STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF MACOMB

KENNETH GRIMM, Personal Represetnative of the ESTATE OF HELEN GRIMM, Deceased,

Plaintiff,

NO. 83-2872 NO

vs.

FORD MOTOR COMPANY, a foreign corporation, et al,

Defendants.

PRETRIAL STATEMENT

Plaintiff alleges that defendants, in their industrial endeavors, have contaminated the air with pollutants which caused deceased-plaintiff to incur a carcinoma resulting in her death. Plaintiff has heretofore filed interrogatories, defendants shall answer the same within thirty (30) days hereof.

The parties are going to complete discovery. It is anticipated that at the next pretrial hearing that plaintiff will be in a position to more specifically demonstrate the basis for his cause of action.

Adjourn this pretrial to June 19, 1985 at 9:00 a.m.

RAYMOND R. CASHEN
Circuit Court Judge

Dated: December 17, 1984

cc: Francis P. Hughes
Ralph Valitutti
John Lynch
Ralph Safford
Ralph Barbier
Steven Nadeau
Dennis M.Haffey

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_____/

Case No. 83-2872 NO

Hon. Raymond R. Cashen

DEFENDANT STAUFFER CHEMICAL COMPANY'S
ANSWERS TO PLAINTIFF'S FIRST SET OF INTERROGATORIES

Defendant Stauffer Chemical Company (Stauffer) in answer to Plaintiff's First Set of Interrogatories, states:

1. 1963 by American Chemical Company, since acquired by Stauffer.

2. See answer to previous Interrogatory.

3. No.

4. No.

5. a. SCC 676, SCC 676P, SCC 40.

b. SCC 676 1973-1976
SCC 676P 1974
SCC 40 1975-1976

c. SCC 676 and SCC 676P are polyvinyl chloride. SCC 40 is a copolymer of vinyl chloride and vinyl acetate.

d. 1973: 5,000 lbs. of SCC 676.
1974: 2,797,320 lbs. of SCC 676 and SCC 676P.

Information regarding later years is irrelevant due to the fact Plaintiff's Decedent no longer lived in the vicinity of the Ford plant.

6. a. See answer to Interrogatory 5(c).

b. See answer to Interrogatory 5(a).

c. Stauffer believes that SCC 676 and SCC 676P were used in the calendering process and that SCC 40 was used as an adhesive.

d. SCC 676 and 676P: Bulk and 50 lb. bags. SCC 40: 50 lb. bags.

e. PVC resins are usually processed with other materials to produce different end products. Stauffer has no specific knowledge of the nature or scope of the processing used by Ford.

7. SCC 676 and SCC 676P:

a. April, 1966.

b. April, 1966 in Delaware City.

c. Don Hunter, now of Tenneco.

d. Stauffer sold the Delaware City plant several years ago and is no longer in possession of the described records. To the extent that Stauffer still has records available, these are located in Westport, Connecticut.

e. See answer to previous Interrogatory.

f. Stauffer objects to the Interrogatory as irrelevant and unduly burdensome and for the further reason that since Mr. Hunter has not been employed by Stauffer since approximately 1971, the requested information is not available. It can be obtained by deposition.

SCC 40:

a. July, 1969.

b. March, 1968 in Delaware City.

c. Charles Harrington, whereabouts unknown.

d-e. See corresponding answers for SCC 676 and SCC 676P above.

f. Stauffer objects to the Interrogatory as irrelevant and unduly burdensome and for the further reason that since Mr. Harrington has not been employed by Stauffer since approximately 1971, the requested information is not available. It can be obtained by deposition.

8. Stauffer objects to the Interrogatory as overbroad, irrelevant and unduly burdensome.

9. Stauffer Chemical Company supplied the warning specified

in 29 CFR §1910.1017 to purchasers of its product.

10. No. There was no scientifically recognized basis for recommending a restriction or limitation on the use of PVC products.

11. Yes.

a. Not applicable.

b. A 500 ppm threshold limit value for VCM was set in 1961 by the American Conference of Government Industrial Hygienists. In 1974, this value was reduced to 200 ppm. OSHA itself adopted this value as the OSHA standard for vinyl chloride which was in effect until April, 1974 when an emergency temporary standard was established, reducing the level to 50 ppm. In late 1974, OSHA adopted the present VCM standard for occupational exposure. Stauffer has complied in full with all applicable federal, state and local safety standards, orders, regulations and laws in connection with the manufacture of its PVC products. No standard has ever been adopted which specifically applies to the PVC resins supplied to Ford Motor Company.

12. Yes. See answer to Interrogatory 11.

13. The PVC products sold to Ford Motor Company were not materially changed during the period Plaintiff's Decedent was living on Lafayette Street. Changes made since that time are irrelevant pursuant to MCL 600.2946(3) and are only related to plant process modifications made to satisfy Federal Environmental Protection Agency plant emission requirements.

14. No.

15. No.

16. Yes.

a. No.

b-c. Not applicable.

17. Yes.

a. Amounts of VCM in PVC generally are unknown.

18. The best answer Stauffer can provide to this technical question is to refer Plaintiff to a paper, "Small Molecule Migra-

tion in Products Derived from Glassy Polymers", attached to these Answers. Stauffer has no data regarding its products which would provide the information requested.

19. See answer to Interrogatory 18.

20. See answer to Interrogatory 16.

21.

Stauffer Product	Ford Specification
SCC 676	96F51
SCC 676P	None
SCC 40	96F181

22. No.

23. No studies have been conducted on volatility since PVC is not volatile. Studies concerning heat stability are irrelevant. Heat stability relates to the usefulness of the product, not its safety.

24. No.

a. Stauffer has no duty or right to inspect the operations of users of its products. Users of Stauffer's PVC products are sophisticated in the use of those products and are responsible for the implementation of their own safety systems under applicable law and government regulations.

b. Not applicable.

25. See answers to previous Interrogatory.

26. a. Stauffer's PVC products were inspected by Stauffer's quality control personnel prior to release for shipment.

b. Prior to Stauffer's sale of the plant, the person responsible was Edward Weleski, Quality Control Manager, Delaware City, Delaware.

c. To the extent that records are still in Stauffer's possession, they are located in Westport, Connecticut.

27. Yes. Failure to meet standards for one or more of the following:

a. particle size;

b. molecular weight;

c. resin color;

d. contamination;

e. volatiles (water);

f. fish eyes (gels).

28. Stauffer has from time to time made changes in manufacturing procedures as a result of rejections which occurred prior to shipment.

29. To the extent that Stauffer is still in possession of these records, they would be in the custody of Tom Tighe, Stauffer Chemical Company, Westport, Connecticut.

a. To the extent of Stauffer's knowledge, this answer is not applicable.

30. Stauffer has no record of ever having engaged in any media advertising of the PVC products in question.

31. Yes. The nature of the materials distributed prior to 1977 is unknown. Copies of representative 1977 materials are attached.

32. See position papers and other data presented during OSHA hearings in 1974 which are all matters of public record.

33. See answers to Interrogatories 9 and 32.

34. See answer to Interrogatory 6(e).

35. See answers to Interrogatories 18, 19 and 32.

36. See answers to Interrogatories 6(e) and 32.

37. No. PVC products have never been shown to be associated with liver cancer, brain cancer, breast cancer or hematolymphopoietic system cancer.

38. No.

39. There have been no deaths or cases of liver cancer, brain cancer, breast cancer or hematolymphopoietic system cancer among employees which Stauffer attributes to the inhalation of VCM.

40. No. See answer to Interrogatory 39.

41. With respect to the physical conditions listed in Interrogatory 39, no.

42. a. See answer to Interrogatory 39.

b-e. Based upon available records and to the best of Stauffer's knowledge and belief, there are no such cases.

43. Yes.

I. a. Plaintiff claimed exposure to polyvinyl chloride and other chemicals.

b. Court of Common Pleas
Cuyahoga County, Ohio
Docket No. 976623
Helen Arthur, Executrix v.
B. F. Goodrich Chemical Company, et al

c. Approximately November 3, 1977.

d. Dismissed with prejudice as to Stauffer.

e. Not applicable.

f. See answer to Interrogatory 43(d).

II. a. SCC 676, SCC 676P and SCC 40.

b. United States District Court
Eastern District of Michigan
Civil Action No. 78 70769
Stanley & Joyce Mikyska v.
Union Carbide Corp., et al

c. April 5, 1978

d. None. The claim was settled by the parties.

e. Not applicable.

f. Several of the Defendants, including Stauffer, paid a lump sum to the Plaintiffs to settle the claim without any admission of liability by Stauffer or the other Defendants.

44. None at Ford Motor Company, except for a tour by our counsel in April, 1979 as part of another lawsuit. Inspections of other facilities are not relevant to this action.

a. See answer to Interrogatory 24.

b. Not applicable.

45. 1965. Delaware City, Delaware.

46. Yes. The monitoring was the result of an inspection by a fire insurance carrier.

47. Air was pumped through a filter and the matter collected was analyzed by gas chromatography for VCM and other substances. At VCM levels above 1 ppm a warning was given and the condition was

corrected. If the level exceeded 5 ppm, employees used respirators.

48. Yes.

a. A succession of rules since 1974.

b. Not applicable.

c. Plant Standard Practice Instructions. Frank Doyle, Delaware City, Delaware.

d. See answer to Interrogatory 9.

49. Manufacturing Chemists Association
1825 Connecticut Avenue
Washington, D.C. 20009

Organization Resources Counselors
1625 I Street
Washington, D.C. 20006

Vinyl Chloride Safety Association
(no fixed address)

The Society of the Plastics Industry
355 Lexington Avenue
New York, New York 10017

American National Standards Institute
1430 Broadway
New York, New York 10018

Chemical Specialties Manufacturing Association
Washington, D.C.

50. Stauffer employs an epidemiologist at Westport, Connecticut whose name is Mary Palshaw.

51. August, 1975.

a. To organize and direct a department of occupational medicine.

b. Westport, Connecticut.

c. Organize and institute a corporate medical program with emphasis on industrial hygiene, toxicology and occupational medicine; advise operating divisions of corrective and protective measures in the interest of employee health and safety.

d. Wayne C. Jaeschke, Stauffer Chemical Company, Westport, Connecticut.

Medical Director: Herbert L. Northrop, M.D., Stauffer Chemical Company, Westport, Connecticut.

52. 1973.

a. Primarily to measure airborne materials in the workplace.

b. Westport, Connecticut.

c. Primarily the measurement of airborne materials in the workplace.

d. James Call, Westport, Connecticut.

Hygienist: John Bolton, no longer with Stauffer.

53. No. There was no scientifically recognized reason for doing so.

54. While it is believed Stauffer may have done so, the information requested is privileged, irrelevant and unlikely to lead to admissible evidence.

55. There has been no such testimony.

56-63. Stauffer will provide the requested information when it becomes available.

The answers to these Interrogatories were obtained from Stauffer's records by various Stauffer employees and from answers to similar interrogatories previously provided in Mikyska v. Union Carbide, et al, Civil Action No. 78 70769, U.S. District Court, Eastern District of Michigan. In addition, Stauffer employee Herb Northrop provided information for answers to Interrogatories 37 through 42.

AFFIDAVIT

STATE OF Connecticut)
)ss.
COUNTY OF Fairfield)

ANDREW F. FINK, being first duly sworn, deposes and says that he is an employee of Stauffer Chemical Company, authorized to make this Affidavit and to state that the foregoing Answers to Plaintiff's First Set of Interrogatories are true to the best of Stauffer's knowledge, information and belief.

Andrew F. Fink

Subscribed and sworn to before me
this 17th day of December, 1984.

Notary Public

DENISE M. TTLER
NOTARY PUBLIC
MY COMMISSION EXPIRES MARCH 31, 1989

GENERAL ARTICLES

Small Molecule Migration in Products Derived from Glassy Polymers

William J. Koros* and Harold B. Hopfenberg

Department of Chemical Engineering, North Carolina State University, Raleigh, North Carolina 27650

The implications of the combined dual mode sorption and partial immobilization transport models on the migration of small molecules from glassy polymers have been considered. Neither dual-mode sorption nor "immobilization", in the sense used here, contributes to irreversible retention of sorbed species if the assumption of local equilibrium between the two sorption modes is satisfied. An apparent trapping of otherwise diffusable species results from the equilibrium partitioning of migrant between polymer and extractant phases rather than from an inherent kinetic limitation to transport. The interrelationship between the transport predictions of the partial immobilization model and the more general concentration-dependent diffusion coefficient phenomenology is emphasized throughout the analysis. A case study which presents calculated predictions of extraction kinetics of vinyl chloride monomer from 40-mil poly(vinyl chloride) sheet is discussed. The analyses pertain to idealized migration of small molecules into foods or more directly to actual extraction protocols for detection of trace components in polymeric materials.

The migration of trace amounts of solvents, reaction and degradation byproducts, additives, oligomers, and monomers from polymeric barriers used in food and beverage packaging can affect consumer acceptance, product quality, and regulatory approval of candidate packaging materials. The serious economic impact related to accurate prediction of long-term migration effects has motivated this presentation of the thermodynamic and kinetic realities governing transport in and from glassy polymeric barriers.

Much of the detailed experimental characterization of the migration of low molecular weight molecules in glassy polymers has focused on CO_2 transport in support of the massive research and development of polymeric containers for carbonated beverages (Michaels et al., 1963a,b; Vieth and Sladek, 1965; Toi, 1973; Fenelon, 1974; Kollen, 1975; Koros and Paul, 1978a–c). There is increasing recognition that dual-mode behavior is a rather general characteristic of nonswelling (low concentration) penetrant sorption in glassy polymers (Berens, 1974a,b; 1979; Koros et al., 1977; Barrie et al., 1978). The migration of trace amounts of solvents, monomers, oligomers, additives, and degradation byproducts should, therefore, be governed by the equilibrium and kinetic implications of the dual-mode model. An unfortunate and somewhat inconsistent designation has been assigned to the Langmuirian component of dual-mode sorption. These species were originally termed "immobile" although the useful transport analyses have also typically assumed local equilibrium between the Langmuirian and Henry's law species. The local equilibrium assumption implies that there can never be a significant difference in the chemical potential of penetrant sorbed in the two modes in any local region of the polymer. This suggests that interchange between species in the two modes is rapid compared to the time scale required for a significant change in the local value of the chemical potential. The validity of the local equilibrium assumption has been verified by pulsed NMR for ammonia in glassy polystyrene (Assink, 1975) and by numerous transport studies for gases in glassy polymers in concentration ranges corresponding to several thousand parts per million (Fenelon, 1973; Toi, 1973; Koros and Paul, 1978a–c).

The confusion regarding the implications of the term "immobilization" on ultimate release kinetics has prompted the analysis presented here regarding the effects of Langmuir sorption and "immobilization" on the migration kinetics of trace components from glassy polymers. Although the "equilibrium criterion" per se is sufficient to permit complete removal of all species into an infinite sink, the detailed kinetics of migration are, in fact, affected by the relative populations of Langmuir and Henry's law species and the degree of "immobilization" of the Langmuirian penetrant. The primary objective, therefore, of this paper is the presentation of a detailed and quantitative case study to aid in prediction of migration rates from glassy polymers complicated by multiple sorption modes and various kinetic and equilibrium possibilities.

Analysis and Discussion

Equilibrium Sorption Isotherms. A variety of equilibrium isotherms relating penetrant concentration in a polymer to penetrant concentration in a contiguous fluid phase are observed for polymeric substrates. Gas sorption in rubbery polymers follows Henry's law (Michaels and Bixler, 1961; Stern et al., 1969). Sorption of organic vapors in many rubbers is characterized by an exponential increase in the concentration plotted as a function of vapor activity (Rogers et al., 1960). Such isotherms are sometimes referred to as having a BET type III form. More complex behavior involving an inflection is frequently observed for vapor sorption in glassy polymers over extended concentration intervals. At low concentrations, the concave downward form of such isotherms suggests dual mode sorption effects, while at higher concentrations it is believed that swelling or penetrant clustering effects predominate (Berens, 1975; Stannett et al., 1978). The dual-mode isotherm, typical of low concentration penetrant

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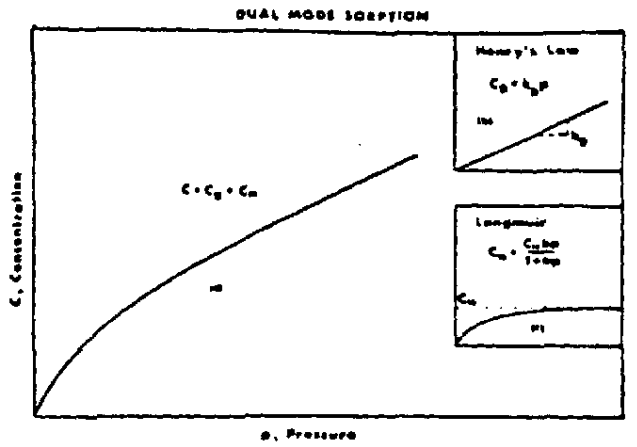

Figure 1. Schematic representation of dual mode sorption isotherm (curve a) comprised of a Henry's law component (curve b) and a Langmuirian component (curve c).

sorption in glassy polymers, is presented schematically in Figure 1.

The terminology "dual-mode" derives from the hypothesis that the concave downward shaped isotherm shown in Figure 1 results from the linear superposition of the individual Henry's law and Langmuir isotherms presented in Figures 1b and 1c, respectively (Barrer et al., 1958; Michaels et al., 1963a,b).

The relationship between the total concentration, C, and the parameters describing the dual-mode sorption isotherm is given by eq 1

$$C = k_D p + \frac{C_H' b p}{1 + b p} \tag{1}$$

Thus

$$C = C_D + C_H \tag{2}$$

where C_D is the concentration of penetrant dissolved in the matrix obeying Henry's law (i.e., $C_D = k_D p$) and C_H is the concentration of penetrant presumably held in unrelaxed gaps and described by the Langmuir isotherm. The parameter k_D is the Henry's law constant for the dissolved mode and p is the gas partial pressure in the surrounding bath (alternatively, C_g, the surrounding bath concentration could be used instead of p). The parameters, C_H' and b are the Langmuir saturation constant (cm^3(STP)/cm^3) and the Langmuir affinity constant (atm^{-1}), respectively. The relative contribution of the Langmuir and Henry's law modes to sorption is conveniently expressed by the dimensionless parameter $K = C_H' b / k_D$. Algebraic simplifications in the following treatment are also provided by defining $\alpha = b/k_D$.

Fickian Transport. The phenomenological transport law describing unidirectional migration of low concentrations of small molecules in polymeric materials is given by

$$N = -D_{eff} \frac{\partial C}{\partial x} \tag{3}$$

where N is the observed flux of migrant, D_{eff} is the effective diffusion coefficient of the migrant, C is the total concentration of dissolved component, and x is the direction of diffusion. Behavior which conforms to eq 3 is termed Fickian: various concentration dependencies of D_{eff} are often observed and are completely consistent with the definition of Fickian transport (Crank, 1975). The monotonically increasing, albeit inflecting dependence of D_{eff} on C shown in Figure 2 is characteristic of diffusion coefficients for a number of penetrants at relatively low concentrations in glassy polymers (Koros et al., 1976; Chan et al., 1978; Barrie et al., 1978). According to the dual mode theory, such concentration dependency of the effective diffusion coefficient can arise when the mobilities of penetrants in the two molecular environments described by the Henry's law and Langmuir isotherm terms are not equal (Paul and Koros, 1976; Koros et al., 1976; Koros and Hopfenberg, 1979).

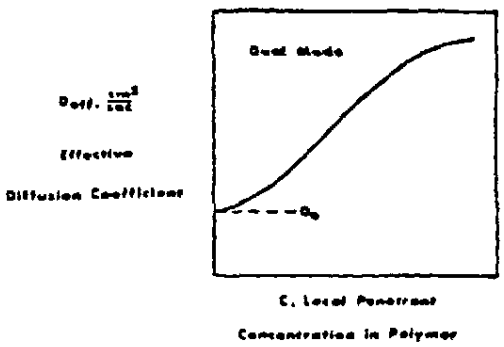

Figure 2. Schematic representation of the typical form of the effective diffusion coefficient, D_{eff}, vs. local concentration of penetrant for dual mode systems in which the mobilities of components in the two modes are not equal.

Regions of localized lower density (frequently referred to as "holes") which are frozen into amorphous polymers as a result of incomplete volume relaxation during quenching of the polymer from the rubbery to the glassy state are presumably the locus of the Langmuirian sorption mode (Barrer et al., 1958; Michaels et al., 1963a,b; Koros and Paul, 1978a–c). The diffusional mobility of species in the Langmuirian mode appears to be significantly lower than the corresponding diffusional mobility of penetrants in the Henry's law mode for most penetrants (Koros et al., 1977).

The Dual Mobility or Partial Immobilization Model. The flux resulting from Fickian diffusion of penetrants in the two sorption modes, C_D and C_H, can be described by eq 3 using an effective diffusion coefficient or, alternatively, by eq 4, which recognizes explicitly the possibility of different diffusivities and gradients of penetrant dissolved in the matrix and dispersed in "holes" (Paul and Koros, 1976). Specifically

$$N = -D_D \frac{\partial C_D}{\partial x} - D_H \frac{\partial C_H}{\partial x} \tag{4}$$

where D_D and D_H are the constant diffusion coefficients at each temperature associated with diffusion of penetrant sorbed in the Henry's law mode (C_D) and in the Langmuir mode (C_H) respectively. Equation 4 is the basis of a generalized transport model generally referred to as either the partial immobilization model or the dual mobility model.

Since eq 3 and 4 describe identical fluxes, explicit relationships can be derived for D_{eff} in terms of the various dual mode kinetic and equilibrium parameters if the assumption of local equilibrium (equality of chemical potentials) of components in the two modes at planes of constant x in the membrane is made. The resulting expression is given by eq 5 (Paul and Koros, 1976).

$$D_{eff} = D_D \frac{\left[1 + \dfrac{FK}{(1 + \alpha C_D)^2}\right]}{\left[1 + \dfrac{K}{(1 + \alpha C_D)^2}\right]} \tag{5}$$

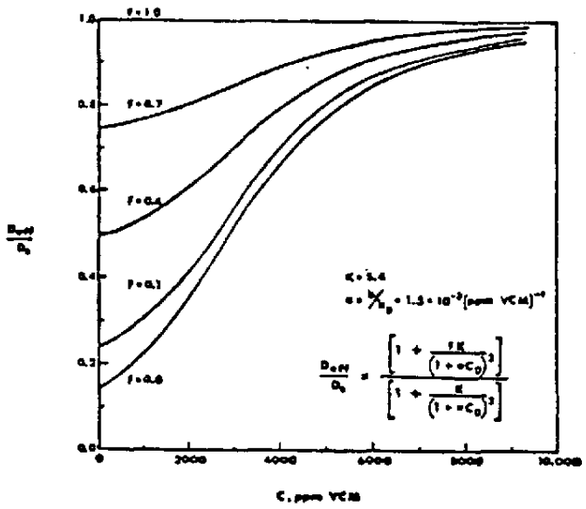

Figure 3. Calculated dependence of the ratio of D_{eff}/D_D using dual mode sorption parameters characteristic of VCM in PVC at 30 °C for values of $F = D_H/D_D$ between 0 and 1.0.

The kinetic parameter $F = D_H/D_D$ characterizes the mobility of species in the Langmuirian mode relative to species in the Henry's law mode. If $F = 1.0$ (no immobilization), D_{eff} is a true constant equal to D_D. On the other hand, if $F = 0$ (total immobilization of Langmuirian species, $D_H = 0$) the effective diffusion coefficient can be a strong function of C_D, depending on the values of C_H', b, and k_D appearing in eq 5 as K and α. The range of behavior of D_{eff} is shown in Figure 3 for hypothetical cases where $K = 5.4$ and $\alpha = 1.5 \times 10^{-3}$ ppm^{-1} for values of F between 0 and 1.0.

For the migration of trace additives C_D is *extremely* small and, therefore, eq 5 simplifies to

$$D_{eff} = \frac{D_D(1 + FK)}{(1 + K)} \tag{6}$$

Under these conditions D_{eff} is effectively a constant and corresponds to the zero concentration intercepts in Figure 3 for each value of F.

Over a significant concentration range, D_{eff} varies considerably as is demonstrated by eq 5 and Figure 3. It is often useful, therefore, to define a mean diffusion coefficient which corresponds to the concentration interval in question. A mean diffusion coefficient, $\bar{D}$, is typically defined as (Crank and Park, 1968)

$$\bar{D} = \frac{\int_{C_1}^{C_2} D_{eff}\, dC}{C_2 - C_1} \tag{7}$$

Substituting eq 5 into eq 7 and making the common assumption of local equilibrium between species in the two modes, i.e., $C = C_D + C_D K/(1 + \alpha C_D)$ (Vieth and Sladek, 1965; Paul and Koros, 1976), one finds

$$\bar{D} = D_D \frac{\left[1 + \dfrac{FK}{(1 + \alpha C_{D_2})(1 + \alpha C_{D_1})}\right]}{\left[1 + \dfrac{K}{(1 + \alpha C_{D_2})(1 + \alpha C_{D_1})}\right]} \tag{8}$$

The above form of $\bar{D}$ will be discussed further in the context of VCM migration from PVC in the following case study.

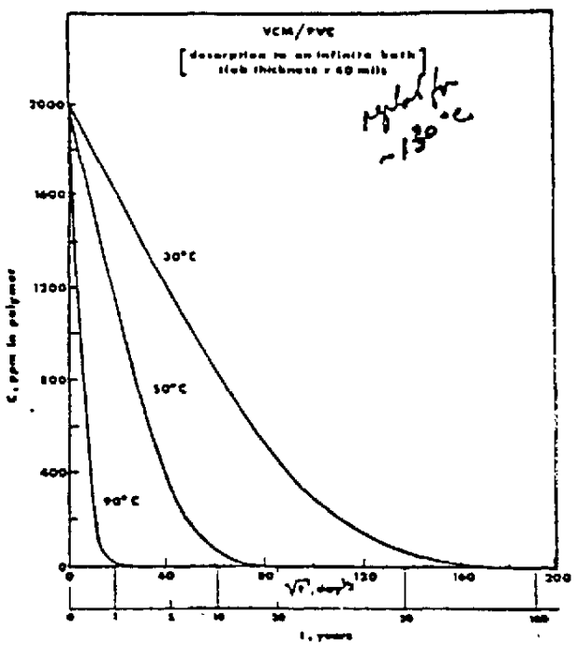

Figure 4. Calculated kinetics for VCM migration from PVC between 30 and 90 °C.

Case Study Describing Factors Affecting Migration

Temperature. The migration of vinyl chloride monomer (VCM) in poly(vinyl chloride) (PVC) has been studied actively since 1974 (Berens, 1974a,b; Berens and Hopfenberg, 1977; Berens, 1979; Haefner and Hughmark, 1978). Berens has measured the temperature dependence of mean diffusion coefficients of VCM in PVC microspheres for concentrations up to several thousand ppm of VCM. The reported values can be used directly over this concentration range to predict the temperature dependence of the migration rate of VCM from a PVC sheet of given thickness. These calculations have been performed using graphical solutions for constant diffusion coefficients (Crank, 1975) for diffusion from a 40-mil PVC sheet into an infinite well-stirred bath. The calculated results are presented in Figure 4 over the temperature range from 30 to 90 °C. Since Berens measured mean diffusion coefficients, and the migration calculations were performed within the same concentration interval (0 to 2000 ppm), the mean coefficients can be used in the calculations without requiring assumptions regarding the specific nature of the concentration dependence of D_{eff}.

The highly activated nature of the diffusion process is reflected in Figure 4. In all cases the migration is slow; at 30 °C, it takes more than 50 years to reduce the average concentration in the sheet to a value of less than 1 ppm, while at 90 °C, the corresponding diffusion time is reduced to approximately 1 year.

Bath Size. One of the most severe limitations to migration can be imposed by the relative volumes of the polymer and the extracting bath. Using values for the distribution coefficient of VCM between PVC and H_2O derived from Berens' data (Berens, 1975) migration calculations have been performed for various ratios, β

$$\beta = \frac{V_B}{V_p K^*} \tag{9}$$

where V_B is the bath volume, V_p is the polymer volume,

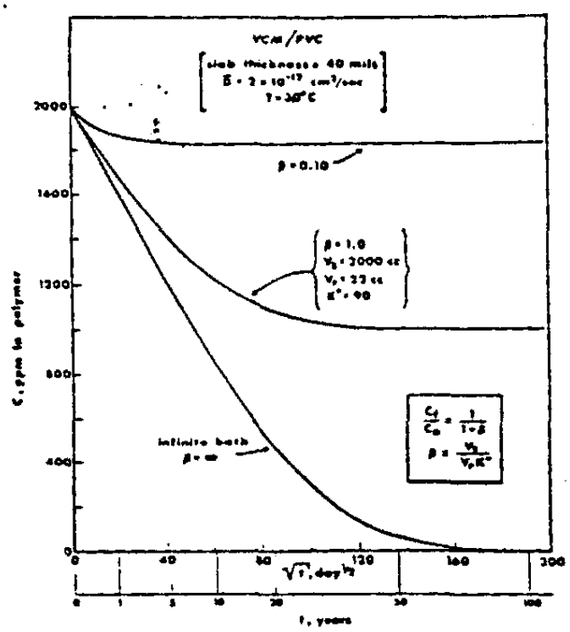

Figure 5. Calculated kinetics for VCM migration from PVC at 30 °C illustrating finite bath effects on apparent trapping of residual VCM.

and K^* is the distribution coefficient equal to C/C_B. The asymptotic value of K^* at low concentrations becomes quite large and $K^* = 90.1$ was used in the calculations here as the most realistic case. Berens has measured a mean diffusion coefficient equal to 2×10^{-12} cm²/s for sorption of VCM in PVC over the concentration interval 0–2000 ppm at 30 °C. This mean diffusion coefficient was used with the graphical solutions mentioned above (Crank, 1975) for release kinetics into a well-mixed finite bath. The plots presented in Figure 5 compare residual concentration curves for $\beta = 0.1$, 1.0, and infinity. The curve for $\beta = 1.0$ corresponds to a 22 cm³ sheet of 40 mil thick PVC suspended in 2000 cm³ of well-stirred H_2O for the extrapolated distribution coefficient $K^* = 90.1$ under the assumed conditions. Even for rather large bath-to-polymer volume ratios, apparent trapping of residual penetrant will be observed. Specifically, if a value of $\beta = 100$ were used corresponding to immersion of 22 cm³ of 40 mil thick PVC in 53 gal of water, a residual or limiting concentration of 20 ppm would appear to be inextractable from a sheet initially containing 2000 ppm. The ratio of initial to final concentration in the polymer is dependent only upon the parameter β, and although the kinetics may be affected slightly by extracting over different concentration ranges (e.g., 2000 to 0 ppm, 2000 to 1000 ppm, 2000 to 1800 ppm, or even 2 to 0 ppm) the residual concentrations can be shown to be approximated reasonably well by curves such as those in Figure 5 using the constant value of $D = 2 \times 10^{-12}$ cm²/s. The final concentration, C_f, divided by the initial concentration, C_0, is given by

$$C_f/C_0 = 1/(1 + \beta) \tag{10}$$

Therefore, extraction of 22 cm³ of PVC containing 2 ppm by a 53 gal bath would result in a limiting concentration of 20 ppb in the VCM-containing sheet and a maximum concentration of 0.30 ppb in the water after 60 years. In all cases migration rates are slow and a half-time in excess of 7 years was calculated for the limiting case of the infinite bath.

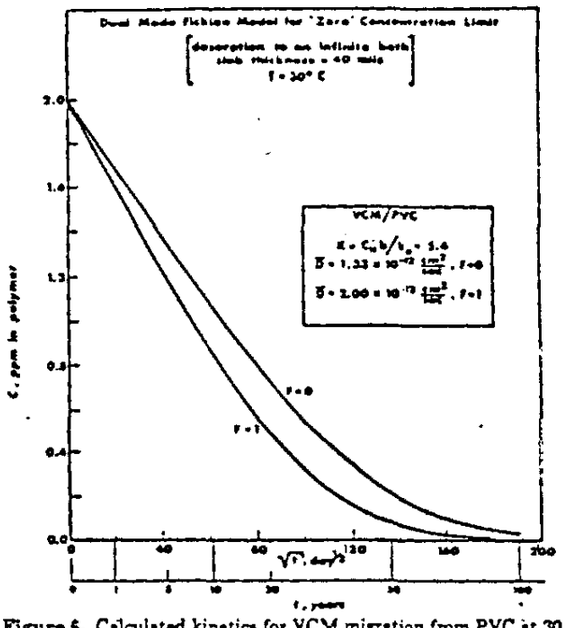

Figure 6. Calculated kinetics for VCM migration from PVC at 30 °C illustrating the effects of the ratio $D_H/D_D = F$.

Special Case of the Mean Diffusion Coefficient for Sorption and Desorption. Equation 8 simplifies somewhat for the case corresponding to integral sorption where $C_{D_1} = 0$. The corresponding expression for the mean diffusion coefficient for an integral sorption experiment involving generalized dual-mode sorption and transport in such a case is

$$D = D_D \frac{\left[1 + \dfrac{FK}{(1 + \alpha C_{D_2})}\right]}{\left[1 + \dfrac{K}{(1 + \alpha C_{D_2})}\right]} \tag{11}$$

A corresponding form for desorption when $C_{D_2} = 0$ with $C_{D_1} \neq 0$ results from substitution in eq 8. A value of D_D (which is by definition concentration independent) can be calculated if a value of F is specified or known for systems conforming to dual mode sorption with measured values of K and α. Berens has demonstrated that a dual mode sorption isotherm describes the low concentration region of VCM/PVC sorption (Berens, 1979). The Berens study provides a value of $K = 5.4$ and $\alpha = 1.5 \times 10^{-3}$ (ppm⁻¹). Since a value of F had not been measured for this system, the migration kinetics corresponding to the limits $F = 1$ and $F = 0$ have been calculated for equilibrium parameters $K = 5.4$ and $\alpha = 1.5 \times 10^{-3}$ ppm⁻¹ and are presented in Figure 6. The values of D_D used in Figure 6 were calculated from the measured values of D, C_{D_2}, K, and α reported by Berens for a total concentration, C, of approximately 2000 ppm of VCM using eq 11 with the two assigned values of F (0 and 1.0). The zero concentration limit of $D_{c\to 0}$ was subsequently calculated from eq 6 and used to prepare Figure 6. Once again, infinite bath conditions were assumed and the graphical solution to the diffusion equation given by Crank was used to perform the calculations.

Although total immobilization of Langmuirian species significantly retards migration, complete evacuation of penetrant from the polymer is achieved for both limiting

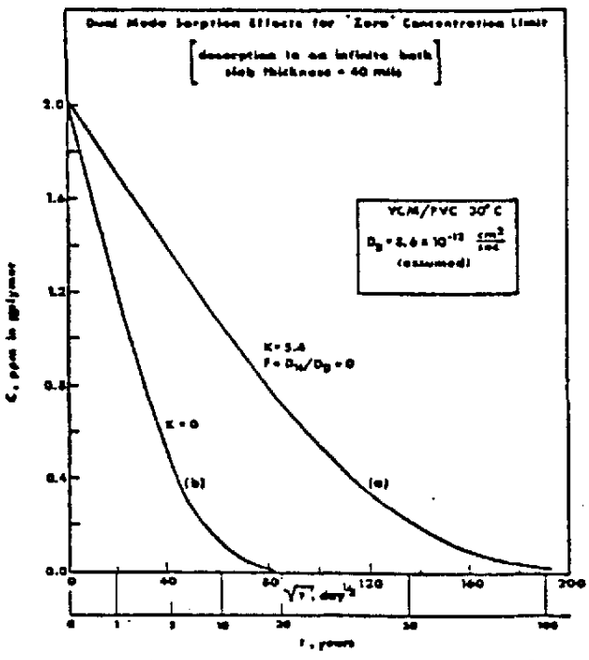

Figure 7. Calculated kinetics for VCM migration from PVC at 30 °C illustrating the effects of eliminating the Langmuir capacity ($K = 0$) for the limiting case of $F = 0$.

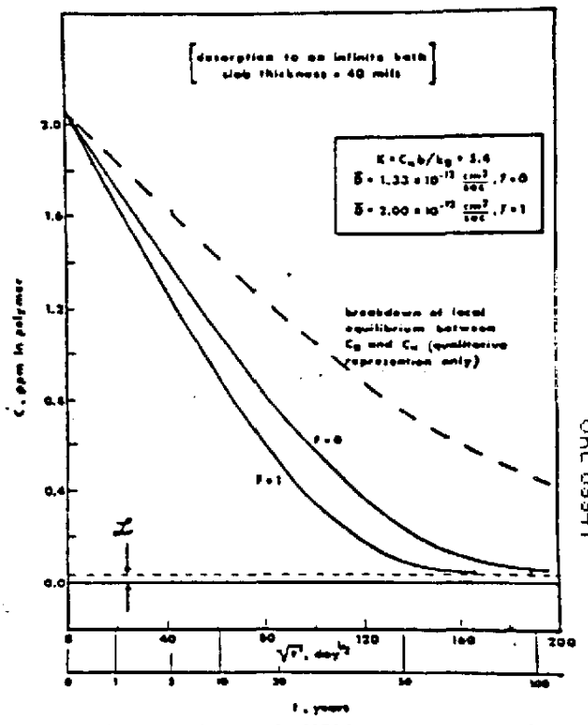

Figure 8. Migration kinetics for VCM extraction from PVC at 30 °C illustrating the effects of a hypothetical morphologically entrapped component, $\mathcal{L}$. The dashed line qualitatively illustrates the protraction of extraction of VCM which would occur if the assumption of local equilibrium is not satisfied.

cases at sufficiently long times. Clearly, the effect of bath volume on release kinetics is much more dramatic than "immobilization" per se. "Zero migration" is not consistent with the assumptions underlying common dual mode sorption regardless of the degree of "immobilization" of Langmuirian species. The effect of the Langmuirian sorption mode per se on migration kinetics is explored further in Figure 7. The total immobilization limit ($F = 0$) has been arbitrarily selected in preparing curve (a) of Figure 7. Clearly, other values of F could be treated as well. The $K = 0$ limit (Henry's law sorption) gives rise to much more rapid desorption than for $K > 0$ since the mean diffusion coefficient corresponding to eq 11 is equal to D_D if $K = 0$. For all values of $K > 0$, $\bar{D} < D_D$ and desorption is retarded, however total evacuation of the sample is eventually predicted given times which, although noninfinite, are indeed extraordinarily long.

Multi-Mode Sorption Involving Relaxation of the Local Equilibrium Criterion between the Various Sorption Modes. If one postulates that an additional sorption mode exists, say "$\mathcal{L}$", corresponding to irreversibly bound or "locked-in" sorbate (Gilbert, 1978), then the relationship between C, the concentration of all sorbed species, and the individual components is

$$C = C_D + C_H + \mathcal{L} \tag{12}$$

This postulate becomes a self-fulfilling prophecy in that now the concentration, $\mathcal{L}$, of species originally present in the polymer will persist indefinitely (by definition) regardless of the extraction conditions. These effects are shown graphically in Figure 8 assuming that a 40-mil PVC sheet containing a total concentration of originally sorbed species of 2.03 ppm desorbs into an infinite sink. The calculations reveal that the results of Figure 7 are obeyed nearly identically; however, the additional component, $\mathcal{L}$ = 30 ppb, by definition, remains in the polymer. Morphological entrapment of trace amounts of unpolymerized monomer between crystalline regions or within crystal defects has not, however, been demonstrated experimentally although significant retardation of extraction has been noted (Cope and Comyn, 1979). True equilibrium in the system would only, in principle, be achieved after the defects have healed or after impurities have been excluded by slow recrystallization.

If the Langmuirian and Henry's law species are not in local equilibrium, an additional retardation will result due to the kinetic limitation retarding equilibration between Henry's law and Langmuirian modes. Completely irreversible sorption would lead to "trapping"; however, any reasonable nonzero rate of transfer between "holes" and matrix would be achieved within some academically long time scale of desorption. These notions have been treated for the case of the total immobilization model ($F = 0$) (Tshudy and von Frankenberg, 1973). The dashed plot in Figure 8 qualitatively reflects the direction of the anticipated effect.

Conclusions

The migration rates of small molecules from glassy polymers are exceedingly low. In the absence of irreversible entrapment or binding of a fraction of the sorbed population, complete evacuation of sorbed species to an infinite bath must occur. The observation of a zero-migration rate can result from non-infinite bath conditions, especially for migrant molecules which partition strongly toward the polymeric phase. Moreover, the time-scale of predicted total extraction of species in many realistic cases is of the order of 100 years; therefore, detection limitations can compromise explicit characterization of these inordinately protracted desorption processes. It is, therefore, difficult to separate experimentally truly zero migration from situations corresponding either to finite bath condi-

tions or analytical detection limitations related in part to the staggering values of the predicted extraction times. The wealth of recent literature data in support of the dual-mode model for sorption and transport suggests, however, that the finite bath realities, boundary layer resistances in the extracting phase, and the time scale of desorption per se, are very likely to be responsible for apparent "zero" extraction results.

Acknowledgment

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Sperm Whale Oil Replacements from Halogenation of Jojoba Oil

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Kinetics of the chlorination and bromination of jojoba oil has been studied at different temperatures, solvents, and operating conditions. Chlorination follows first-order kinetics in double bond concentration with an activation energy of −11 kcal/mol. Solvents with high dielectric constant increase the rate of reaction. Bromination involves a two-step mechanism with one or two bromine molecules per double bond; up to 0.2 s reaction time the rate is first order in each of the reagents, and afterwards it is first order in the double bonds and second order in bromine. Faster rates are obtained lowering the temperature and/or increasing the dielectric content of the solvent. The chlorinated derivative improves the load carrying capacity of the lubricating oil.

The purpose of this work was to study the influence of operating variables on the kinetics of the reaction between jojoba oil and chlorine or bromine. Previous work on the sulfur halogenation of the oil has shown that the straight halogenated derivative may have value as a lubricant additive (Wisniak and Hanoch, 1975, 1978).

Jojoba is an evergreen bush of the *Buxaceae* family that grows in semi-desertic areas and yields a nut that contains about 50% of an oil composed mainly of monoesters of the C_{20} and C_{22} alcohols and acids, with two double bonds per chain. Details of the economic potential and chemical properties and technology have been recently summarized (National Academy of Sciences, 1977; Wisniak 1977). Jojoba oil and its derivatives have been shown to replace sperm whale in all its uses and also as a source material for new derivatives. Commercial plantation of the shrub is developing at a growing pace and large quantities of oil should be available within ten years.

Halogenated fatty materials find extensive use in the preparation of quaternary compounds, antirotting, flame proofing, and fungicide additives (Sonntag, 1963). Brominated vegetable oils have long been used as a weighting oil in carbonated beverages.

The reaction between halogens and organic compounds has been extensively studied, and the literature is well covered by Poutsma (Kochi, 1972) and De La Mare and Bolton (1966). Elementary fluorine normally reacts in a violent manner with olefins and leads to charred products. The other halogens can add to unsaturated compounds by heterolytic processes.

The addition of halogens to a double bond features a number of anomalies including high reaction orders, negative temperature coefficients, and a strong dependence on the nature of the solvents (De La Mare and Bolton, 1966). Chlorination of olefins in nonpolar media has been explained by ionic and free-radical reaction mechanisms (Poutsma, 1965); strong evidence indicates the possibility of a radical mechanism in the case of bromination (Sergeev and Sergeev, 1972).

According to Barnford and Tipper (1973), the rate of addition to a double bond is increased by the proximity of an electron donor group and decreased by an electron-

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SAFETY DEPARTMENT

10/70

cc: Mr. F. F. Hoy

Firestone
Safe Operating Procedure

THE FOLLOWING PROCEDURE HAS BEEN DRAWN UP FOR YOUR PROTECTION AND MUST BE OBSERVED.

GENERAL SAFETY RULES FOR ALL EMPLOYEES

The following rules have been drawn up for your protection and must be observed:

1. Keep your mind on the job at all times and give your work your entire attention.
2. If you feel ill or in such condition as to interfere with your work, report at once to your Supervisor or Foreman.
3. Cleanliness is a must. Do not allow oil, grease or other refuse to gather on machines or floor. Good housekeeping must be observed at all times.
4. All cuts and bruises should be treated at the plant Dispensary. Be sure to notify your Supervisor or Foreman if you cut or bruise yourself.
5. "Horseplay" will not be tolerated.
6. DO NOT REMOVE ANOTHER PERSON'S RED TAG FROM EQUIPMENT OR MACHINERY OR START WITHOUT PROPER AUTHORIZATION WHEN TAGGED!!!
7. Employees are encouraged to wear safety shoes at all times.
8. Safety glasses or other appropriate eye protection should be worn when needed.
9. Check your equipment daily and promptly report any unsafe conditions to your Foreman or Supervisor.
10. Show the proper respect for other employees. Be concerned with their safety as well as yours.
11. Gambling and/or the use of intoxicants will not be permitted.
12. Smoking is permitted only in areas designated as such.
13. Care should be taken when using compressed air. Do not point nozzle at yourself or any other person. Never use compressed air to blow dust from your clothing or hair.
14. No wrist watches, rings or loose clothing should be worn.
15. Care should be used when moving hastily throughout your own area or the plant proper. Refrain from running.
16. Employees are required to learn the specific safety procedure which applies to the job assignment.
17. Know the proper handling of chemicals, solvents, flammables, or other hazardous materials. Check with your supervisor if you are not certain.

SAFETY DEPARTMENT
2/68

CERTIFICATE OF SERVICE

The undersigned hereby certifies that he caused a copy of the foregoing Answers of Defendant Firestone Tire & Rubber Company to Plaintiffs' First Set of Interrogatories to be served on all defense counsel of record by United States Mail, this 25th day of April, 1980, as follows:

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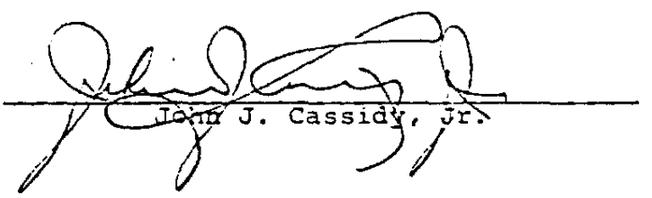

John J. Cassidy, Jr.

Stauffer Chemical Company
PLASTICS DIVISION
Sales Office: Westport Connecticut Telephone (203) 222-3000
Products Laboratory, P.O. Box 320, Delaware City, DE Telephone (302) 834-7455

SCC-40

PVC COPOLYMER DISPERSION RESIN

SCC-40 is a vinyl chloride - vinyl acetate copolymer plastisol dispersion resin with low paste viscosities, excellent heat stability and rapid fusion characteristics. SCC-40 has been successfully used in mechanically frothed foam, solid rug backing, laminating, fabric coating, flocking adhesives and many other applications.

TYPICAL RESIN PROPERTIES:

ASTM Classification	ASTM D-1755-66	D-334
Appearance	Visual	White Powder
Relative Viscosity	1% in cyclohexanone, 25°C	2.35
Inherent Viscosity	ASTM D-1243 (A)	1.04
Volatiles	SCC C-2, 1 hr. @ 105°C	0.4%
Apparent Density	ASTM D-1895A	14 lb./cu. ft.
Methanol Extract	ASTM D-2222-66	1.6%
Specific Gravity	ASTM D-792	1.39
Bound Vinyl Acetate	SCC C-103	4.8%

TYPICAL PLASTISOL[1] PROPERTIES:

Brookfield Viscosity	SPI-VD-T1, 2 rpm aged 2 hrs.	65 poise
	20 rpm aged 2 hrs.	60 poise
Severs Viscosity	SPI-VD-T2, 20 psig aged 24 hrs.	221 poise
	60 psig aged 24 hrs.	246 poise
	100 psig aged 24 hrs.	187 poise
Fineness of Dispersion	SPI-VD-T10	50 microns
Gelation Temperature	SPI-VD-T18	148°F
Air Release	SCC C-15, Height of Rise % Increase	800%

TYPICAL FUSED FILM[2] PROPERTIES:

Haze	ASTM D-1003	13%
Heat Stability	350°F oven aging, initial discoloration	20 min.
Tensile Strength	ASTM D-882	3000 psi
Ultimate Elongation	ASTM D-882	500%
100% Modulus	ASTM D-882	850 psi

Notes:
1. 500 gm. resin and 300 gm. DOP, Hobart N-50 mixed 5 min. @ No. 1 speed and 10 min. @ No. 2 speed, undeaerated.
2. 20 mils containing 60 phr. DOP and 2 phr. Mark 29, deaerated plast sol. fused 5 min. @ 350° F on Ferro Plate.

VALUES PRESENTED ARE TYPICAL AND INTENDED TO SERVE AS GUIDES ONLY

Stauffer CHEMICALS

Stauffer Chemical Company
PLASTICS DIVISION

Sales Office: Westport Connecticut/Telephone (203) 222-3000
Products Laboratory: P.O. Box 320, Delaware City, DE/Telephone (302) 834-7458

SCC-676

PVC HOMOPOLYMER

SCC-676 is a medium molecular weight PVC homopolymer suitable for both rigid and flexible applications. SCC-676 is exceptionally low in gels and finds wide acceptance in calendered film and sheeting. Also SCC-676 because of its low fines level is suitable for rigid dry blends where bulk handling requires good dry flow.

Suggested Applications: SCC-676 is useful in both flexible and rigid uses. Successful applications include:

- Calendering
- Wire and Cable
- Pipe - both NSF & DWV
- Electrical Conduit
- Blown Flexible Films

URL 09948

TYPICAL RESIN PROPERTIES:

ASTM Classification	ASTM D-1755	GP4-15453
Relative viscosity	1% in Cyclohexanone, 25°C	2.24
Inherent viscosity	ASTM D-1243(A)	.95
Volatiles	SCC C-2, 15 min. @ 105°C	0.25%
Apparent Density	ASTM D-1895	0.50 gms/ml.
Sieve Analysis	ASTM D-1921(B)	
Retain on 40 mesh		none
Retain on 100 mesh		40%
Retain on 200 mesh		95%

VALUES PRESENTED ARE TYPICAL AND INTENDED TO SERVE AS GUIDES ONLY

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF MACOMB

KENNETH GRIMM, Personal Representative of the ESTATE OF HELEN GRIMM, Deceased,

Plaintiff,

vs.

FORD MOTOR COMPANY, a foreign corporation, et al,

Defendants.
_____/

Case No. 83-2872 NO

Hon. Raymond R. Cashen

PROOF OF SERVICE

STATE OF MICHIGAN)
)ss.
COUNTY OF OAKLAND)

Kathleen Wilson, being duly sworn, deposes and says that on the 18th day of December, 1984, she served copies of Defendant Stauffer Chemical Company's Answers to Plaintiff's First Set of Interrogatories and Proof of Service upon:

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by placing the documents in fully addressed, stamped envelopes and by depositing the envelopes in a United States Mail Receptacle.

Kathleen Wilson

Subscribed and sworn to before me
this 18th day of December, 1984.

Patrick K. Rode, Notary Public
Oakland County, Michigan
My commission expires: 1/9/88

URL 09949