil.

Experimental results—evaporation

Experimental values for α_{BA} were determined by adding 4000ppm VCM to methyl ethyl ketone (MEK) and following the concentration of VCM as a function of the per cent MEK evaporated. Figure 1 shows a gas chromatogram of the VCM/MEK mixture after 4 per cent MEK and 48 per cent VCM are evaporated. Collected results from similar analyses are shown in Figure 2.

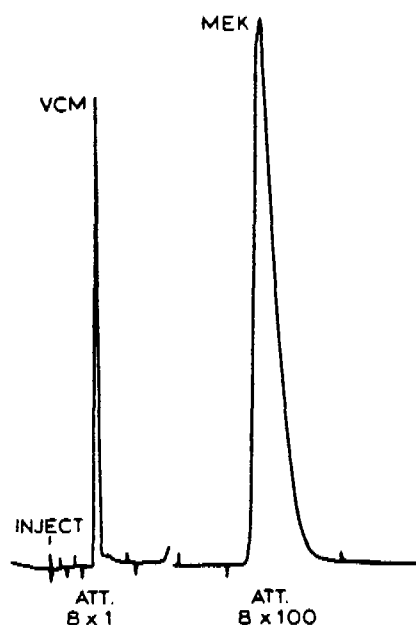


Fig. 1. Gas chromatogram of a 4000ppm solution of VCM in MEK after 4% has evaporated. Calculated VCM content: 2080ppm. Column: 2m, 1/16" id. Porapak Q; Temperature: 170°C; N_2 ; 30ml/min; Detector: FID; Att: 8 x 1 and 8 x 100; Gas chromatograph: Perkin Elmer F 30

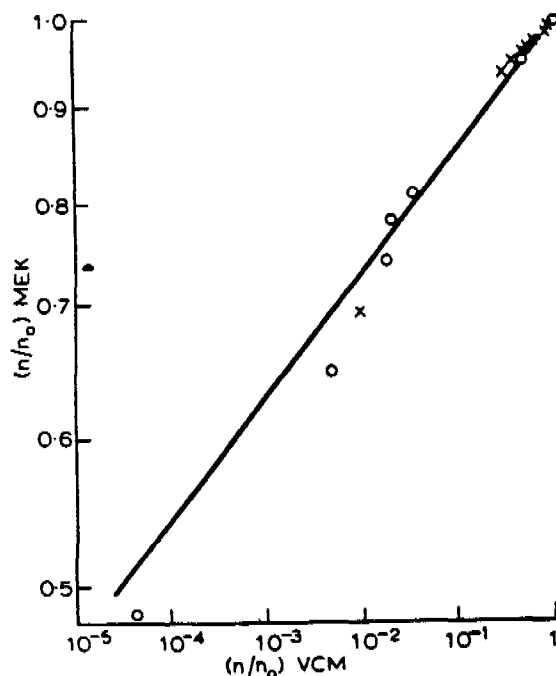


Fig. 2. Evaporation of VCM from MEK, x Initial concentration 4000ppm in MEK, O Initial concentration 25% vinyl resin solids, 75% MEK, 4000ppm VCM (dry resin)

The same evaporation experiment was repeated using the vinyl lacquer formulation described above (25 per cent vinyl polymer). 10 per cent of the MEK in the lacquer was

evaporated and replaced by a solution of about 10 000ppm in MEK. The original VCM concentration was about 4000ppm based on the dry polymer. The experimentally determined concentration of VCM was about 0.2ppm after half of the MEK was evaporated. These results are also included in Figure 2.

Figure 3a shows a gas chromatogram of the head space over a sample of vinyl lacquer after half of the MEK was evaporated. Figure 3b shows a corresponding sample where VCM was added in an amount equal to 1.5ppm based on the dry polymer.

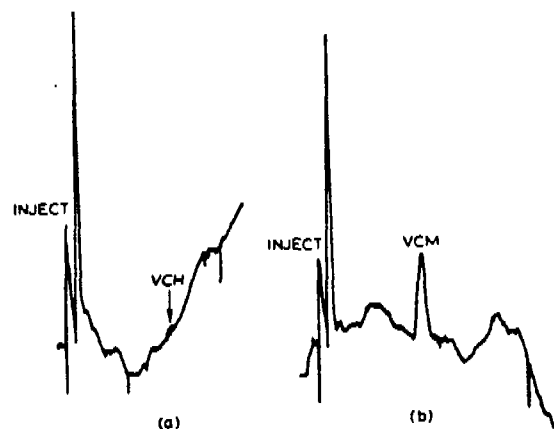


Fig. 3a. Gas chromatogram of the head space over a vinyl lacquer after 50% MEK is evaporated

Fig. 3b. Gas chromatogram of the head space over a vinyl lacquer after 50% of the MEK has evaporated, 1.5ppm VCM added (based on dry solids)

Column: 2m, 1/16" id. Porapak Q; Temperature: 130°C; 10 min prog-20°/min to 200°C; N_2 ; 30ml/min; Detector: FID; Att: 1 x 1; Gas chromatograph: Perkin Elmer F 30

Conclusion

As can be seen in Figure 2 there is no significant difference in the evaporation data between the VCM/MEK solution and the VCM/MEK/vinyl polymer solution. In both cases α_{SA} is about 15. Thus it cannot be expected that measurable amounts ($N/N_0 \approx 10^{-11}$) of VCM will be present at the end of the first phase of the evaporation process when the MEK content is reduced to 1/15 of the original amount.

Polymers containing small amounts of VCM are used in coatings applied from solutions. The VCM present in the liquid product will evaporate together with the solvent during the first phase of the film formation process. The dry film will never contain significant amounts of VCM, because the relative volatility will be high regardless of the solvent used. The present results using MEK as solvent justify this conclusion, since MEK is among the most volatile of solvents used and α_{SA} will also be high for any other solvent used.

Acknowledgment

The author would like to thank Karen Eng who has been responsible for the experimental work.

(Received 25 February 1977)

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SCANDINAVIAN PAINT AND PRINTING INK RESEARCH INSTITUTE
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DENMARK
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Vor ref.: CMH/IMcG

Deres ref

DATO 1977.09.02

Dear Mr. Ginsberg:

I can inform you that the vinyl polymer used in our VCM retention studies did not originate from Union Carbide. I am informed also that European sources of this type polymer can also now supply at less than 1 ppm VCM.

Sincerely,

Charles M. Hansen

Charles M. Hansen

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DEPARTMENT OF THE INTERIOR

(BUREAU OF RECLAMATION)

Request for copies of Department of the Interior specifications should be directed to Commissioner's Office, Building 53, Denver, Colorado 80202

VR 3

VINYL RESIN PAINT; Aug. 14, 1964. UNION CARBIDE Formulation VP-3010 using BAKELITE vinyl resins VMCH and VYHH.

FORMULA SUGGESTION VP-3010

Maintenance Primer Finish*

FORMULA	Parts by Weight
BAKELITE Vinyl Resin VMCH	11.1
BAKELITE Vinyl Resin VYHH	4.5
FLEXOL Plasticizer DOP	3.0
Pigment (1)	13.0
Asbestine 3X	3.9
Methyl Isobutyl Ketone	16.1
2-Nitropropane	16.2
Toluene	32.2
	<u>100.0</u>

PROCEDURE

1. Dissolve resins into the solvent system.
2. Charge all ingredients** and resin solution into pebble mill and grind for 24 hours.

**except in aluminum system

- (1) White System - Titanium Dioxide, rutile, non-chalking.
 Red System - Synthetic Red Iron Oxide.
 Black System - Synthetic Black Iron Oxide.
 Aluminum System - Aluminum Powder, 9.9 parts rather than 13.0 parts.

*For best adhesion of primers to bare steel, it is helpful to add 1% of 85% phosphoric acid, based on the weight of vinyl resin VMCH, to the primer formulation.

VR-6

VINYL RESIN PAINT; July 6, 1960. UNION CARBIDE Formulation VP-3001 using BAKELITE vinyl resins VMCH in prime coat, VYHH in body coat and VYHH and VYNS in seal coat.

FORMULA SUGGESTION VP-3001

Maintenance System

FORMULA	Parts by Weight			
	Prime* Coat	Red Body Coat	Grey Body Coat	Seal Coat
BAKELITE Vinyl Resin VMCH	15.00	15.00	15.00	13.47
BAKELITE Vinyl Resin VYHH	-	-	-	5.77
BAKELITE Vinyl Resin VYNS	-	-	-	1.38
FLEXOL Plasticizer 10-10	3.00	3.00	3.00	-
TiO ₂ , rutile, non-chalking	15.00	-	15.00	-
Carbon Black	1.00	-	1.00	-
Synthetic Red Iron Oxide	-	15.00	-	-
Aluminum Powder (1)	-	-	-	(1)
Methyl Isobutyl Ketone	16.50	17.00	16.50	19.88
2-Nitropropane	16.50	17.00	16.50	19.84
Toluene	33.00	33.00	33.00	39.69
	<u>100.00</u>	<u>100.00</u>	<u>100.00</u>	<u>100.00</u>

(1) "Alcoa" 408 suggested - 1.5 lbs. to be added to 1 gallon of solution for use.

*For best adhesion of primers to bare steel, it is helpful to add 1% of 85% phosphoric acid, based on the weight of vinyl resin VMCH, to the primer formulation.

PROCEDURE

1. Dissolve resins in solvent system.
2. Charge all ingredients** and resin solution to pebble mill and grind for 24 hours.

**except in aluminum system

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VP-3010 JAN. 24 '78

RAW MATERIAL	WEIGHT	POUNDS 100 GAL	GALS/ 100 GAL	PERCENT BY WEIGHT
VMCH RESIN	11.10	99.85	8.89	11.10
VYMH RESIN	4.50	40.48	3.57	4.50
FLEXOL DOP	3.00	26.99	3.31	3.00
TI PURE R-960	13.00	116.94	3.41	13.00
TALC	3.90	35.08	1.52	3.90
MIBK	16.10	144.93	21.62	16.10
2-NP	16.20	145.73	17.69	16.20
TOLUENE	32.20	289.66	40.01	32.20
TOTALS	100.00	899.57	100.00	100.00

CONSTANTS

PVC: 23.89 %
 THEOF. 1 MIL COVERAGE: 331.74 SQ.FT./GALLON
 NONVOLATILES BY WEIGHT: 35.50 %
 NONVOLATILES BY VOLUME: 20.68 %
 WEIGHT PER GALLON: 9.00 LBS.
 RAW MATERIALS COST: 2.41 DOLLARS/GALLON
 = 0.7259 CENT/SQ.FT./DRY MIL
 SPECIFIC GRAVITY: 1.08 G/CC

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