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DEVELOPMENT OF A HYDROPHOBIC SUBSTANCE TO MITIGATE PAVEMENT ICE ADHESION



Municipal Environmental Research Laboratory
Office of Research and Development
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Cincinnati, Ohio 45268

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DEVELOPMENT OF A HYDROPHOBIC SUBSTANCE
TO MITIGATE PAVEMENT ICE ADHESION

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FOREWORD

The Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimony to the deterioration of our natural environment. The complexity of the environment and the interplay between its components require a concentrated and integrated attack on the problem.

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The program described here was undertaken to investigate the feasibility of the use of hydrophobic substances on highways to reduce ice adhesion. Such a coating could reduce or eliminate the possibility of pollution of ground water by currently used deicing chemicals and the multi-billion dollar yearly cost of automotive frame, bridge deck and highway surface deterioration caused by such chemicals. The feasibility of this approach is demonstrated and specific recommendations are presented to optimize the concepts developed in this program.

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ABSTRACT

This research was directed specifically to the development of hydrophobic material coatings for highway surfaces to reduce the adhesion of ice on such surfaces. In addition to the technical goal of functional usefulness, other primary ground rules included:

- Cost effectiveness as compared to conventional de-icing methods
- Minimum pollution of the environment during application and subsequent runoff water exposure
- Employment of standard road coating equipment and techniques
- Consideration of only hydrophobic materials as opposed to conventional materials used to melt ice and snow
- Maximum coating life permitting, as a goal, once-per-season application
- Minimum corrosiveness to automotive frames and bridge substructures, minimum deleterious effect on highway surfaces, and maximum safety in use
- Investigation of only existing commercially available materials with no synthesis of new compounds

The following four-phase program was conducted:

Phase I. Identify through literature searches, vendor contacts and consultants, as many commercial products as possible which might meet the requirements.

Phase II. From the list compiled in Phase I, select materials for laboratory characterization and functional testing and conduct such tests.

Phase III. From the data of Phase II

- Rate materials and material combinations on the basis of the ground rules listed above

- Establish selection criteria for road testing
- Consider application techniques and calibrate the application equipment

Phase IV. Conduct highway and parking lot evaluation of three formulations from Phase III.

Of the three coatings applied and tested in Phase IV (the applied cost for these three ranged from 8¢/m² to 69¢/m²), two demonstrated considerable promise. These two exhibited satisfactory traction on wet roads, produced very low runoff water contamination and demonstrated a significant reduction in ice adhesion. However, they were inadequate in meeting the goal of a season-long effective life. The components of these two formulations comprise three classes of materials which, if optimized, should yield effective coatings.

In addition, several test methods were developed by BBRC that should prove useful in this and other fields.

This report is submitted in partial fulfillment of BBRC Project Number 2075 under the sponsorship of EPA Contract 68-03-0359. Work was completed as of July 1975.

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Chapter 1

RECOMMENDATIONS AND SUMMARY

THE PROBLEM

The use of salt (and other deicing chemicals such as calcium chloride, urea and glycol mixtures) and sand to remove ice from highway surfaces in winter is the commonly accepted method used in the United States and Canada. For example, three years ago, usage was cited (Ref. 4*) as over nine billion kilograms (ten million tons) per year in the U. S. Application rates are also cited (Ref. 4) as high as 14,000 kg per lane km (25 tons per lane mile) in some areas per season. While other deicing chemicals present somewhat fewer problems in vehicle damage and bridge deck/highway surface deterioration, they are more expensive, are less effective and some increase the oxygen demand of runoff water in the areas where they are used. Thus, the primary objections to the use of chemical deicers are:

- Direct environmental impact.
- Indirect environmental effects including vehicle corrosion and pavement and bridge deck structure damage, with consequent safety hazards and considerable economic loss in all cases.

The specific problem to which this report is addressed is the development of a hydrophobic substance to mitigate the adhesion of ice to pavement as an alternative to deicing chemicals. The factors involved in evaluating this concept are outlined below.

Economics. The coating used to reduce ice adhesion must be economically justified. Considering only salt replacement, elimination of vehicle damage and reduction of highway structure damage, the latest data (see Chapter 5, Section 6 of this report) indicate that the cost of a coating applied *once-per-season* must be less than about 28¢/m². Note that reduction

* For reasons discussed later, cited references in this report are not necessarily numbered in the same order as they first appear in the text.

of pavement damage and virtual elimination of direct environmental impact costs are not considered. These latter effects could substantially increase the above allowable cost.

Safety. The principal safety aspects to be considered are toxicity, flammability and other potentially hazardous properties of the coatings. Such considerations apply equally to the formulation before application, conditions present during application to the pavement and the dried film on the highway. Storage requirements are defined by Occupational Safety and Health Administration (OSHA) flammability and toxicity requirements. Hazards during application are defined by the State of Colorado version of EPA Regulation No. 7 (attached as part of Reference 62 in Appendix B). The dried coating hazards are discussed in Chapter 4 of this report. It was also hypothesized that oleophilic (oil attracting) coatings might create a skid hazard.

Environmental Impact. The environmental impact of currently used deicing materials (primarily inorganic chlorides) is known to be severe. Current estimates (Ref. 70) indicate annual damages to water supplies, health, vegetation and utilities in excess of \$100 million. In addition to these damages, any alternative coating must be evaluated in terms of water solubility, application hazards and personnel hazards. In relatively thin coatings (say, 0.01 cm), inert hydrophobic coatings overcome most of the above impact problems. As shown in this report, application hazards constitute the one area requiring further work.

Coating Effectiveness. Although effectiveness is certainly the most basic criterion for an ice-release coating, it is perhaps the most difficult to define in quantitative terms. Deicing chemicals either do or do not melt snow and ice at a given temperature whereas ice release is a function of temperature, shear rate, substrate roughness, applied force vector and other factors. The approach in this work was to demonstrate hydrophobicity and to rank shear adhesion force for the coatings by laboratory test, followed by real-life field testing.

In the same way, coating effective life (involving, among others, factors of coating/substrate chemistry, coating-to-substrate adhesion, abrasion resistance, shear strength, traffic patterns and densities and the presence of salt, sand, dirt, etc.), was evaluated by actual highway wear testing.

To be effective, the coating must not create, in itself, the very condition it is intended to alleviate, namely, slippery roadway surfaces. In fact, demonstration of this property was found to be *the* primary concern of the Colorado Department

of Highways and a large amount of friction-coefficient data was obtained for the coatings on asphaltic and concrete surfaces.

Finally, stability in the presence of ultraviolet radiation (sunlight) and oxygen is required. This was evaluated by both laboratory and outdoor exposure methods.

Potential Pavement Damage. In order to avoid potential pavement damage, strong acidic or basic water solutions must be avoided, especially on concrete, and any application-phase solvents must not degrade asphaltic surfaces. It was also demonstrated in this program that oleophilic materials badly degrade asphaltic surfaces. To be even more economically attractive than deicing chemicals, hydrophobic ice-release coatings should actually protect highway surfaces from water penetration and subsequent freeze-thaw damage.

RECOMMENDATIONS

As a result of this program, two coating formulations (exact formulae are given in Chapter 5 of this report), have been identified as showing considerable promise as semi-permanent, hydrophobic, road coatings with reduced ice adhesion. They are:

- A modified (no pigment) Federal Specification TT-P-115D traffic paint containing a room-temperature-curing silicone rubber (Dow Corning DC732) as a release agent. Formulation is identified as A in the Phase IV road test evaluation.
- A silicone resin waterproofing compound (Dow Corning DRI-SIL-73) combined with the same silicone rubber as above and identified as formulation C in Phase IV.

One major achievement in this program was the discovery of a method for stabilizing the highly reactive silicone rubber in a fluid solution for spraying.

As applied to roadway surfaces in dried films about 0.01 cm (0.004-inch) thick, these two coatings:

- Show greatly reduced ice adhesion until physically worn away.
- Have an applied cost of about 40¢/m² and 69¢/m² (33¢ and 58¢/yd²) as of December, 1974.

- Show excellent stability to weathering.
- Exhibit total water-soluble material equivalent to a maximum of about 18 grams per lane meter (64 pounds per lane mile) (see Section 9.3 of Chapter 4).
- Have low pollution impact.
- Show negligible corrosiveness and zero road damage.
- Can be applied with standard spray truck techniques.
- Require a maximum lane closure time of one to three hours depending on ambient temperature.
- Do not greatly reduce rubber-to-road friction coefficient.

The deficiencies of these formulations are:

1. An estimated effective wear life of only 150,000 to 300,000 vehicle passes (one to two months on the tested roads) for the thickness employed.
2. The release of flammable vapors, mostly VMP naphtha, into the atmosphere during application.

In view of the above and other program data, the following specific recommendations are made:

- Neither formulation fully meets the target goals of a material that easily releases ice and can be applied only once per season to existing roadway surfaces. However, Formulation A above should be useful and nearly invisible on low traffic areas such as concrete driveways and sidewalks, while Formulation C above, applied at perhaps twice the rate used here, should release ice from and help protect asphalt-surfaced bridge decks. Other processes for surface sealing, such as in Reference 39, are estimated to cost more than five times as much.
- Both formulations should be optimized by variation of component ratios for minimum ice adhesion. In addition, the paint formulation contains components such as clays and other extenders that are probably hydrophilic and therefore harmful in this application. Variations of the basic paint formula should be tested.

- Future road tests should be conducted in more *consistently* colder regions.
- Laboratory ice adhesion testing should be performed on real substrates over a wider range of temperatures and shear rates than those used here (see Chapter 4, Section 4.2).
- Water-based emulsion systems coupled with elevated temperature curing should be studied. Slow evaporation and the primarily budgetary limitation of ambient temperature curing resulted in long lane-closure times and negative results for such systems in this study. However, such systems remain attractive from material cost and pollution-during-application standpoints.
- One ground rule of the present work was the application of the coatings to *existing* road surfaces. However, roads are *resurfaced* and incorporation of hydrophobic materials in the resurfacing mix should vastly extend the effective wear life. This should be investigated, especially with respect to Petroset AT and Viscospin-B (see Appendix B).
- In view of the current expense of highway repair, a separate study is also needed of the substrate protection afforded by hydrophobic materials applied *with* the asphalt or concrete.

INTENT OF REPORT

The purpose of this report is fourfold, namely:

- To describe the work conducted in sufficient detail to permit evaluation of the data by independent investigators.
- To explain the material selection/screening procedures and justify the conclusions reached.
- To record data which, while not directly applicable to the stated goals, might be useful in other work. Two examples are the tabulation of *unsuccessful* material combinations in Phase II and the citation of references, including some not specifically used in this report, as background material in this field.
- To present recommendations which appear to be a logical extension of this basic study.

SUMMARY OF REPORT

Since some sections of this report may be of only limited interest in some cases, such as theoretical discussions might be to those interested primarily in practical results, this portion summarizes the contents of the chapters presented herein.

Chapter 2, Theory. In this chapter are presented the basic theory of wetting, surface energy balance considerations as applied to hydrophobicity, work of adhesion as related to water and ice and practical modifications of theory applicable to this study.

Chapter 3, Phase I. In this description of the Phase I program work are included the results of a complete literature survey, a review of applicable test programs, a summary of all contacts made with vendors and other organizations and a review of the material candidates to be evaluated in the Phase II screening tests.

In tabular form are presented all the materials considered and the test/reject criteria employed. Tabulated are over 55 materials and material classes.

Chapter 4, Phase II. The laboratory and outdoor property screening of 33 materials are presented in this chapter. Included are contact angle data (a measure of hydrophobicity), infrared analyses and vendor-supplied data, ice adhesion test results, friction coefficient data for the coatings on asphalt and concrete, environmental hazard test results, ultraviolet-exposure and other degradation evaluations and highway wear test results. Also included are an extensive list of rejected materials and material combinations and the criteria employed.

Chapter 5, Phase III. Phase III, the application study, includes the following:

- Rating factors for 28 materials and the selection procedure used for the three highway-tested coatings.
- A summary of the exact composition for the coatings as applied.
- Material property (composition, density, etc.) data required to determine the bulk application rates desired.
- Formulae used to determine application-vehicle speeds required during application.

- A discussion of spray techniques, spray rate calibration and application-vehicle speed calibration.
- A summary of applied-coating-cost computations.
- A summary of environmental impact considerations.

Chapter 6, Phase IV. Evaluation of the three selected formulations on asphalt and concrete is discussed in Chapter 6. Presented are:

- Weather data
- Visual observations
- Qualitative skid data
- Ice release data

for three high-traffic-density areas, one low-traffic-density area and two zero-traffic-density locations.

Also included are:

- Wear life estimates
- Additional quantitative friction coefficient data
- Supplemental laboratory ice adhesion data
- Additional environmental impact test data

As required by the funding contract, the International System of Units is employed "except when the use of such units would obviously impair communication or reduce the usefulness of a report to the primary recipients" (Ref. 38). Two specific examples where such impairment would result are:

- Ice Adhesion Shear Force. The use of kg/cm^2 (as opposed to N/m^2) results in conveniently small numbers facilitating comparison of values

$$\text{kg/cm}^2 \times 9.8 \times 10^4 = \text{N/m}^2$$

- Surface Energy and Dispersion Energy. The use of ergs/cm^2 is so common in the literature that comparison would be difficult without retention of these units.

$$\text{ergs/cm}^2 \times 1.0 \times 10^{-3} = \text{N/m}$$

Finally, for one-time computations used in illustrative examples where only relative values are important (such as those in Paragraph 5.4.3, Chapter 6), conventional metric (though not SI units) are employed.

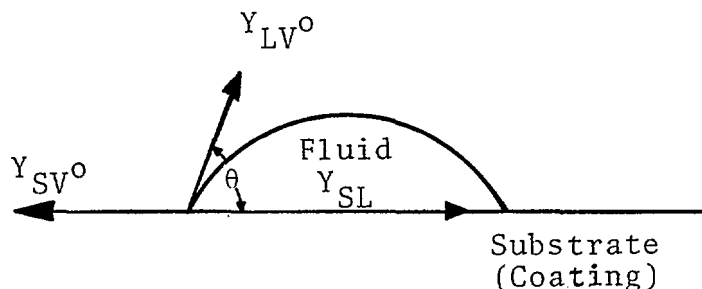
Chapter 2

THEORY

This chapter is concerned with the theoretical and applied surface physics and chemistry technology of wetting phenomena as specifically related to the objective of producing a water or ice repellant. The various parameters involved in wetting and the testing of wetting phenomena are discussed together with the chemical types desired for candidate screening. Theoretical studies from the literature survey of Phase I are then discussed. Finally, the derivation of surface energy values from laboratory contact angle measurements is reviewed.

1.0 BASIC AND APPLIED WETTING THEORY AND MODIFICATIONS

The basic theory of wetting, of which hydrophobicity is a specialized case, has been extensively treated in the literature (see References 3, 67, 41 and 2): The initial concept is shown in the sketch below. This shows the equilibrium forces for a drop of fluid on a flat substrate.



where

θ = Contact angle

γ_{SV}^0 = Interfacial free energy of solid/saturated vapor interface

γ_{LV}^0 = Interfacial free energy of liquid/saturated vapor interface (this is sometimes erroneously used as identical with liquid surface tension)

γ_{SL} = Interfacial free energy of solid/fluid interface

For equilibrium,

$$Y_{SV}^{\circ} - Y_{SL} = Y_{LV}^{\circ} \cos \theta \quad (1)$$

It is apparent that the smaller the left-hand side of the equation (which is related to the surface free energy of the solid coating), the greater is θ .

From Reference 3,

$$W_A = Y_S^{\circ} + Y_{LV}^{\circ} - Y_{SL} \quad (2)$$

where

W_A = The work of adhesion

Y_S° = The surface energy of the solid in vacuum

Substituting (1) in (2):

$$W_A = Y_S^{\circ} - Y_{SV}^{\circ} + Y_{LV}^{\circ} (1 + \cos \theta) \quad (3)$$

the so-called Dupré equation. The first two terms represent the decrease of the specific free energy of the solid when immersed in the saturated liquid vapor. This quantity can rarely be quantitatively determined, but however it is determined, it will be smaller the lower the specific free energy of the solid. Y_{LV}° is fixed for water, so the only other variable is θ .

From the above equations, if the work of adhesion is to be minimized:

- The surface free energy of the coating must be as small as possible
- θ should be as large as possible

From the above alone, for minimum adhesion it would therefore only be necessary that the coating consist of a single monolayer with a very low surface free energy, since such a surface also increases θ as is shown by Equation (1).

Many such molecular types are specified in References 3 and 41. However, the above simplified theory must be modified to include other important technical factors.

1.1 Practical Modifications of the Basic Theory

Solubility. The simplified theory does not account for solubility of the fluid phase in the substrate or coating. This

has been demonstrated in tests at BBRC. For example, low energy coatings have been tested upon which certain fluorochemicals (with $\gamma_{LV}^o = 18$ ergs/cm²) will not spread but upon which certain silicones (with $\gamma_{LV}^o = 23$ ergs/cm²) will spread. This explains the need for solubility testing of candidate coating materials in water and the practical difficulties of deriving surface energy from contact angle measurements (discussed in Section 3 below).

Long Range Forces. Most intermolecular forces are relatively short range (less than 10 Å, or about one monolayer). However, permanent and induced dipole forces (water is a strong dipole) have ranges of many monolayers. This explains the need for relatively thick coatings (greater than one micron, or 10,000 Å), to block the adhesive effect of these forces. This effect is discussed further in Section 2 below.

Roughness. The theory assumes perfectly flat surfaces. This is never true in reality. On rough surfaces, Wenzel's equation:

$$\cos \theta' = r \cos \theta \quad (4)$$

where

θ' = Contact angle observed

r = Ratio of *actual* surface area to apparent (envelope) surface area

θ = Contact angle on a smooth surface

applies. For example, should θ be 60° and r be 2.0, θ' will be 0° and total spreading will occur. Such phenomena have been observed and studied at BBRC. This is the explanation for the use of "rough" more "real-life" surfaces in the contact angle screening tests (Chapter 4).

1.1.1 Requirements of a Truly Hydrophobic Coating

From this brief (and in some respects superficial) examination of surface theory, a *truly hydrophobic coating must have*:

- Minimum specific surface energy
- A thickness of several thousand angstroms
- Virtually no solubility in water

- A contact angle as large as possible with a minimum value of 60° on a smooth surface

Equation (3) demonstrates why the work of adhesion is unlikely to ever be zero. The first two terms represent a positive value and the third term is always greater than zero (since $\theta = 180^\circ$ has never been observed in practice - see page 145 of Reference 3).

1.2 Applied Theory and Water/Ice Phobicity

The theory outlined above is now directed to the specific problem of water/ice phobicity of coatings applied to rough roadway surfaces.

Testing Considerations. The relationship between hydrophobicity and the corresponding property for ice has been treated in some detail in Reference 2, pages 46-77. Specific points to be considered in testing are:

- The tensile strength of ice itself decreases with increasing ice volume. This, together with the assumption that water will bead up on hydrophobic coatings, suggests the use of small ice volumes to measure the adhesive ice/coating shear strength.
- The number of factors indicated in the problem discussion in a previous section are also emphasized in Reference 2. This confirms the conclusion already reached that complete simulation of reality *in the laboratory* is not economically feasible and that only individual specific parameters can be treated experimentally.
- The surface free energy of ice is cited in Reference 2 as 109 ergs/cm^2 and of water as about 75 ergs/cm^2 . On this basis, ice should have lower work of adhesion. That it does not (see discussion in Reference 2) indicates the existence of long range forces discussed above.
- Some solid polymers exhibit no change in shear-release force of ice with repeated release while other hydrophobic materials (having equal or lower surface energies), such as the perfluorinated acids, show an increase with each release, indicating coating removal. The use of tough binders appears to be a logical approach. In binders, the effective hydrophobic groups can be exposed at the surface by preferential

density effects and maintained by wear of the coating. Experimental verification is obviously a necessity.

- In the conclusion of the Reference 2 article, the difficulty of simulating reality is again emphasized. The partial absorption of water by the coating and subsequent coating removal by cohesive failure during shear testing is stated as the major problem area.

1.2.1 Desirable Chemical Types

The surface free energy of a large class of materials is given in Reference 3. Molecular orientation is vital and the species predominating on the coating surface control the surface energy. Some examples are:

<u>Predominant Species</u>	<u>Surface Energy (ergs/cm²)</u>
-CF ₃	6
-CF ₂ H	15
-CF ₂ -	18
-CF ₃	22-24
-CH ₂ -	31

Water (surface energy about 75 ergs/cm²) cannot spread on a lower energy surface, in general, so that such surfaces are highly hydrophobic. This explains the action of organo-fluorochemicals (the first three types in the table above), and of cationic surface-active agents (exemplified by the latter two types). As explained in the Glossary, surface active agents have hydrocarbon "ends" rich in -CH₃ and -CH₂- groups. The exact hydrophobic mechanism of organo-silicone resins is more complex and is not solely a function of surface energy. This is illustrated by data in References 17 and 18. The work of adhesion (ice in shear) is cited as being much lower for silicone resin XZ-8-3057 than for FEP Teflon. This is the reverse of what would be expected from surface energy considerations (silicones generally have surface energies ranging from 18 to 22 ergs/cm² for dimethyl types to 35 or so for substituted polymers), and suggests cohesive failure within the resin. Strongly hydrophobic silicas are also known and compromise a fourth class of potential candidates for this application. The fifth class, a rubber-based coating, is discussed below with respect to binder coatings.

1.2.2 Binder Coatings with the Water/Ice Phobic Chemicals

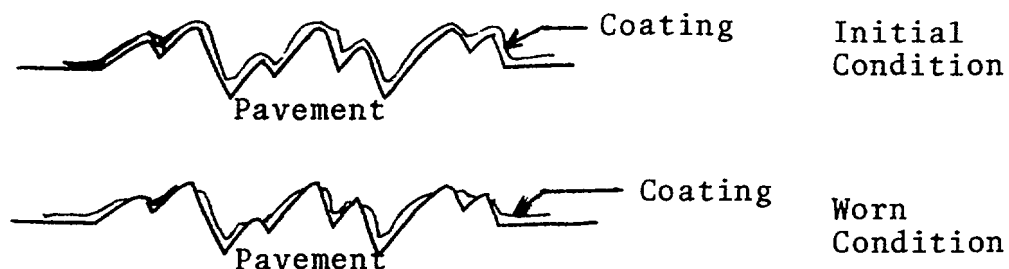
Some aspects of thickness effects have been presented earlier, namely,

- Thin films are likely to be more durable from a stress standpoint and are more economical from a materials standpoint.
- Thicker films are required to prevent long range dipole adhesion (of water) effects and may have longer abrasive wear life.

The use of "inert" binders is suggested by these considerations. The binder serves to dilute the active ingredient (thus saving costs in the case of the fluorochemical class especially), holds hydrophobic materials with low pavement adhesion in place and possibly acts as a reservoir of active material by diffusion through the binder. Also, as discussed in detail below, thicker films will reduce roughness and hence wetability. Highway paints have already been developed that have the required six-month service life (based on verbal data from Hauser laboratories, BBRC's principal subcontractor on this program). The extended wear life of rubber-based paints is also confirmed by the wear data of Reference 49. In addition it is interesting to note that the one part of a car that does not ice up is the tires, suggesting that rubber-based paints might also be advantageous themselves. This is also suggested by the wear data of Reference 49.

1.2.3 Roughness Influence Upon Coating Effectiveness

The situation may be visualized as in the following sketch for coatings much thinner than surface roughness profiles.



This schematic representation indicates what would be expected after a period of wear (traffic) on a treated pavement surface. Note that wear will proceed more rapidly at the points of highest stress. The result will be:

- Traction will be good due to exposed pavement at high points, even if the coating itself had a lower coefficient of friction than the pavement.
- The effectiveness of the coating will not be greatly affected since most of its surface area is still intact in the valleys where water would tend to collect and freeze.
- Pavement crack propagation is most likely at the root (or bottom) of surface irregularities where stress concentrations exist. These areas will be the last to be worn off so the sealing effect (pavement life improvement) will be maintained.
- For the thicker coatings, the valleys will remain filled and thus the wetability due to surface roughness will be reduced.

Even on extremely rough roadways, the possibility of water-to-water (ice-to-ice) bonding at the peak of the ridges exists. This can be evaluated only by field tests.

2.0 LITERATURE SURVEYS

Theoretical studies directly applicable to this program have been found to be quite rare. In fact, a very recent report (Reference 40) states that no satisfactory icephobic coatings, as yet, exist.

Such observations as are given below are inferred from References 1, 2, 3, 5, 7, 15, 16, 26, 31 and 36. As some of the theoretical studies cited contain test data, the division between theoretical and test studies is somewhat arbitrary. An attempt by any author to arrive at generalized rules was the criterion used in defining theoretical work. Test study conclusions are summarized in Chapter 3.

It must be emphasized that the conditions for a truly hydrophobic coating (Part 1 above) are sufficient for water but only necessary (not *sufficient*) conditions with regard to the adhesion of ice. The reasons for this are discussed below.

2.1 Dispersion Forces and Work of Adhesion

As cited in Chapter 1 of Reference 1, long-range dispersion forces (sometimes called London dipoles) are the controlling forces across interfaces (as between water or ice and a solid). This value for water (Y_w^d) is only 22 ergs/cm² as opposed to the total surface energy for water (Y_w) of 75 ergs/cm². For a solid with a dispersion force of Y_s^d , the spreading coefficient (S)* is:

$$S = -2Y_w + Y_s^d + Y_w^d \quad \begin{matrix} \text{(Ref. 1)} \\ \text{(5)} \end{matrix}$$

Using the values for water cited above, we have the rather surprising conclusion that any solid where Y_s^d is less than about 130 ergs/cm² should be hydrophobic. Since $Y_s^d \leq Y_s^{**}$, any solid with a total surface energy (Y_s) less than 130 should be hydrophobic and (per Reference 36), this includes virtually *all* organic films.

Per Reference 3, the work of adhesion (W_A), is:

$$W_A = S + W_C \quad (6)$$

where W_C is the work of cohesion of the water or ice. Equation (6) explains part of the observed adhesive ability of ice. For example, Jellinek (Reference 2) gives the theoretical tensile strength of ice as 10,000 kg/cm²*** and actual measurements cited as 16 to 80 kg/cm²****. Tensile strength cannot be directly converted to work of cohesion, although these properties should be proportional to each other. Therefore, since these actual values of tensile strength for ice are much larger than those for water, the work of cohesion for ice should be much larger than that for water. This partially explains the adhesion of ice on some surfaces, even though S for liquid water may have a large negative value.

* Remember that if S is negative, spreading will not occur, a finite contact angle will exist and the surface is thus hydrophobic.

** Reference 1, Chapter 1.

*** About 1×10^9 N/m², or 140,000 psi.

**** 230 to 1,140 psi.

2.2 Hydrophilic Sites and Temperature Effects

In addition to purely practical difficulties in achieving ice release (see Chapter 3), two other phenomena complicate matters even with carefully prepared laboratory surfaces.

As discussed in Reference 1, Chapter 1, all known solid hydrophobic surfaces have residual hydrophilic sites. Whether introduced inadvertently as in the emulsion application of TFE or as an inherent part of the structure (such as carboxyl groups in alkyd resins), the effect is the same and creates some unexpected properties. These are:

- Although Y_S^d decreases with decreasing temperature (thus making ambient temperature contact angle measurements conservatively low), the number of hydrophilic sites (as measured by crystal formation below 0 C) increases with *decreasing* temperature.
- The amount of water adsorbed on the surface (as measured by water adsorption between zero and 25 C) increases with *increasing* temperature.

We thus have two opposing phenomena affecting adhesional strength and which can vary from lot to lot of material. This might help explain the wide variation in reported ice adhesion test values. The first effect seems most pronounced with very hydrophobic (i.e., low surface energy) materials and explains the strong adhesion of ice to TFE at temperatures below 258 K (-15 C) (see Reference 15 and 34, for example). The hydrophilic site phenomenon also implies that fluids should be more efficient than solids in reducing adhesion, since localized sites of any sort cannot be maintained in a mobile medium.

Finally, the increase in adsorbed water with increasing temperature may explain the semi-fluid region between 263 K (-10 C) and 273 K (0 C) reported by many investigators (see References 2 and 26, for example).

2.3 Conclusions

Other theoretical work has been reviewed but has added little to the practical solution of this problem. On the basis of theory:

- Little seems to be gained by the selection of extremely hydrophobic materials (those with large negative values of S , including *most* organics).

- Extremely hydrophobic materials may actually exhibit high adhesion (due to the hydrophilic site effect).
- Materials containing Si-C bonds are probably not UV stable (Reference 7).
- A *stable* contact angle of water on the material, indicating very low solubility, is probably more important than a very high angle.
- Adhesion tests should be conducted below 263 K (-10 C) to avoid the semi-fluid layer region.
- Any coating must be at least several hundred nanometers (thousands of Angstroms) thick to block the London dispersion forces (Reference 41, page 55).

3.0 SURFACE ENERGY AND CONTACT ANGLE

It was proposed that solid-surface dispersion energies, and thus work of adhesion for these surfaces, could be derived from laboratory-measured contact angles of different substances, (e.g., water and oil), on these surfaces. From Reference 1, pages 8 and 9, the following equations can be derived:

$$Y_{\text{SH}_2\text{O}}^d = [(1 + \cos \theta_{\text{H}_2\text{O}})/0.13]^2 \quad (7)$$

$$Y_{\text{Soil}}^d = [(1 + \cos \theta_{\text{oil}})/0.340]^2 \quad (8)$$

$$Y_S^d = [(\cos \theta_{\text{oil}} - \cos \theta_{\text{H}_2\text{O}})/0.210]^2 \quad (9)$$

where

Y_S^d = The solid dispersion energy directly related to work of adhesion as explained in Section 2.1 above, ergs/cm²

θ = Contact angle with water or oil as noted

It was hypothesized that Equation (9) could be used to rank the dispersion energy of the coatings studied using contact angle data (Chapter 4). If the equation is valid, Equations (7) and (8) should give identical values. This was not found to be the case. It is suspected that solubility effects (see

Section 1.1 above) and/or impurities in the commercial materials studied (a basic program ground rule was the use of commercially available substances) explain the disagreement between Equation (7) and (8) values computed from the contact angle data generated in this program. The reason is that zero solubility of *both* water and oil in the coatings or impurities (a requirement of the equation's theoretical basis) is very unlikely.*

The equations are presented to permit independent evaluation of this phenomenon from the data cited in Chapter 4 and to thereby illustrate the harmful effect of solubility on dispersion energy (and thus on work of adhesion).

* This is evident from the classification of materials into oleophilic and oleophobic (or hydrophilic) groups (see Glossary). Virtually all materials fall into one class or the other and are thus solvated by either water (and other polar compounds like alcohols) or oil (hydrocarbons).

Chapter 3

PHASE I

INTRODUCTION

This chapter presents the work performed during Phase I of this investigation. The following aspects of this phase are discussed below:

- The literature survey
- The review of theoretical studies, test programs and property identification efforts
- Material vendor contacts
- Other organizations contacted
- The selection of material candidates to be evaluated in the Phase II screening tests

1.0 SEARCH SUMMARY

The results of the literature survey conducted are given in this section. Excluded are other searches (as of vendors, highway material data and meteorological data records) which are described in later sections.

1.1 Search Literature

The specific sources searched are given in Table 3-1. As indicated, a wide range of United States Government and industrial publications were reviewed. National Technical Information Service (NTIS) was not asked to search. Prior experience at BBRC has indicated that NTIS searches are duplicated by other sources. As is also indicated, most of the searches were limited to documents from 1968 to the present in view of the very complete search of Reference 20 and the search cited in Reference 4, which thoroughly covered most work prior to 1968. In many cases, of course, references cited earlier works which were also reviewed.

Table 3-1
EPA LITERATURE SEARCH* SUMMARY

Descriptors: Deicers- Ice Removal, Ice Adhesion, Hydrophobic/
Icephobic Materials, Highway Deicing

1. AEROSPACE RESEARCH APPLICATIONS CENTER (ARAC)
 - (a) Government Report Announcements
 - (b) Engineering Index, Compendex
2. COLORADO TECHNICAL REFERENCE CENTER (CTRC, University of Colorado, Boulder)
 - (a) Engineering Index
 - (b) Applied Science and Technology Index
 - (c) British Technology Index
 - (d) Chemical Abstracts
 - (e) Highway Research Information Service (HRIS) Abstracts
 - (f) Bibliographic Index
 - (g) Highway Research Abstracts
 - (h) Government Reports Index
 - (i) International Aerospace Abstracts
 - (j) Monthly Catalog of U.S. Government Publications
 - (k) Subject Guide to Books in Print
 - (l) Library of Congress Catalog, Books: Subjects
3. DEFENSE DOCUMENTATION CENTER (DDC)
 - (a) All DOD Documents
4. NASA SCIENTIFIC AND TECHNICAL INFORMATION FACILITY
 - (a) NASA Documents

*Most searches were limited to the period 1968 to date in view of the comprehensive search (over 200 applicable references) made by Cold Regions Research and Engineering Laboratory in Icing Occurrence, Control and Prevention, by K. L. Carey, July 1970 (AD 711 534).

1.2 Search Comments

During the search, abstracts of over 500 references were reviewed. In general, most were concerned with aircraft or stationary installations with auto-release, i.e., no applied force other than gravity or air streams, of ice from the structures being the goal. This goal accounts for the rather pessimistic conclusions (of References 17 and 21) that "passive ice removal techniques are not feasible". In contrast, it must be recognized that in the present case there are additional forces available (traffic load, for one) which can aid in ice removal. In addition, the concern here is only with adhesion *reduction*, not elimination. Complete adhesion elimination, in fact, would create a dangerous roadway skid hazard.

1.3 References

All program references are given at the end of this report. Many have been derived from sources in addition to the survey conducted during this initial phase. For this reason, and also because of the reorganization of material during the preparation of this program report, cited references are not necessarily listed in the same order as they first appear in the text.

2.0 THEORETICAL STUDIES

Theoretical studies reported in the literature have been summarized in Chapter 2.

3.0 TEST PROGRAMS

The test programs reported in the literature were not especially applicable to the current program. However, some generalizations could be inferred which were helpful in material selection.

3.1 General Comments

In all the programs reviewed, two general features appeared in most studies:

- The coatings and substrates were made as smooth as possible (References 2 and 31 are exceptions)
- Low ice adhesive shear strength was the sole criterion for successful coatings

3.2 Common Results of Tests

In spite of the wide variation in test techniques and reported values, certain conclusions seemed common to (or at least were not contradicted by) most studies:

- Some degree of flexibility of the coating reduces ice adhesive strength (References 2, 12, 14, 15 and 30)
- Polar sites increase adhesion, as would be expected from the hydrophilic site theory discussed in Chapter 2 (References 13, 16 and 27)
- Nonporous surfaces are required to promote low adhesion (References 15 and 21)
- Fluid films give lowest adhesive force (References 17, 22, 27, 29, 31, 34 and 37)
- Rough substrates give higher adhesion values (References 2 and 31)
- Monolayers of even very hydrophobic substances are ineffective in reducing adhesion, as would be expected from the range of London dispersion forces discussed in Chapter 2 (References 2 and 3)

3.3 Anomalous Results

The number of anomalous results reported in the literature, remembering that hydrophobicity is a necessary but not sufficient condition for low ice adhesion strength, were few. Most anomalies concerned the effect of temperature on adhesive strength. This is really not surprising in view of the two opposing effects cited in Chapter 2. As is also pointed out in Chapter 24 of Reference 1, the effect of temperature on contact angle can even be reversed depending on whether the area of water coverage is increasing or decreasing.

With regard to material selection, in general it was found that hydrophobicity is some guide to low ice adhesion even though one reference (Jellinek in Reference 2) concludes that there is no correlation of adhesion with either coating surface energy or contact angle. The detailed problems in material selection and the criteria used by BBRC are discussed next.

4.0 MATERIAL PROPERTIES AND SELECTION CRITERIA

Identification of candidate material properties are presented below along with tradeoff problems and the selection criteria actually used on this program.

4.1 Hydrophobicity

The degree of hydrophobicity of the materials selected requires special mention. As pointed out in Chapter 2, to give a finite contact angle, theory requires only that the surface energy of the coating be less than about 130 ergs/cm². Of the 43 polymers listed in Reference 36, the highest value is 61 ergs/cm². Thus, at first glance, virtually any organic coating should be considered.

However, greater hydrophobicity also implies lower water solubility and a breakup of impinging water into small drops, which gives trapped air bubbles at the interface of the ice/coating and thus reduces adhesive strength even further (see Reference 26). We certainly wish to minimize solubility and to maximize bubbles. Consequently, relatively higher contact angles are desirable, indicating that relatively lower surface energy materials should be selected.

4.2 Problems in Property Identification

Properties of proposed coatings under specific conditions can be determined. Of concern here is the fact that any given property has both desirable and undesirable consequences. A few such tradeoff problems are illustrated below.

Phase. Solid films are less likely to be removed by traffic and are less likely to be slippery. On the other hand, liquids cannot maintain hydrophilic sites and they also provide more uniform coverage.

Thickness. Relatively thick layers are required to provide desirable flexibility and to block London forces. However, they will be more costly and will promote skidding by smoothing the highway macrostructure (see Reference 6 and Reference 62 in Appendix B).

Solubility. Water soluble coatings are much safer and cheaper to apply, but unless they are very reactive with the highway surface or tend to form polymers, they will not remain in place. Solvent-soluble polymer coatings have the reverse characteristics.

Biodegradability. This is desirable if the material is removed from the road surface, but it could result in rapid deterioration of the material on the surface.

Highway Materials. As discussed in detail in Reference 54, Appendix A, the diversity of highway surfaces, (e.g., concrete is porous and alkaline while asphalt surfaces are non-porous and acidic or neutral), makes the existence of an all-purpose coating appear unlikely. Further, the hydrocarbon (and thus somewhat oleophilic) nature of asphalt indicates the need for solvent application, in contrast to the procedures possible for the easier-to-treat concrete roads. Unfortunately, the latter comprise only about six to seven percent of the total miles of surface (verbal from the Colorado Department of Highways).

Cost. An inexpensive material may require a very expensive solvent for application. While such considerations are properly a part of the Phase III application study, it would be futile to even screen a material that requires a \$20 per kilogram (\$9 per pound) solvent.

The above tradeoff problems complicate material selection and make postulation of "ideal" coating properties virtually impossible.

4.3 General Selection Criteria

In spite of the difficulties cited in Section 4.2 above, selections for the screening tests had to be made. Since this was a somewhat arbitrary area, *all* materials considered are given in Table 3-2 to facilitate independent review.

Selection criteria were:

Fluidity. Any material existing and *remaining* in a fluid state after application was rejected. Rapid removal by traffic and skid promotion were believed to govern here.

Cost and Solubility. Any extremely expensive material or any material requiring an expensive organic solvent was rejected unless it was believed that valuable technical information, possibly for future programs, might be gained.

Other Factors. High toxicity for either the as-received or dried-film condition (judged from OSHA standards), vendor recommendation, likely low surface energy and prior usage in related applications were considered.

Table 3-2
HYDROPHOBIC HIGHWAY MATERIALS

Class: Cationic Surface-Active Agents

MATERIAL			Vendor/Contact	FORM		Solubility Data	Application Methods	Surface Energy	Availability	Toxic Rating	UV Stability	COST	BBRC Comments	Vendor Comments	Test Decision
Trade Name	Chemical Name	Other		Basic	As Purchased							¢/kg or as Noted			
Aliquat H-226	Dimethyl Di-hydrogen, tallow ammonium chloride		General Mills Chemical, Inc. Minneapolis, Minnesota (612) 540-2461 Al deMuerisse, Marketing	Solid	Paste (75% in solids aqueous isopropanol)	Soluble in alcohol. Forms H ₂ O dispersion	Spray, immersion	No information	Poor in mega-kilo lots at present	Very low	No information	44		Work has been done at Texas A&M on soil stabilization using this material	Test
Aliquat 254	Dimethyl Di-fatty ammonium chloride		General Mills Chemical, Inc. Minneapolis, Minnesota (612) 540-2461 Al deMuerisse, Marketing	Solid	Semi-fluid (75% solids in aqueous isopropanol)	Soluble in alcohol. Forms H ₂ O dispersion	Spray, immersion	No information	Poor in mega-kilo lots at present	Very low	No information	26			Test if different than H-226
Chemical 39 High Conc	Fatty amide		Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Waxy Solid	Solid	Soluble in H ₂ O	Spray, immersion	No information	Good	Very low for skin contact	No information	121			Test
Chemical 39S High Conc		Cationic "Compound"	Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Solid	White Colloidal dispersion	Forms H ₂ O dispersion	Immersion	No information	Good	No information	No information	56			Test if different than 39
Ceramine HCA granules		Weakly Cationic Softener	Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Solid	Solid	Soluble in boiling H ₂ O	Immersion	No information	Good	Very low for skin contact	No information	146			Reject only weakly cationic
Cartarelin-F	Polyamide Amine		Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Liquid	Liquid	Soluble in H ₂ O	Immersion	No information	Good	No information		50-56			Test
Viscospin-B	Poly-ethoxy fatty imidazoline		Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Liquid	Liquid	Soluble in H ₂ O, acid base		35.1 ergs/cm ² for 0.1% H ₂ O solution	Good	No information (blode-grades)	No information	134		Cation-ogenic surfactant for viscose; has also been used in asphalt to hydrophobe surface	Test;
Ceramine PHS		Weakly Cationic Softener	Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Solid	Solid	Soluble in boiling H ₂ O	Pad, exhaust, spray	No information	Good	No information	No information	120	Use as antistat implies water absorption (not good)		Reject, weakly cationic
Cartarex FL		Optical Quencher	Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown, Tech. Service	Liquid	Liquid	H ₂ O soluble	Dip, spray	No information	Good	No information	No information	396		Used to reduce fluorescence in paper making	Reject; stays fluid
Chemfil Car Gloss	Blend of: • Nonionic surface • Waxes • Cationic fatty nitrogen complex	"Chemsheen"	Chemfil Corporation Troy, Michigan (313) 689-0720 Ray Charles, Product Devlpmt	Solid-Liquid Mixture	Liquid	Water soluble	Spray	No information	Good	No information (blode-grades)	No information	200-300	Currently used to hydrophobe car paint		Test
Arosurf TA-100	Dimethyl-tallow-ammonium chloride		Onyx Chemical Jersey City, New Jersey											Same as Ali-quats and viscospin B	Do not test
Softener "X"	Alyki-imidazoline Derivative														
"Hipochem" "Aquafruf"	Quaternary Methol Amide		Highpoint Chemical Corp Highpoint, North Carolina Kim Stevens J. P. Stevens Company, Inc. Garfield, New Jersey (201) 772-7100 Pete Drexler	Aquafruf has not been produced for several years									Unable to locate vendor	5/10/74; not available	Reject; not available

Table 3-2 HYDROPHOBIC HIGHWAY MATERIALS (Continued)

Class: Organo-Silicones

MATERIAL			Vendor/Contact	FORM		Solubility Data	Application Methods	Surface Energy	Availability	Toxic Rating	UV Stability	COST \$/kg or as Noted	BDR Comments	Vendor Comments	Test Decision
Trade Name	Chemical Name	Other		Basic	As Purchased										
	Silicone	XI-8-3057	Dow Corning Midland, Michigan (517) 636-8955 Daryl Dickson, Ext. 8594	No longer	available per	Daryl Dickson					Poor				Reject; not available
Dow-Corning 92-009	Silicone Rubber		Dow Corning Midland, Michigan (517) 636-8955 Daryl Dickson, Ext. 8594	Solid	32% by weight dispersion	Soluble; VMP naphtha	Spray, paint, etc.	Not known	Good	Low	Claimed to be high	1500 (32% dispersion)			Test
	Organo Silicones		Union Carbide Technical Division Tarrytown, New York () J. A. Schofield											U.C. has experimented and cannot develop any silicone compound that is not slippery.	Reject
	Z-4141 series silanes		Dow Corning Midland, Michigan (517) 636-8955, Ext. 9484 Ward Collins, Tech Serv Div	Solid		Not known by Ward Collins	Spray, paint, etc.	Not known	No information	No information	No information	No information		Z4141 will make glass hydrophobic when applied; received no additional information from Dickson	Reject; not available
1) Anti-static #79 2) Wipe 3) Anti-fog	Fatty amine, Glycol mix		Merix Chemical Company Chicago, Illinois (312) 231-8242 Dave Sonnenman	1) Liquid 2) Liquid 3) Liquid	All three are liquids	All three H ₂ O soluble	1) Spray, brush, dip 2) Wipe on 3) Wipe on	No information	No information	No information	No information	No information	H ₂ O soluble materials; not useful for hydro, road coating		Reject; remains water soluble
	Silicone varnish		3M Company Minneapolis, Minnesota (612) 733-9710/4619 John Norwood (Varnish Prod) (referred to Fred Schwabe Tech Elect Prod)											Not 3M product; suggested we contact primary suppliers of silicone varnish (Dow Corning and General Electric)	Reject
	Silicone	EC-1981	3M Company Minneapolis, Minnesota (612) 733-1110 L. Shaver (Comm, Chemicals)											EC-1981 is an experimental silicone compound; it is not being marketed at present	Reject; not available
Caulking Compound #684	Silicone Rubber	DC 732 clear	Dow Corning Midland, Michigan (312) 671-3100			Soluble in VMP							To be studied as cheaper version of 92-009		Test
Formula 125	Polysiloxane base		Transcontinental Research and Development Tucson, Arizona (602) 294-3463 Tom Wallace	Liquid	Liquid in solution	Supplied as water solution	Spray; dilute 44:1	No information	Good	Low	No information	300 (for concentrate)		Has been lab tested by Arizona H.D.; used in new road mix, building foundations, and soil stabilization; self cures to solid	Test
	Methyl-silicone		General Electric Schenectady, New York (518) 237-3330 Scott Hurley (technical)	Solid	Solvent solution	Mineral spirits	Dilute from 70% concentration and spray	No information	Good	Low	No information	\$3.85/kg (70% concentrate)		Silicone varnishes not recommended	Reject; varnishes require high temperature cure and are brittle
SC-3700	Silazane		General Electric Schenectady, New York (518) 237-3330 Scott Hurley (technical)	Liquid	Liquid chemical	Benzene, toluene, hexane, CCl ₄	Dilute and spray	No information	Good	Low	No information	\$22/kg (100% concentrate)			Test one of these
Z-6079	Hexamethyl-disilazane		Dow Corning Midland, Michigan (517) 636-8594, 8597, 8374 Lou Arends Mr. Henderson	Liquid	Liquid chemical	Benzene, toluene, hexane, CCl ₄	Dilute and spray	No information	Good	Low	No information	\$1000 (100% concentrate)		Reactive with water and methanol	
DC772	Sodium methyl silicate CO ₂ reactive		Dow Corning Midland, Michigan (517) 636-8000 Ray Humphrey	Solid	30 percent solids in liquid dispersion	H ₂ O dispersion	Dilute to 5% and spray	No information	Good	No information	No information	66-77 (30% concentrate)	Caustic solution.	Humphrey has worked with the Michigan H.D. on various problems; DC-772 will into road surface to depth of 2-4 mm.	Test
Dri-sil 73 H ₂ O repellent	Silicone H ₂ O reactive		Dow Corning Midland, Michigan (517) 636-8000 Ray Humphrey	Solid	Solvent solution	Mineral spirits or stoddard solvent	Dilute and spray	No information	Good	No information	No information	500 (60% concentrate)	Flash Point = 65°C	Reported used with Silanox 101 to hydrophobe sand. 400 (101) 101 (73)	Test
G11dair #1903	Silicone		XIN Products, Incorporated Westlake, Ohio (216) 871-4737 Harold Vertman	Silicone fluid	Sprayable solution		Spray	No information	Poor; sold only in 16 ounce spray cans	No information	No information			Vendor states material is very slippery. Not applicable.	Reject; remains fluid

Table 3-2 HYDROPHOBIC HIGHWAY MATERIALS (Continued)

Class: Rubber Base Coatings

MATERIAL			Vendor/Contact	FORM		Solubility Data	Application Methods	Surface Energy	Availability	Toxic Rating	UV Stability	COST \$/kg or as Noted	BBRC Comments	Vendor Comments	Test Decision
Trade Name	Chemical Name	Other		Basic	As Purchased										
	Silicone rubber		General Electric Construction Silicone Department New York, New York (518) 237-3330 Vic Jordan; referred to Rich Gibbons	See com- ments										GE has applied some sili- cone rubbers to road sur- faces. Abrasion resis- tance poor; coatings peeled off; not recom- mended	Reject; poor performance
	Silicone rubber		Pecora, Incorporated Philadelphia, Pennsylvania (215) 247-5342 Garland, Texas (214) 278-8158 Marketing Division, refer- red to Don King, Texas	See com- ments										Pecora markets construc- tion caulking compounds. Not applicable to EPA road study.	Reject; not applicable
Rubber base paints per Fed Specs TT-P-85, TT-P-110, and TT-P-115			Various										See references 54 and 49.	See following lines	Test or con- sider further
	Paint per Fed Spec TT-P-115D, Type II		Goodyear Chemicals Akron, Ohio (216) 794-4400 Dave Bognar, Chemist	Per Fed Specification		Per Federal Specification		Unknown	Can be formu- lated by two companies	Low; meets EPA solvent re- quirements	Good	Unknown	Contact 5/17/74	Should cure in 40 minutes	Test
Aroclon 376	Safflower Alkyd resin		Ashland Chemical Kansas City office (816) 221-7177 Jerry Perrine	Solid	H ₂ O solution (40% solids)	H ₂ O	Spray, paint	Unknown	Good	Low	Good	80	Contact 5/17/74; use as binder	Cures tack-free in two hours	Test
Aroclon 585	Modified Safflower Alkyd		Ashland Chemical Kansas City office (816) 221-7177 Jerry Perrine	Solid	H ₂ O disper- sion (42% solids)	H ₂ O dispersion	Spray, paint	Unknown	Good	Low	Good	80	Contact 5/17/74; Use as binder.	Cures tack-free in ten minutes	Test
Arothane 150	Urethane		Ashland Chemical Kansas City office (816) 221-7177 Jerry Perrine	Fluid	Mineral spirits (50% solids)	Mineral spirits	Spray, paint	Unknown	Good	Low	Good	85	Contact 5/17/74; use as binder.	Cures tack-free in 30 minutes	Test

Class: Fluorochemicals

MATERIAL			Vendor/Contact	FORM		Solubility Data	Application Methods	Surface Energy	Availability	Toxic Rating	UV Stability	COST \$/kg or as Noted	BBRC Comments	Vendor Comments	Test Decision
Trade Name	Chemical Name	Other		Basic	As Purchased										
Holykote 519 Barrier Coating	Not known but presumed fluo- oro-derived resin		Dow Corning Midland, Michigan (517) 636-8955 Steve Miller	Pre- sured solid	Solvent solution	Not known yet	Solvent solution	Not known; pro- bably <22 ergs/ cm ²	Good per vendor	Should be low; stable to 288°C	Not known	1000 (as solution)	No information from vendor on conc.	Not a Dow product	Do not test; (more expensive from non-prime source)
Frekote 33	Proprietary to Frekote		Frekote, Inc. Florida (305) 395-3083 Indiana (317) 547-6388 Nancy Layman, President Jim Shultz, Research Dir	Solid	Solvent solution	No solvents af- ter cure per vendor	Spray paint, etc.	Not known	Good per vendor	Very low	Not known	2000 (base resin)	Telecon 6/27; high- er conc.; being shipped	Can be chemically modified	Test
	Proprietary fluorochemi- cal	L-1481	3M Company Minneapolis, Minnesota (612) 733-1110 L. Shaver											L1481 is an experimen- tal fluorochemical not being marketed at pre- sent. 3M has some com- pounds (unnamed) they have applied to garage floors with good hydro- results. Not marketed because of high cost.	Reject; not available
Scotchgard	Proprietary to 3M	FC-321	3M Company Minneapolis, Minnesota (612) 733-5057 Chauncey Martin (Adhesives, Coatings, and Sealants)	Solid	20% solvent solution	No data given; chloroethane freon	Spray	Not known	Good	Low	Not known	4000 (base resin)			Test one of these
Scotchgard	Proprietary to 3M	FC-210	3M Company Minneapolis, Minnesota (612) 733-5057 Chauncey Martin (Adhesives, Coatings, and Sealants)	Solid	28% H ₂ O sus- pension		Spray	Not known	Good	Low	Not known	4000 (base resin)		Scotchgards are tall- ored for textile ap- plication and not recommended. 3M re- ports internal tests show no good. (Verbal 3/21/75)	
Nyabar Type C	Proprietary Fluorochemi- cal		M. F. Nye, Incorporated New Bedford, Massachusetts (617) 996-6721 George Mock	Solid	Solvent Solu- tion	Xylene hexa- fluoride	Brush, spray, dip	~11 ergs/cm ²	Good	Air concen- tration should be kept below 100 ppm	No informa- tion	26400 (2% solution)			Reject; far too expensive
Nyabar Type F	Proprietary Fluorochemi- cal		M. F. Nye, Incorporated New Bedford, Massachusetts (617) 996-6721 George Mock	Solid	Solvent solu- tion	Freon TF, chlo- roethane	Brush, spray, dip	~14 ergs/cm ²	Good	Keep <1000 ppm with Freon solvent and <300 ppm with chloro- ethane	No informa- tion	5280 (8% solution)			Test

Table 3-2 HYDROPHOBIC HIGHWAY MATERIALS (Continued)

Class: Inorganic

MATERIAL			Vendor/Contact	FORM		Solubility Data	Application Methods	Surface Energy	Availability	Toxic Rating	UV Stability	COST	BBRC Comments	Vendor Comments	Test Decision
Trade Name	Chemical Name	Other		Basic	As Purchased							\$/kg or as Noted			
Silanox 101	Silane treated silica	fumed	Cabot Corporation 125 High Street Boston, Massachusetts 02110 Technical Center (617) 865-3455 8111 Gfrelda	~0.05 dry powder	Dry powder or powder/resin system	Insoluble	Flame spray or in binder 0.03 lbs/1000 ft ²	No data yet; like CH ₃	May be limited in mega-kilo lots	Very low	Not known	700		Silanox now mixed directly into concrete to improve H ₂ O resistance of new and resurfaced roads.	Test

Other Materials

MATERIAL			Vendor/Contact	FORM		Solubility Data	Application Methods	Surface Energy	Availability	Toxic Rating	UV Stability	COST	BBRC Comments	Vendor Comments	Test Decision
Trade Name	Chemical Name	Other		Basic	As Purchased							\$/kg or as Noted			
Cartarex FL			Sandoz Colors & Chemicals Hanover, New Jersey (201) 386-7690 Harmon Brown	Liquid	Liquid	Soluble in H ₂ O	Immersion	No information	Good	No information	No information			Used for the reduction of fluorescence in paper-making	Reject; remains fluid
UCAR Runway Deicer	Glycol base		Union Carbide Func. Chemical Department New York, New York (212) 551-5114 R. Kennedy	Liquid	Liquid	Soluble in H ₂ O, ether, alcohols	Spray	No information	Good	No information	No information			Used as thin film. Not suitable for semi-perm. coating. U.C. has not pursued road coatings because of need for periodic application	Reject; remains fluid
PCL-300 and PCL-700	Polycaprolactone	Crystalline chemoplastic resin	Union Carbide Func. Chemical Department New York, New York (212) 551-3887 Chicago (312) 822-7163 J. A. Schofield	Solid	Solid: PCL300 flakes; PCL 700 pellets	Soluble in aromatic H ₂ C, and some chlorinated solvents		No information	Good	Slight	No information	230		Biodegrades	Reject; like nylons, absorbs water
	Monosilicic acid		Stauffer Chemical Company Industrial Chemical Division New York, New York (212) 421-5000 Jack Blum, Los Angeles Jack McLaughlin, New York	Liquid	Liquid	Soluble in H ₂ O	Mix with other chemicals	No information	Good	No information	No information	~100	Used as a binder w/ ceramics and glass; cures w/basic compounds		Test
"D", "H", "RU"	(Si ₂ O ₅ /Na ₂ O) silicates		Philadelphia Quartz Company Valley Forge, Pennsylvania (215) 687-8400 Jerry Bernstein	Liquid	Liquid	Soluble in H ₂ O	Mix with other chemicals	No information	Good	No information	No information	~100		"D" colloidal control; "H" binder for cements, leak sealer; "RU" fast setting gel, cures with acidic materials	Test
Ethyl Silicate cond. E.S. E.S. 40	Tetraethyl orthosilicate		Union Carbide Chicago, Illinois (312) 822-7104 Cliff Schwahn	Liquid	Liquid	Soluble in water		No information	Good	100 ppm per OSHA for ethyl silicate	No information	170 110 120		Reacts with acid, ethanol, water to form gel, then H ₂ O from air to give silica	Do not test; too toxic
"ISOLV"	Proprietary fluid deicer		Kaiser Agricultural Chemicals Savannah, Georgia (912) 954-4311 F. E. Burton	Fluid and soluble components	Fluid	Soluble in H ₂ O	Spray	No information	Not now available (5/10/74)	Low and biodegradable	No information	30 (if available)	Not hydrophobic	Will advise BBRC if becomes available	Reject; not available
	Phenylene diamine		Naval Civil Engineering Lab Port Hueneme, California (805) 982-4679 Dr. Alumbaugh	Solid	Solid	Soluble in H ₂ O				High		1000			Reject; too hazardous per OSHA
	Polysiloxane, fluorocarbon, polyester, polyurethane polyvinyl fluoride		Naval Civil Engineering Lab Port Hueneme, California (805) 982-4657 Dr. Peter Hearst	Liquids and solid films	Same	See addendum to reference 17		No information	Good	No information	No information				Reject; see text
	Fluorinated epoxies		Naval Research Lab Washington, D. C. (202) 767-2529 J. R. Griffith, Research					Should be very low	Poor at present			Very high at present	Contact Mr. Griffith in March		Test
Xylan 1010	Acrylo-amide		Whitford Corporation Westchester, Pennsylvania (215) 436-0600 Tom Sloan	Solid	Solvent suspension	Dimethyl-formamide	Spray; preheat to 150°F					~2000	Coatings quite slick but not tested against rubber	Thirty-minute cure at 280°F required	Reject; high temp cure reqd
Xylan 2014 and 2052	Complex acrylo-amide with TFE		Whitford Corporation Westchester, Pennsylvania (215) 436-0600 Tom Sloan	Solid	Solvent suspension	Dimethyl-formamide	Room temperature cure	Probably low	Good	No information	No information	~2000		Vendor commented material probably not suitable without other binders	Test
DAP Caulking materials	Not known	Not known	DAP, Incorporated Dayton, Ohio (513) 253-7151 Walt Abreth, Research Eng.										Old not seen at J22 willing to supply samples	Feels that the basic paint vehicle approach we're taking is correct. 5/23/74.	Reject

5.0 CONTACTS

This section presents the contacts made with vendors and other outside experts in various fields and also briefly describes the contact/materials work sheets included as Table 3-2. This table is included primarily to illustrate the number of contacts made and the selection procedure.

5.1 Vendor Contacts

The work sheets included as Table 3-2 illustrate the value of these contacts. In many cases vendors made specific recommendations and a few cited applications for cases similar to the problem here. While the recommendations of a few vendors were not taken (e.g., 3M's opinion that Scotchgard would not work), vendor contact proved to be the most valuable source of practical data found.

A few comments concerning materials listed on the work sheets are included here:

- The Onyx Corporation materials are the same as others and the High Point Chemical material could not be located.
- In addition to materials suggested from the literature, other materials were included for at least preliminary consideration. These included vendor-suggested materials, materials used by BBRC previously, and consultant-suggested materials.
- Note that the only fluids selected to be screened were those which either solidify or could be expected to strongly react with the highway surface.
- Some materials have been used in highway mixes or for soil stabilization although none have, to date, been applied to roadway *surfaces*.
- Rather than pushing products, some vendors (note Entries 3, 10 and 15 on page 27) stated that some of their products were *not* recommended. It must be pointed out that 3M also did not feel their Scotchgard material would work, even though we believed they should be screened.
- Note also that the plastic films on page 29 (ninth entry, Other Materials) which were suggested by some sources, either proved very high in ice adhesion or similar compounds were screened in this study.

- In most cases, comments in the right-hand column indicate the reason for rejection.

The detailed final list of materials selected for screening is presented in Table 4-1 of Chapter 4. A summary list by categories plus comments on mixtures and combinations of materials is given in Section 6 of this chapter.

5.2 Other Contacts

Other major contacts are summarized in Table 3-3. The three most important areas are discussed in somewhat more detail in this section.

5.2.1 Hauser Laboratories

Hauser Laboratories was requested to summarize roadway materials' properties and to present the state-of-the-art in highway marking paint. Their complete report is included as Reference 54 in Appendix A. Some of the more important aspects of this report have been given in Section 4.2 of this chapter.

It is noted, from the Hauser report, that concrete roadways present a much easier application and coating retention problem due to the void content of the concrete and its somewhat greater roughness than asphalt. Good adhesion to asphalt may necessitate the use of organic solvents which partially attack the binder. This conclusion agrees with that expressed by the Colorado Department of Highways (Appendix B). Note also the inclusion of the friction coefficient curves used as a guideline for the screening friction tests. Finally, note the typical paint application rate of 0.39 l/m² (equal to 105 feet² per gallon) to give reasonable life. The original thought of using such paints as binders required re-evaluation in view of this rather low and therefore costly coverage rate.

5.2.2 Colorado Department of Highways (CDH)

The original contact report with CDH is given as Reference 62 in Appendix B. Of special interest are:

- The availability of accident records with prevailing weather conditions for most areas of the state.
- The availability of full scale and portable road skid test apparatus.

Table 3-3

CONTACTS OTHER THAN VENDORS

Organization	Personnel	Purpose/Results
Colorado Department of Highways, Denver, Colorado	B. B. Gerhardt B. A. Brakey	<ul style="list-style-type: none"> ● Obtain traffic density map of test sites. ● Obtain Colorado State Solvent Pollution rules. ● Obtain data on in-situ friction testing of roads. ● Obtain agreement for cooperation during road tests. ● Contact Accident Data Center personnel. ● Obtain roadway surface roughness data.
Naval Research Laboratory, Washington, D. C.	J. R. Griffith	<ul style="list-style-type: none"> ● Obtain agreement to coat test samples with experimental fluoroepoxy.
Debell and Richardson, Enfield, Connecticut	E. S. Childs	<ul style="list-style-type: none"> ● Try to obtain more data (specific compositions) on water-borne coatings. No luck. Index only sent.
NASA Lewis Research Center, Cleveland, Ohio	Vernon Gray	<ul style="list-style-type: none"> ● Check report (from ARAC survey) that NLRC made road tests on hydrophobic materials during WW II. Report incorrect.
U. S. Army Cold Regions Research and Engineering Laboratory, Hanover, New Hampshire	Technical Library	<ul style="list-style-type: none"> ● Obtain copies of References 29, 30 and 31 from EPA Report R2-72-125.
National Oceanic and Atmospheric Administration (NOAA), Boulder, Colorado	Receptionist	<ul style="list-style-type: none"> ● Inquire regarding local weather recording facilities. No longer record such data.
Boulder Municipal Airport	Receptionist	<ul style="list-style-type: none"> ● Inquire regarding local weather recording facilities. No data or instrumentation.
National Center for Atmospheric Research (NCAR), Boulder, Colorado	D. Baumhefner	<ul style="list-style-type: none"> ● Inquire regarding local weather recording facilities. NCAR records climatological data only when their needs require.
Hauser Laboratories, Boulder, Colorado	Dr. Ray L. Hauser	<ul style="list-style-type: none"> ● Obtain report (Appendix A) on roadway materials' properties and current highway prints.

- Their opinion of normal *coating* coverage rates of 0.09 to 0.18 ℓ/m^2 (0.02 to 0.04 gallons per square yard) which are quite close to the coverage rate cited in Reference 4.
- The cited macrostructure roughness (which controls high speed skid resistance) of about $10^{-3}m$ (40 mils) arithmetic mean square (ams) implies that if rather thick coatings are needed for wear life, they can be used without covering the peaks (in agreement with Reference 6).
- Even the microstructure, controlling low speed skid resistance per CDH, has a roughness of about one-tenth of that above (Reference 6). This still permits a $10^{-4}m$ (4 mil) coating without covering the tire/road contact asperities.

5.2.3 Weather Recording

As noted in Table 3-3, use of outside sources to obtain local meteorological data during the road test phase was not possible. For budgetary reasons, sophisticated weather recording equipment also could not be employed. Instead, as is discussed in Chapter 6, intermittent measurements were made of air and road surface temperatures along with estimates of wind speed, precipitation, etc.

6.0 PHASE II MATERIAL'S LIST

Listed in this section by chemical/physical class (see Section 1.2.1 of Chapter 2), are the 35 materials selected for Phase II screening. By screening is meant any combination of the tests used (see Chapter 4) to indicate promise of a material or to eliminate one from further consideration. To avoid duplication of much of the data presented in Chapter 4, and in Table 3-2 of Section 4.3 above, only the class, material and vendor are given here. A few brief comments on material combination philosophy are also given.

6.1 Materials

6.1.1 Cationic Surface Active Agents

- Aliquat H-226 (General Mills)
- Aliquat 264 (General Mills)
- 39 High Concentrate (Sandoz)

- 39S High Concentrate (Sandoz)
 - Cartaretin-F (Sandoz)
 - Viscospin-B (Sandoz)
 - Chemsheen (Chemfil Corporation)
- 6.1.2 Fluorochemicals
- Scotchgard FC-321 (3M)
 - Scotchgard FC-210 (3M)
 - Nyebars F (W. F. Nye, Incorporated)
 - Fluoroepoxy (NRL)
 - Xylan 2052 (Whitford Corporation)
- 6.1.3 Silicone Base
- Frekote 33 (Frekote, Incorporated)
 - DC 92-009 (Dow-Corning)
 - Formula 125 (Transcon R&D)
 - DC 772 (Dow-Corning)
 - DRI-SIL 73 (Dow-Corning)
 - SS-4044 (General Electric)
 - DC 732 (Dow-Corning)
 - RTV-11 (General Electric)
 - G31, 2X (BBRC)
 - G31, Thin (BBRC)
- 6.1.4 Paints and Paint Base Binders
- Federal Specification TT-P-115D (Goodyear)
 - Federal Specification TT-P-115D without TiO₂ pigment (Goodyear)

- Federal Specification TT-P-85D (Kwal Paint)
- Arothane 190M50 (Ashland Chemical)
- Arolon 376 (Ashland Chemical)
- Arolon 585 (Ashland Chemical)

6.1.5 Hydrophobic Silica

- Silanox 101 (Cabot Corporation)

6.1.6 Other

- Silicates "D", "N", "RO"; possible binders (Philadelphia Quartz)
- Monoaluminum Phosphate; possible binder
- Z-6079; possible hydrophobe (Dow-Corning)
- Petroset AT (Phillips Petroleum Company)

Note that the above listing includes some materials that were not even suspected of being hydrophobic and more than twice the number of items originally planned for screening. The two BBRC formulations developed from other work were selected for only cursory screening.

6.2 Material Combination Philosophy

It was recognized that some very hydrophobic materials, as judged from the literature, had other properties such as lack of abrasion resistance (e.g., DC92-009 and DC732), or zero cohesive strength (e.g., Silanox 101, a dry powder), which required the use of binders. Thus, the emphasis on binders was believed to be a necessary requirement in view of the ultimate program goal -- a successful *coating*.

Also, it appeared unlikely that any single commercial material could have all the needed characteristics (hydrophobicity, impermeability to water, toughness, abrasion resistance, adequate friction coefficient, etc.). From the start it seemed evident that a composite formulation would be required.

Chapter 4

PHASE II SCREENING TESTS

This chapter presents the data and observations for both pure (single component) materials and formulated (multicomponent) coatings acquired during the screening (basic property determination) tests.

As stated in the Intent of Report (see Recommendations and Summary, Chapter 1), an attempt to present all data obtained, whether positive or negative, is the basic rationale here.

Organization of the data has proved to be a major problem. This is due to both the amount of material to be presented and, because of the receipt of test materials over an eight-month period, an inability to run tests on all materials in a sequential step-by-step fashion. Accordingly, the data are organized by major category and chronologically within each category. Observations, especially on degradation phenomena, and computations are interjected as necessary.

Finally, the basic goal of a coating that is applicable to roadway surfaces with standard spray equipment and which employs commercially-available material must be kept in mind. The real-life target of this investigation strongly affected the test methods employed.

1.0 INFRARED ANALYSIS AND OTHER MATERIAL CHARACTERISTICS

Infrared spectra were run on most materials using either a Perkin-Elmer Model 700 or a Beckman Model 20AX spectrometer. These data were required for molecular identification and to serve as a quality control guideline. All spectra were run on the dried solids to remove possibly conflicting absorption bands due to solvents or diluents. In some cases, the as-received materials were also run to determine the solvents employed. Spectra could not be obtained from the Silanox 101 (because of scatter from the sample), or from the fluoroepoxy supplied by NRL (because of the substrate on which the sample was supplied). The formulations of the two BBRC coatings as well as those of all other formulated coatings are defined in Chapter 5.

These data are presented in Table 4-1. Also included in this table are other material characteristics derived during the Phase II work and not properly belonging in other major categories. The other solvents listed were determined by experiment. The toxicity ratings were inferred from the chemical natures of the materials and solvents, using OSHA recommendations. The as-supplied pH and non-volatile solid content were determined by test at BBRC. The tack-free drying rate was also determined experimentally at ambient temperature.

2.0 SURFACE ENERGY AND WETTING CHARACTERISTICS

The data, observations and some typical photographs accumulated during this extensive effort are presented in this section. The need for this basic data has been discussed in Chapter 2.

2.1 General Approach

It was originally planned to use filter paper as an absorptive, "rough" substrate for this work and some of the earlier data were so obtained. However, it was found that even applications of the materials were quite difficult and contact angle measurements nearly impossible due to the tendency of the paper to curl. Therefore, most of the work was performed on AISI 52100 steel polished to about a 10^{-7} m rms (four micro-inch) finish. As will be shown below, this also (a) permitted evaluation of spraying techniques, (b) permitted easy evaluation by visual observation of a material's tendency to self-level and/or separate, (c) provided a hard substrate for qualitative hardness checks, and (d) provided a very sensitive means of detecting water penetration of the film (since 52100 rusts quite easily).

In most cases, the materials and formulations (diluted to sprayable consistency) were applied to the discs, ambient temperature cured for two to four hours, checked for oil and water contact angles, exposed to 100 percent relative humidity (or soaked in water per the tabular data sheets) and rechecked.

Approximately five microliter-sized drops of distilled water and a highly purified hydrocarbon oil (Apiezon C) were applied to the horizontal surfaces of the discs. Contact angles of these materials were then measured using a Bausch and Lomb stereo microscope with a Unitron Model PTV Goniometer eyepiece.

Table 4-1
INFRARED ANALYSES AND OTHER MATERIAL CHARACTERISTICS

Material	Vendor or Supplier	Infrared Analysis	Solvent(s) as Supplied	Other Solvents and/or Diluents	Toxicity as Supplied	pH as Supplied	Nonvolatile Solids Content (kg/t)	Drying Rate (Tack Free)	Other Comments
Aliquat H-226	General Mills	High Mol. Wt. Linear Hydrocarbon/Amide	Paste with Isopropanol	Water Alcohols	Low	8	Not measured	Days	Remains as a waxy film
Aliquat 264	General Mills	Same as H-226 with Ketone groups	Emulsion with isopropanol	Water Alcohols	Low	8	Not measured	Days	Remains semi-fluid
39HC	Sandoz Chem	Aliphatic Amide with organic acid salt	None	Water Alcohols	Low	8	Not measured	Solvent Evap time	
39S HC	Sandoz Chem	Same as 39HC with ether groups	Solution with water/alcohol	Water Alcohols	Low	6	Not measured	Solvent Evap time	
Cartaretin-F	Sandoz Chem	Pure Polyamide	Water	Water Alcohols	Low	8	0.38	=1 hour	
Viscospin-B	Sandoz Chem	Similar to 39S	Water	Xylene Hexane	Low	8	Not measured	Days	Remains semi-fluid
Chemsheen	Chemfil Corp	Aliphatic Amide/Ether/Acid Salt mix	Water		Low	9	Not measured	Days	Remains as a fluid/solid mix
Frekote 33	Frekote, Inc.	Hydroxyl Rich Silicate/Silicone	P-Dioxane, Chloroethane	VMP Naphtha	Rather high due to solvent	Non-Aqueous	0.006	10 min.	
FC-321	3M	Fluoroacrylate Ester with Organic Acid Salt	Chloroethane	VMP Naphtha	Same as Chloroethane	Non-Aqueous	0.30	=30 min.	
FC-210	3M	Fluoroacrylate/Amide with Sulfonate	Water Emulsion		Low	7	0.39	=1 hour	
Nybar F	W. F. Nye, Inc	Fluorocarbon/Ether/Acrylate & Acid Salt	Chloroethane		Same as Chloroethane	Non-Aqueous	0.030	1 min.	
DC92-009	Dow-Corning	Polymethyl Silicone	Naphtha	VMP Naphtha Hexane	Low	Non-Aqueous	0.28	=30 min.	
Formula 125	Transcon R&D	Methyl Silicone and Hydroxyl Rich Carbonate	Water		Low but burn hazard due to pH	12	0.66	=1 hour	
Z-6079	Dow-Corning	Hexamethyl Disilazane	Pure Material	Hydrocarbons	High	Non-Aqueous	Could not measure		Evaporates too fast to be useful
DC772	Dow-Corning	Same as Formula 125	Water		Low but burn hazard	12	0.71	=1 hour	
Dri-Sil 73	Dow-Corning	Reactive Methyl Sil. & Hydroxyl Rich Alc.	Mineral Spirits	Hexane, VMP Naphtha	Low	Non-Aqueous	0.66	Function of Relative Humidity	
TT-P-115D, Ty. II	Goodyear Chem	See Fed Spec, Spectra on file	Mixture	VMP Naphtha	Low	Non-Aqueous	0.85	30 min max per Fed Spec	
Mod TT-P-115D (No TiO ₂)	Goodyear Chem	See Fed Spec, Spectra on file	Mixture	VMP Naphtha	Low	Non-Aqueous	0.81	30 min max per Fed Spec	
TT-P-85D	Kwal Paints (Denver)	Proprietary	Mixture	VMP Naphtha	High (long term); 0.5+ percent lead	Non-Aqueous	1.02	40 min max per Fed Spec	
Silanox 101	Cabot Corp	Spectra not possible; is a silanized silica	None	No solvents, will mix	Low	Non-Aqueous	Dry powder 0.046		
Monoaluminum Phosphate	Stauffer Chem	Spectra on file	Water		Low but acid hazard	2	Not measured	Water Evap time	
Silicates	Philadelphia Quartz	Spectra on file	Water		Low	10	Not measured	Water Evap time	
NRL Fluoro-epoxy	Naval Research Laboratory	Spectra not possible	None		Low	Solid film	Received as solid film		
Xylan 2052	Whitford Corp	Fluorinated Benzyl Acryl Amide	Dimethylformamide Freon TF		High due to solvent	Non-Aqueous	0.40	=3 hours	Complete cure requires days
SS-4044	General Elec	Dimethyl Silicone, Adhesive type	Ether/IEK/Xylene/Alc. mix	VMP Naphtha	Typical of solvents	Non-Aqueous	0.103	10 min	
DC732	Dow-Corning	Trifunctional, adhesive type silicone	None	VMP Naphtha Alcohols	Low	Non-Aqueous	0.98	10 min	
RIV 11	General Elec	Polydimethyl Silicone w/Pigment and Carbonate	None	VMP Naphtha Alcohols	Low	Non-Aqueous	1.13	=1 hour	Requires catalyst
Arothane 190450	Ashland Chem	Alkyd Type Ester/Urethane Copolymer	Naphtha	VMP Naphtha Alcohols	Low	Non-Aqueous	0.57	30 min	
Aroton 376	Ashland Chem	Modified Safflower Resin	Water Butyl Alcohol; Butoxy Ethanol	VMP Naphtha Alcohol	Low	7	0.57	2 hours	
Aroton 585	Ashland Chem	Modified Safflower Resin	Water		Low	7	0.55	10 min	
Petroset AT	Phillips Petroleum Co	Styrene-Butadiene Copolymer w/sulfonated Hydrocarbon	Water Emulsion		Low	7	0.63	Days	
G31, 2X	BBRC	Proprietary	Freon TF		Low	7	0.26	=45 min	Requires catalyst
G31 Thin	BBRC	Proprietary	Freon TF		Low	7	0.62	=1.5 hours	Requires catalyst

2.2 Numerical Data

The numerical contact angle data are presented in Tables 4-2 through 4-6. Table 4-2 compares data from the literature with measurements at BBRC as a check on the technique used here. The agreement is seen to be quite good where comparisons could be made.

Table 4-2
CONTACT ANGLES OF WATER ON VARIOUS SUBSTRATES

Material	Literature Data		BBRC Data	
	Contact Angles (Degrees)	Reference	Contact Angles (Degrees)	Date
Stainless Steel Mirror	11	43	11	5/16/74
Teflon	97	44		
Aluminum	68	44		
Glass	34	44	29/30/28	6/25/74
Control Plate (High Nickel Steel)			37/38	5/16/74
Plate A2 (High Nickel Steel)			37/37	5/16/74
Disk 64 (High Nickel Steel)			36/38	5/16/74
High Nickel Steel Disk			42/40	5/16/74

Table 4-3 is essentially a compilation of failed attempts to utilize various materials having specific advantages, such as low cost or low inherent pollution during application (e.g., water-based systems). The difficulty in curing these systems at ambient temperature to a hydrophobic condition is evident. Chemical compatibility problems are also evident.

Tables 4-4, 4-5 and 4-6 give the numerical contact angle data for basic materials, binders and formulations under various conditions. Note especially the poor results for most of the cationic surface active agents. While a monolayer of one of

Table 4-3
MATERIALS OR FORMULATIONS REJECTED FOR FURTHER CONSIDERATION

Material	Reason for Rejecting	Date
39 HC liquid	Very corrosive to steel	5-1-74
RU Silicate	Washes off in H ₂ O	5-1-74
Scotchgard 210	Corrosion; poor wetting	5-1-74
Triton X 100 + 50/50 H ₂ O Silicate	Not miscible	5-2-74
Silicate RU + Merix	0° γ	5-2-74
FC 431 + 50/50 H ₂ O Silicate	Not miscible	5-2-74
DC772 + Silicate + Teepol	Washes off in H ₂ O	5-2-74
Dri-sil 73 + Silicate + Teepol	Not miscible	5-2-74
92-009 + Silicate + Teepol	Not miscible	5-2-74
DC772 + Formula 125	Washes off in H ₂ O	5-2-74
Cartaretin F4 + Silicate + Teepol	Reacts with silicate	5-2-74
Frekote 33 + Silicate + Teepol	Not miscible	5-2-74
39S + Silicate + Teepol	0° γ	5-2-74
Scotchgard 210 + Silicate + Teepol	0° γ	5-2-74
Formula 125 + Silicate + Teepol	0° γ	5-2-74
Dri-sil 73 + Silicate + Teepol	Not miscible	5-2-74
Z-6079 + Silicate + Teepol	Not miscible	5-2-74
39S + Silicate	0° γ	5-3-74
Cartaretin F4 + Silicate	Sets off silicate	5-3-74
Frekote 33 + Silicate	Not miscible	5-3-74
Scotchgard 210 + Silicate	0° γ	5-3-74
Formula 125 + Silicate	0° γ	5-3-74
DC772 + Silicate	Washes off	5-3-74
Dri-sil 73 + Silicate	Not miscible	5-3-74
92-009 + Silicate	Not miscible	5-3-74
DC772 + 39S + Silicate	0° γ	5-3-74
DC772 + Cartaretin F4 + Silicate	Reacts	5-3-74
DC772 + Scotchgard 210 + Silicate	Reacts	5-3-74
DC772 + Formula 125 + Silicate	Washes off	5-3-74
Z-6079	Too volatile	5-6-74
Dri-sil + FC321	Reacts	5-7-74
Dri-sil + 92-009	Much too soft	5-7-74
SWB42V2	Heavy corrosion of steel	6-2-74
Aro 585	Corrosion	6-2-74
Dri-sil + 92-009 + Catalyst	Tacky	6-12-74
Dri-sil + Versamid 125 + Catalyst	Sticky	6-12-74
TTP-115D + Dri-sil 73	Out, unless Dri-sil can be cured	6-12-74
TTP-115D + Viscospin B	Separated badly	6-12-74
Dri-sil + V125	Runny	6-12-74
Dri-sil 73 + 92-009 V125 + Catalyst	Separated; runny	6-12-74
Dri-sil 73 + 92-009 + V125	Sticky	6-12-74
Dri-sil 73 + VMP + XI-2104	Catalyst settled out	6-19-74
Petroset + Dri-sil 73	Not miscible	6-28-74
Petroset + Silicate RU	Sets off rubber	6-28-74
Petroset	0° γ	6-28-74
Petroset + H ₂ O	0° γ	6-28-74
Petroset + monoaluminum phosphate	0° γ	6-28-74
Petroset + FC210	0° γ	6-28-74
Formula 125	0° γ	7-1-74
190M + VMP + FC321	Stays tacky too long	7-29-74
ARO 376	Film destroyed by five hour soak in H ₂ O	6-2-74
DC772/F125; seven mixtures/wetting agents	ALL washed off by water	5-15-74
Dri-sil + catalyst XI-2551	Did not cure	7-26-74
TT-P-85D + 92-009	Reacts	8-12-74
TT-P-85D + DC732	Reacts	8-12-74

γ = Contact Angle

Table 4-4
CONTACT ANGLES OF OIL AND WATER ON PROSPECTIVE MATERIALS

MATERIALS	TEST FLUIDS		DATE	CONTACT ANGLES (DEGREES) Substrate Materials		OBSERVATIONS
	Oil	Water		Paper	Steel	
Aliquat H22-C	X	X	4-29-74 to 5-1-74	13,38,37 51,55	29,72 41	Wet in all cases; data not usable for surface energy
Aliquat 264	X	X	4-29-74 to 5-1-74	13,9 18,50		Wet in all cases; data not usable for surface energy
39 HC	X	X	4-29-74 to 5-1-74	9,22 59,66	56,70 69	Wet in all cases; data not usable for surface energy
39S	X	X	4-29-74 to 5-1-74	21,34 60,90	56	Wet in all cases; data not usable for surface energy
Cartaretin F-4	X	X	4-29-74 to 5-1-74	5,0 7,9	87,86,98 39	
Viscospin B	X	X	4-29-74 to 5-3-74	103,125 90,98	94,96,103,100,99 72	Wet in all cases; data not usable for surface energy
Frekote 33	X	X	4-29-74 to 5-3-74	103,125 90,98	94,96,103,100,99 72	(94°, 96°)*
FC-321	X	X	4-29-74 to 5-4-74	106,121 141,98	103,103,108,91,103,108, 89	
FC-210	X	X	4-29-74 to 5-13-74	106,93 103,109	81,107,99,55 86,79	Problems of even application
Nyebar F	X	X	4-29-74 to 5-1-74	89,111 97,97	100,99 81	Thin film giving erratic results
DC 92-009	X	X	4-29-74 to 5-2-74	88,124 71,99	103,96,103 62	Thicker films on steel look good
Formula 125	X	X	4-29-74 to 5-1-74	108,129 69,77	95,93 49	Uneven film evident
Z-6079						Too volatile
DC 732	X	X	4-29-74 to 5-1-74	72,62 60,77	7,6 45	Uneven film on nonporous surfaces very evident
Dri-sil + Silanox 101	X	X	4-29-74 to 5-13-74	142,129 42,75	105,109,117,107,119,100 43,47	Low energy but variable. (105°, 109°)*
RU-Silicate						No good until some way is found to accelerate cure
G-31, 2X	X	X	5-1-74 to 5-3-74	90 70	100,97,113,93 73	Complete cure doubtful. (93°, 100°)*
G-31, Thin	X	X	5-1-74 to 5-3-74	119 69	98,103,102,95 72	Complete cure doubtful. (95°, 98°)*
Dri-sil + 92-009	X	X	5-4-74		78,87	Need more data
Cartaretin F-8		X	5-16-74		94	
DC 732		X	7-11-74		95,98,94,90,93,93	
			7-22-74		100,99,101	Without isopropanol
			7-22-74		108,108,109,111,111,110	Without isopropanol
			7-22-74		105,105,105	Without isopropanol
			7-22-74		105,104,103	With isopropanol, pour on
Xylan 2052 (24-hour air cure)		X	9-20-74		69,79,63,59	Five minutes exposure to water destroyed film

* After 24 hours at 100 per cent relative humidity, contact angle measurements of water on ice gave indicated values.

Table 4-5
CONTACT ANGLES OF OIL AND WATER ON PROSPECTIVE BINDER MATERIALS

BINDER MATERIALS	TEST FLUIDS		DATE	CONTACT ANGLES (DEGREES) Substrate Materials		OBSERVATIONS
	Oil	Water		Paper	Steel	
SWB 42V2	X	X	5-31-74		25,20 10,16	
TT-P-115D	X	X	5-31-74		87,86 10,11	
4044 Primer	X	X	5-31-74		92,91 51,49	After 24 hr 100% RH After five hrs underwater
	X	X	6-2-74		90,90	
	X		6-2-74		86,85	
Aro-190-M-50	X	X	5-31-74		83,89 14,12	After 24 hrs 100% RH After five hrs underwater
	X	X	6-2-74		90,87	
	X		6-2-74		29,19	
Aro 376	X	X	5-31-74		80,81 15,16	After 24 hrs 100% RH
		X	6-2-74		82,82	
Aro 585	X	X	5-31-74		75,73 15,17	After 24 hrs 100% RH
		X	6-2-74		62,65	
Modified TT-P-115D	X	X	7-22-74		100,87,80	Poured on
		X	7-22-74		82,84,83,82,83,84	Sprayed on
		X	7-22-74		88,90,90,92,89,89	
Petroset						Five discs with different concentrations. None indicated any degree of H ₂ O repellency
Sodium Silicate RU	X	X	4-29-74 to 5-1-74	0,0,48 10,19,41	16	Soaked into paper
Dri-Sil 73	X	X	4-29-74 to 5-2-74	84,114 56,61	95,88,85 35	

Table 4-6
CONTACT ANGLES OF OIL AND WATER
ON PROTECTIVE COATING FORMULATIONS

COATING FORMULATIONS:	TEST FLUIDS:		DATE	CONTACT ANGLES (DEGREES) Substrate Materials		OBSERVATIONS
	Oil	Water		Paper	Steel	
Monocalcium Phosphate + F 125		X			37	H ₂ O completely dissolved coating
Sodium Silicate, H ₂ O, Teepol						
• + Scotchgard FC210		X	5-2-74		19,19	
• + 39S liquid		X	5-2-74		0,7	
• + F 125		X	5-2-74		0	
• + Merix		X	5-2-74		0	
• + DC772		X	5-3-74		0	
• + 92-009		X	5-3-74		6	
Sodium Silicate, H ₂ O						
• + 39S		X	5-3-74		0	
• + Scotchgard		X	5-3-74		0	
• + F 125		X	5-3-74		0	
• + DC 772		X	5-3-74		60	Washes off in seconds
DC 772 + 39S		X	5-2-74		0	
• + F 125		X	5-2-74		26	
Dri-Sil 73 + 92-009		X	5-2-74		78,87	Sticky; put in 100% RH to cure. Contact angles measured after cure.
Dri-Sil + Silanox 101	X	X	5-6-74 to 5-13-74		100,129,117 47	
Dri-Sil 73 + RTV 11	X	X	5-20-74		100,100,99,98 63,63,65	Cured 2 hrs @ 100 C
	X	X	5-20-74		101,99,100 68,68	Cured 2 hrs @ 100 C
	X	X	5-22-74		98,98,98,101,101 98,99,98,99	Room temperature cure
	X	X	5-22-74		60,61,56,57 98,106	Room temperature cure
Dri-Sil 73 + RTV11 + Therm 12	X	X	5-22-74		97,97	
• + V-125 (Versamid -125)		X	5-22-74		106,103,102	Very Slow Curing
• + Viscospin B		X	5-22-74		105,105,107	Very Slow Curing
Dri-Sil 73 + RTV11 + Cat + Viscospin B		X	5-22-74		63,62,75,85,79,66	
Dri-Sil 73 + RTV11 + Cat + V-125	X	X	5-22-74 5-24-74		98,106,106 99,99,98 46,42,48	V-125 not completely dissolved
Dri-Sil 73 + 92-009	X	X	6-12-74		96,98,99	
	X	X	6-13-74		71,73,50,66 100,93,8 44,36	After overnight H ₂ O soak
Dri-Sil 73 + V-125	X	X	6-12-74		82,63,78 38,50,44	Still runny
	X	X	6-13-74		91,83,89 63,56	After overnight H ₂ O Soak
Dri-Sil 73 + 92-009 + V-125	X	X	6-12-74		103,110,98 73,90,61	
	X	X	6-13-74		99,90,99 74,60	After overnight H ₂ O soak
Dri-Sil 73 + Catalyst	X	X	6-12-74		85,91,85 40,41,41	
Dri-Sil 73 + 92-009 + Catalyst	X	X	6-12-74		85,89,90 68,68,73	
	X	X	6-13-74		104,109,97 60,54	After overnight H ₂ O soak
Dri-Sil 73 + V-125 + Catalyst	X	X	6-12-74		66,78,88 36,41,38	
	X	X	6-13-74		86,86,95 55,49	After overnight H ₂ O soak
Dri-Sil 73 + VMP + DC732 + Isopropanol		X	7-22-74		104,103,103	
Dri-Sil 73 + 92-009 + V-125 + Catalyst	X	X	6-12-74		108,98,100 60,62,80	
	X	X	6-13-74		100,101,103 56,63	After overnight H ₂ O soak

Table 4-6 CONTACT ANGLES OF OIL AND WATER
ON PROTECTIVE COATING FORMULATIONS (Continued)

COATING FORMULATIONS	TEST FLUIDS		DATE	CONTACT ANGLES (DEGREES) Substrate Materials		OBSERVATIONS
	Oil	Water		Paper	Steel	
Dri-Sil 73 + V-125 + Catalyst	X	X	6-12-74		66,78,88)After overnight H ₂ O soak
	X	X	6-13-74		36,41,38 86,86,95 55,49	
FC321 + 4044		X	5-5-74 to 6-13-74		108,91,113	
TT-P-115D + Dri-Sil 73	X	X	6-12-74		107,97,93)After overnight H ₂ O soak
	X	X	6-13-74		34,40,38 74,102,93 34,35	
TT-P-115D + 92-009 50/50 Xylene	X	X	6-12-74		91,100,102)After overnight H ₂ O soak
	X	X	6-13-74		62,43,64 113,111,113 33,37	
TT-P-115D + Viscospin B	X	X	6-12-74		11,17,18)After overnight H ₂ O soak
	X	X	6-13-74		55,50,20 21,20,23 24,27	
TT-P-115D + 92-009	X	X	6-12-74		110,107,110)After overnight H ₂ O soak
	X	X	6-13-74		74,85,83 108,108,105 55,61	
TT-P-115D + 4044	X	X	6-12-74		90,90,93)After overnight H ₂ O soak
	X	X	6-13-74		45,47,40 84,88,91 39,43	
	X	X	7-22-74		105,103,103,115,113, 113	
	X	X			105,104,104	
TT-P-115D + Frekote (Straight)	X	X	6-12-74		90,90,87)After overnight H ₂ O soak
	X	X	6-13-74		44,38,40 96,85,97 30,31	
TT-P-115D + DC732		X	7-22-74		119,119,116,115,115, 114,119	
		X			119,117,115	
Cartaretin F4 + F-125		X	5-14-74		87,87	Washes off in one minute
Cartaretin F4 + F-125 + Cellosolve acetate		X	5-14-74		89,86	
Cartaretin F4 + DC732		X	5-14-74		0	
Cartaretin F4 + DC732 + Cellosolve acetate		X	5-14-74		0	
Cartaretin F4 + F-125 + Isopropanol		X	5-14-74		97,90	
Cartaretin F4 + DC732 + Isopro- panol		X	5-14-74		0	
Cartaretin F4 + F-125 + Isopro- panol		X	5-16-74		94	
190MS0 + Dri-Sil 73	X	X	6-12-74		93,97,95)After overnight H ₂ O soak
	X	X	6-13-74		48,42,43 96,97,90 40,36	
190MS0 + 92-009	X	X	6-12-74		100,103,102)After overnight H ₂ O soak
	X	X	6-13-74		76,68,60 98,110,95 50,36,36	
190MS0 + 4044	X	X	6-12-74		93,95,92)After overnight H ₂ O soak
	X	X	6-13-74		49,50,49 83,80,85 36,25	
190MS0 + Frekote (straight)	X	X	6-12-74		94,92,91)After overnight H ₂ O soak
	X	X	6-13-74		59,57,50 93,92,95 45,44	
	X	X	7-22-74		93,91,90	
Cartaretin F4 + ARO 376						No data
Modified TT-P-115D + 4044		X	7-22-74		111,107,107,109,111, 110	
		X			106,105,104	

these materials might exhibit hydrophobicity due to molecular orientation (see Chapter 2, Section 1.2.1), a layer of finite thickness (required to exhibit any wear life whatever), is definitely hydrophilic. Note that contact angle data employed to detect changes during a given screening test are given in later portions of this chapter.

Finally, it was mentioned in Chapter 2, Section 3, that contrary to theory, contact angle data could not be used to estimate solid dispersion energy. Using the data for DC92-009* on steel, where the water contact angle was about 100 degrees and the oil contact angle was 62 degrees, we compute from Chapter 2, Section 3, formulas 7 and 8:

$$\gamma_{S_{H_2O}}^d = 40 \text{ ergs/cm}^2$$

$$\gamma_{S_{oil}}^d = 19 \text{ ergs/cm}^2$$

These should be the same. Whether impurities in the film (we must deal with commercial products) are responsible is not known. More likely, the "maximum contact angle" phenomenon discussed in Chapter 9 of Reference 3 is responsible. After this discovery, the water contact angle alone was used to rank coating hydrophobicity.

2.3 Photos

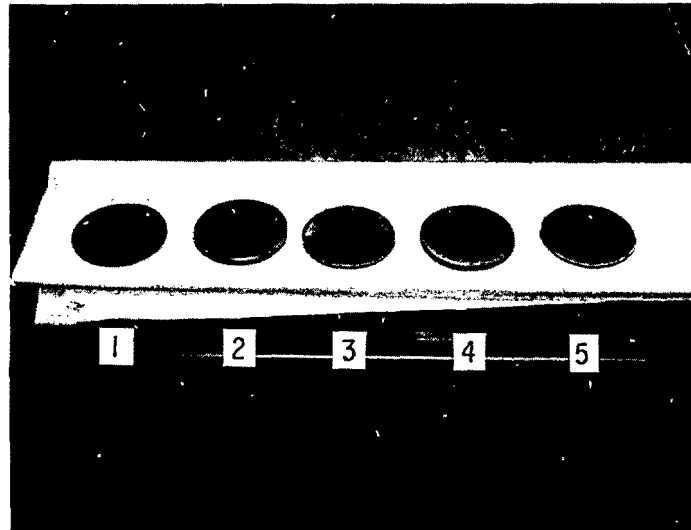
Figures 4-1, 4-2, 4-3 and 4-4 illustrate some of the above discussion.

Figure 4-1 shows:

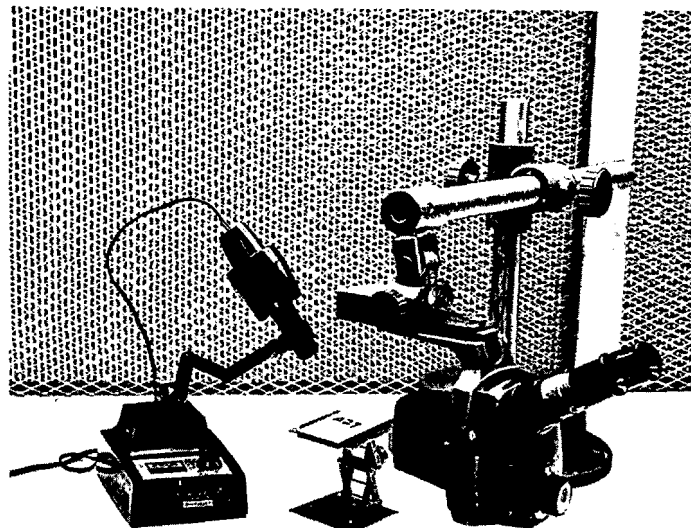
4-1a: Five formulations of Petroset AT on discs (straight, 50/50 diluted with water, 30/70 diluted with water, 30/70 diluted with water plus monoaluminum phosphate and with FC-210). Note differences in appearance.

4-1b: The test setup for measuring contact angles.

* This material, like many other silicones, does not depend on surface active "ends" to orient on a surface and become hydrophobic. Rather, the molecule is surrounded by $-CH_3$ groups and is thus inherently hydrophobic.



(a)



(b)

Figure 4-1 Steel Test Discs and Contact-Angle-Measurement Apparatus

Figure 4-2 shows:

- 4-2a: A water droplet on untreated stainless steel (14X).
- 4-2b: A water droplet on Frekote 33 with the goniometer scale visible (16X).
- 4-2c: A water droplet on modified TT-P-115D paint (14X).

Figure 4-3 shows:

- 4-3a: A water droplet on DC92-009 (14X). Note the refraction. The angle really is greater than 90 degrees.
- 4-3b: Water droplets on DC92-009 (7X) as used for triplicate measurements.
- 4-3c: A water droplet on Petroset AT (14X).

Figure 4-4 shows:

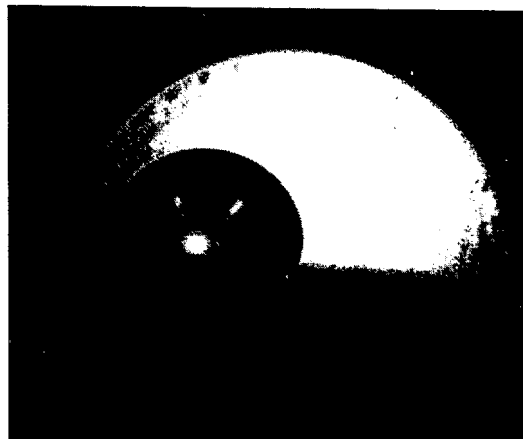
- 4-4a: Badly degraded Cartaretin F-4 after the first freeze/thaw cycle during ice adhesion testing.
- 4-4b: A typical array of binders (SWB 42V2, TT-P-115D, Arothane 190M50, Arolon 376, Arolon 585 and SS 4044) after high humidity exposure. Note rusting indicative of water vapor penetration.
- 4-4c: A typical array of materials after water soaking. Note corrosion, peeling and bubbling indicative of low hydrophobicity.
- 4-4d: A group for plates coated for the first series of ice adhesion tests.

2.4 Conclusion

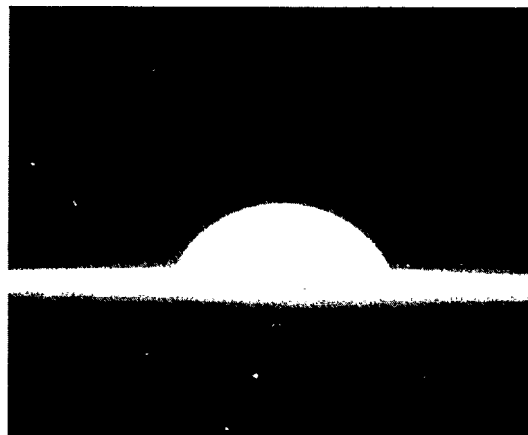
Space does not permit discussion of the mass of data presented above. It is sufficient to state that the data are available for detailed examination, that water (and oil) contact angle measurements permitted rapid screening of a very large number of materials and formulations and that the steel discs proved to be a valuable indicator of coating water resistance.



(a)

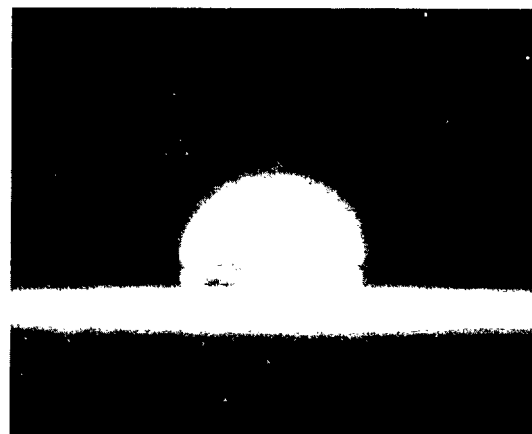


(b)

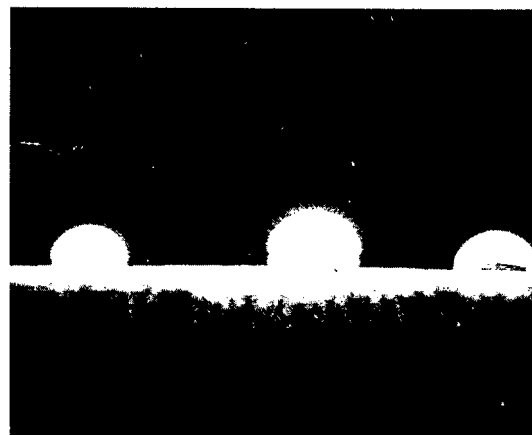


(c)

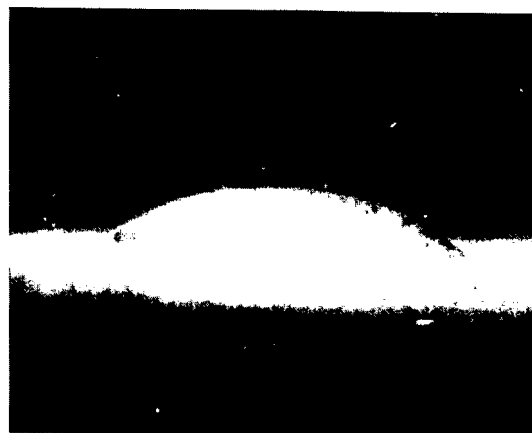
Figure 4-2 Water Droplets on Various Materials for Contact Angle Measurements



(a)



(b)



(c)

Figure 4-3 Water Droplets on Various Materials for Contact Angle Measurements

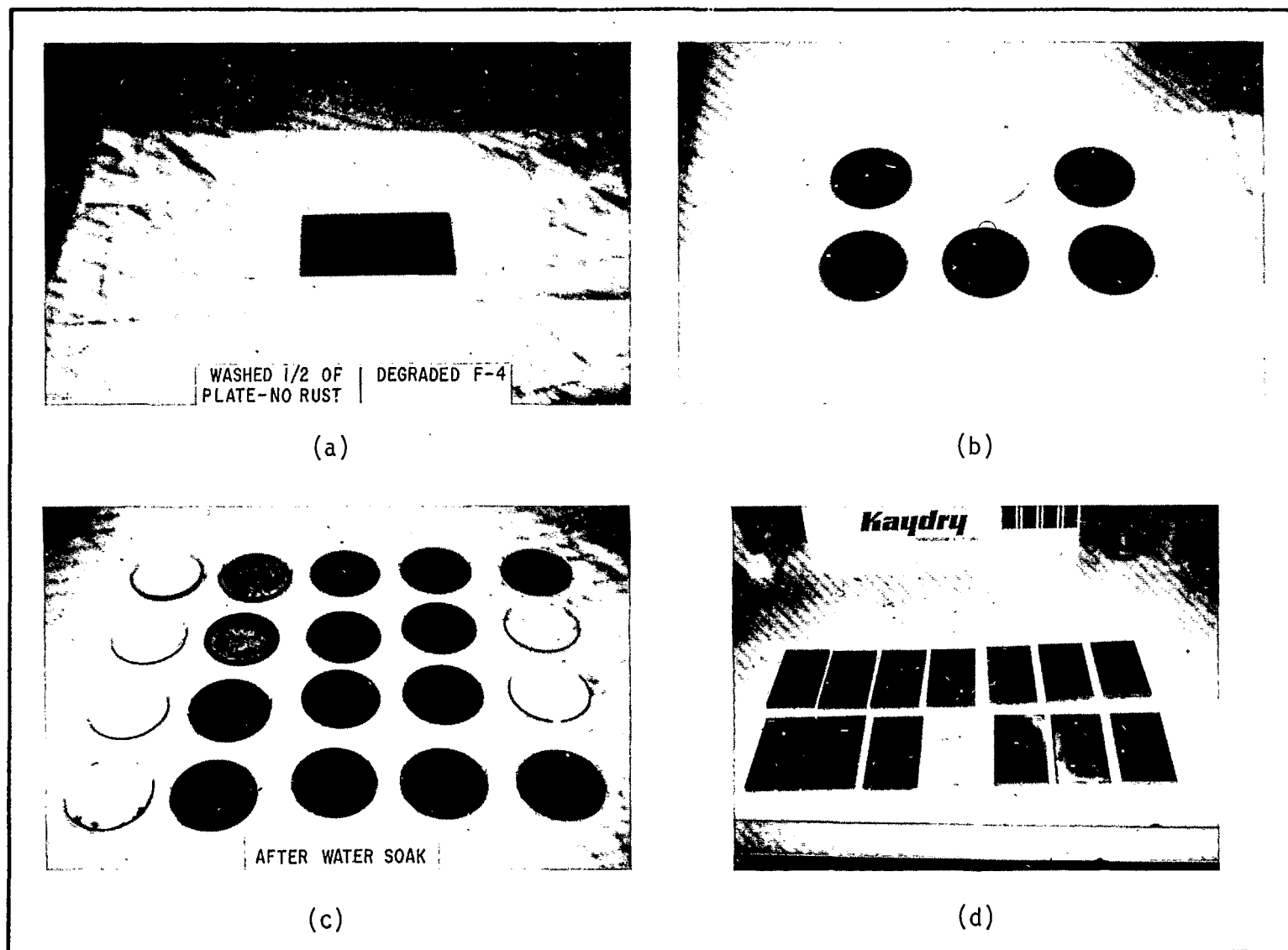


Figure 4-4 Test Discs and Plates Used for Evaluation of Various Materials.

3.0 COMMENTS ON OTHER TESTS

Following the tests and observations described above, the following decisions were made:

- Since sufficient data (solids content, preferable solvents for application and the results of various spray techniques) were now available to compute the solid film to be expected from a given quantity of formula, all subsequent tests were performed on sprayed films having a thickness of about 10^{-4} m (0.004-inch). From Chapter 3, Section 5.2.2, this is about the maximum thickness that could be employed without the danger of decreasing lowspeed highway friction coefficients.
- Many of the materials were discarded from further consideration. This included most of the cationic surface active agents, most of the water-based materials and some of the fluorochemicals.

4.0 ICE ADHESION SCREENING TESTS

The four series of laboratory ice adhesion screening tests are described in this section. The data are from References 55, 56, 57 and 58 included in their entirety in Appendix A. These basic property data were required to rank the various materials and combinations for suitability as ice release agents and for cohesive strength during release.

4.1 Test Procedure

For series 1 and 2, the coatings were applied as specified in 3.0 above to steel plates (5 cm by 10 cm by 0.63 cm) having an rms surface roughness of about 1.3×10^{-6} m (50 microinch). For these two series, the plates were subjected to four hours at 100 percent relative humidity and the water contact angles were checked before testing. In series 3, asphalt and concrete cores were tested to obtain reference values. In series 4, a few formulations were sprayed on cores and tested.

In all cases, the procedure was the same as is completely specified in Reference 55 (Appendix A). The high shear rate used (0.5 cm/sec) was selected as being more representative of highway speed than lower rates. However, such high rates give maximum values of adhesion (Reference 31) and thus may

be pessimistic. The test temperature of -12 C was selected to avoid the hypothesized "liquid-like" interface (Reference 26) between -10 C and 0 C.

4.2 Numerical Data

The numerical data are summarized in Table 4-7. The individual release values (three trials of two releases per trial) are given in the second column. Actual coating thicknesses are listed in the third column. For rapid comparison, the average shear strength and the range are given in the fourth and fifth columns. Contact angle data are given in the next three columns and the last two report qualitative observations.

In series one and two, most coatings gave some reduction in adhesion but few were outstanding.

In series three, cores were taken from Highway 36 (see Chapter 6 for locations of sites).

In series four, a few coatings were sprayed on cores and tested. The results were surprising and somewhat contradictory to series one and two. This emphasizes the recommendation that coating optimization should use only core samples as substrates. Further laboratory tests on such cores were not possible due to scheduling (series four was run in mid-October) and budget constraints.

4.3 Photos

Figure 4-5 shows:

- 4-5a. & 4-5b: The series four core samples before and after adhesion testing. Note that the TT-P-115D/DC 732 (center of each photo) was obviously sprayed on too heavily (compare with Figure 4-11d in Section 6 of this chapter) thus accounting for its poor performance.
- 4-5c: Ice adhesion cold cabinet/pull apparatus at Hauser Laboratories.
- 4-5d: Close-up of the Teflon ring ice holder/sample plate configuration.

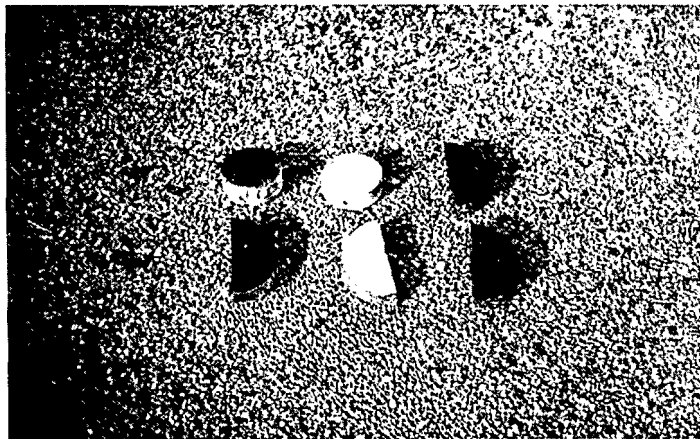
4.4 Conclusion

Ice adhesion testing revealed very wide differences between shear forces for the various coatings. Testing on steel

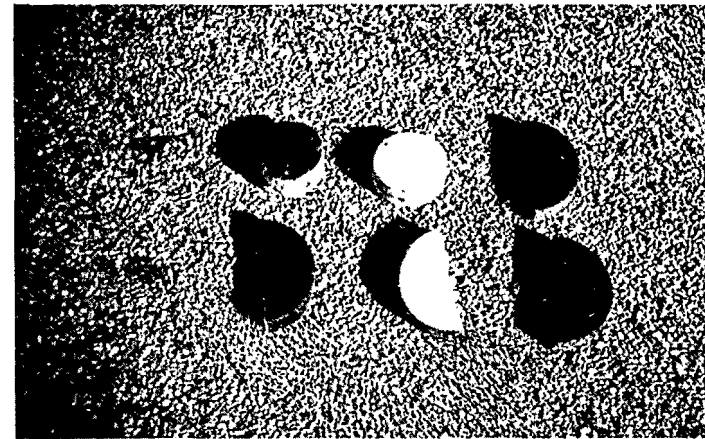
Table 4-7
ICE ADHESION SCREENING TEST RESULTS

MATERIAL	ADHESION STRENGTH kg/cm ²	FILM THICKNESS cm	AVERAGE STRENGTH kg/cm ² x̄	RANGE kg/cm ² R	CONTACT ANGLES - DEGREES			COATING REMOVAL BY ICE	COATING CONDITION AFTER ADHESION TEST
					AS APPLIED (H ₂ O/OIL)	AFTER 4 HR. #1001 R.H. (H ₂ O)	AFTER ADHESION TEST (H ₂ O)		
FIRST TEST SERIES (HAUSER REPORT 74-225) (REF. 55)									
NONE (STEEL PLATE)	7.24/5.62/7.03/8.15/7.45/8.15	-----	7.28	2.53	50/43				
CARTARETIN F-4	COATING REMOVED BY FIRST FREEZE/THAW CYCLE				80/40	83		COMPLETE	
DRI-SIL 73/RTV 11	6.68/6.96/8.95/7.03/7.31/4.78	0.0025	6.94	4.15	99/		95,93,92	IN SPOTS	SOFT, POOR ADHESION
FC - 321	4.50/5.94/2.39/2.88/2.81/2.67	0.005	5.20	2.11	113/87	105	103,110,111	NIL	HARD, GOOD ADHESION
DRI-SIL 73/ SILANOX 101	17.9/12.8/14.8/24.6/7.10/7.59	0.010	14.1	17.5	117/47	103	117,117,116	90% COMPLETE	BRITTLE, POOR ADHESION
NYEBAR F	4.15/6.75/3.87/4.57/4.15/3.87	0.00075	4.55	2.88	103/76	97	98,93,101	NIL	TOUGH, GOOD ADHESION
NEL COATING	7.31/5.62/2.32/4.57/5.20/6.26	0.010	5.21	4.99	90/40	88	52,88,87	NIL	COATING UNCHANGED
FREKOTE 33	6.68/5.06/5.51/4.43/5.34/3.87	0.00025	4.81	3.16	97/78	97	106,105,106	NIL	HARD, EXCELLENT ADHESION
FC210 WITHOUT FC-43	4.99/7.31/5.13/6.26/4.29/3.65	0.010	5.27	3.65	95/		88,101,102	NIL BUT MARKED	HARD, GOOD ADHESION
G31 - 2X	3.51/2.46/2.88/2.81/2.04/1.26	0.010	2.49	2.25	98/60	98	95,93,99	NIL	TOUGH, GOOD ADHESION, SLIPPERY
DC 92-009	1.55/1.55/1.05/1.26/0.91/0.98	0.0037	1.22	0.63	94/70	97	104,100,96	NIL	TOUGH, GOOD ADHESION
DRI - SIL 73	9.28/7.24/9.35/8.58/5.27/5.20	0.0025	7.49	4.15	97/33	91	104,98,92	5% REMOVED	HARD, GOOD ADHESION
FC210 WITH FC - 43	6.15/4.08/4.43/2.81/4.64/6.26	0.0005	4.73	3.44	81/79	80	91,75,83	NIL BUT MARKED	TOUGH, POOR ADHESION
DRI - SIL 73/92009	13.0/11.5/7.73/5.83/7.52/7.87	0.010	8.91	7.17	88/41	88	101,101,101	NIL BUT MARKED	SOFT, POOR ADHESION
G 31 - THIN	5.55/4.36/5.52/3.37/5.73/5.02	0.0025	3.92	2.53	92/72	92	103,95,99	NIL	TOUGH, GOOD ADHESION, SLIPPERY
SECOND TEST SERIES (HAUSER REPORT 74-319) (REF. 56)									
NONE (STEEL PLATE)	4.69/8.51/6.45/8.16/9.27/8.44	-----	7.58	4.59	56,65,63		*81,83,83	----	
TT-P-115D, TYPE II	7.17/7.94/5.51/4.19/8.15/8.29	0.018	6.88	4.11	86,86,92		*77,86,85	NIL	TOUGH
TT-P-115D/DC 732	5.22/8.65/14.9/13.2/3.30/3.80	0.010	8.69	11.7	106,108,105		*114,120,109	50% REMOVAL	SOFT AND FLAKY
TT-P-115D/4044	5.93/4.58/9.56/10.1/9.00/5.94	0.010	7.52	5.54	96,106,106		*105,105,102	NIL	TOUGH
TT-P-115D/DC92 - 009	4.62/4.52/7.87/5.83/6.16/4.94	0.012	5.66	3.35	102,106,106		*107,105,105	NIL	SOFT
TT-P-115D/FC - 321	4.12/3.42/7.46/4.13/7.31/6.92	0.025	5.49	4.04	118,118,118		109,111,104	NIL	VERY TOUGH
MODIFIED TT-P-115D TY 11	9.28/6.19/8.08/7.87/11.7/12.0	0.0075	9.19	5.83	75,83,85		*78,70,77	NIL	TOUGH
MOD TT-P-115D/DC 732	1.02/0.87/2.45/4.19/1.11/2.90	0.016	2.09	3.32	119,117,120		111,112,112	NIL	SLIGHTLY SOFT
MOD. TT-P-115D/4044	6.82/8.93/11.2/10.1/10.9/13.0	0.0075	10.2	6.19	104,110,106		*104,102,104	NIL	SLIGHTLY SOFT
TT-P-115D/FREKOTE 33	6.21/6.89/8.22/7.31/7.10/7.52	0.010	7.21	2.01	93,100,95		*108,110,104	NIL	VERY TOUGH
TT-P-85D	3.52/3.76/6.87/9.07/10.5/9.77	0.010	7.26	6.25	100,100,99		*97,95,99	NIL	VERY TOUGH
TT-P-85D/4044	7.17/5.80/9.77/7.73/9.28/9.07	0.0075	8.08	3.48	104,106,110		*103,106,107	NIL	TOUGH
DC 732	0.86/0.18/0.82/0.91/0.89/0.73	TACKY-NOT MEASURED	0.73	0.73	101,102,103		109,105,106	NIL	TACKY
190K50	4.84/3.65/4.51/8.01/4.15/8.22	0.005	5.56	4.58	78,86,86		*86,83,84	NIL	TOUGH
190K50/FREKOTE 33	4.15/3.97/7.24/4.67/8.01/7.24	0.0025	5.91	3.27	76,80,73		77,86,86	NIL	TOUGH
190K50/4044	4.44/5.71/3.99/6.57/4.15/4.76	0.0075	4.94	2.58	91,82,86		93,89,90	NIL	VERY TOUGH
OMP. C	3.39/5.37/6.03/6.05/7.66/6.62	0.0005	5.85	4.27	*99,99,104		98,101,101	NIL	SOFT, SLIPPERY
THIRD TEST SERIES (HAUSER REPORT 74-343) (REF. 57)									
Reference Data									
CONCRETE:			12.95	3.48					
ASPHALTIC PAVEMENT:			13.45	5.09					
FOURTH TEST SERIES (HAUSER REPORT 74-381) (REF. 58)									
MOD. TT-P-115D/DC 732									
ON ASPHALT:	7.4/9.3/9.5/10.7/11.0/9.5	NOT KNOWN BUT THICK	9.6	3.6				20%	
ON CONCRETE:	8.0/6.2/5.3/5.9/13.0/7.4	NOT KNOWN BUT THICK	7.6	7.7				20%	
PETROSET AT									
ON ASPHALT:	13.3/8.8/9.1/11.2/10.4/10.7	NORMAL	10.6	4.5				NIL	
ON CONCRETE:	9.5/10.2/9.1/7.4/14.3/10.0	NORMAL	10.1	6.9				NIL	
DRI-SIL 73/DC 732									
ON ASPHALT:	4.8/4.5/4.7/3.4/6.3/7.4	NORMAL	5.2	4.0				NIL	
G 31 - THIN									
ON ASPHALT:	10.3/8.5/5.6/10.3/8.5/9.4	NORMAL	8.8	4.7				NIL	

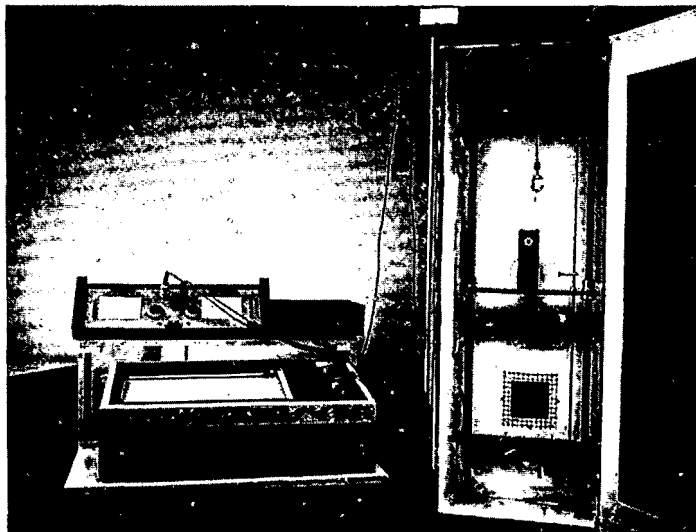
*Wetting by H₂O During Measurement



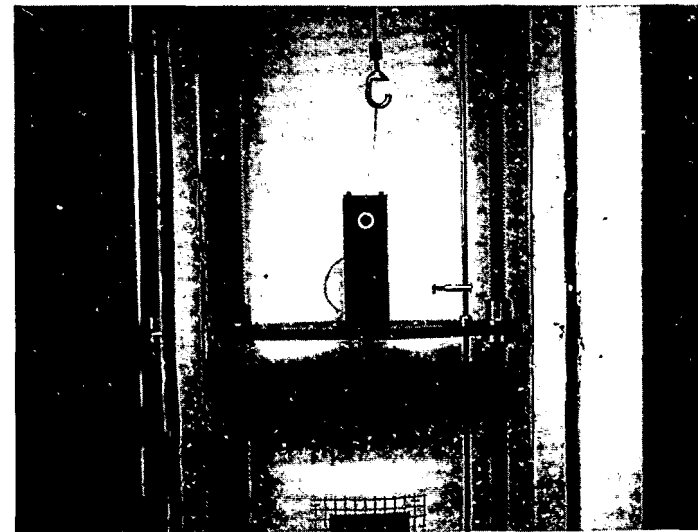
(a)



(b)



(c)



(d)

Figure 4-5 Ice Adhesion Core Samples and Test Apparatus

substrates gave wider differences between the coatings and may thus be more representative of the coatings themselves. However, from limited tests on asphalt and concrete surfaces, the use of a steel (or any nonporous) substrate appears to give optimistic values in this application.

Coatings with the worst (highest adhesion) performance (like Cartaretin F-4 and Silanox 101) have also been shown to be oleophilic and a serious threat to asphaltic surfaces (see Section 6 below).

Finally, in agreement with Reference 2, no strong correlation was found between ice adhesion and water contact angle.

5.0 HIGHWAY WEAR TESTS

The results of the highway surface wear tests are presented in this section. These data were required to estimate the coatings' wear lives as required by the basic contract's cost/effectiveness goal of a once-per-season application.

5.1 Procedure

Highway wear tests were performed on the most promising formulations at this point in time to obtain realistic traffic life estimates. The location of the tests, the strip pattern and configuration and spacing, are defined in Appendix C, Figures C-2 and C-3.

It should be noted that air-type spray techniques were used in applying the stripes. This is known to give poorer adhesion and penetration than the airless spray technique finally adapted for the full scale highway tests (see Chapter 6).

5.2 Data and Results

All data are presented in Table 4-8 for the first 35-day observation period.

Note that:

- Wear was moderate (20 - 30 percent wear).
- From skid values, all coatings were still effectively present after 200,000 vehicle passes.
- Both Arothane formulations exhibited unsatisfactory (low) skid values.
- TT-P-115D/DC 732 held up well on concrete.

Table 4-8
HIGHWAY WEAR TESTS - CITY OF BOULDER

MATERIAL	SUBSTRATE	APPLICATION/ OBSERVATIONS	INSPECTION DATA			STRIPE NUMBER (SEE PHOTOS)	CONCLUDE	
			SKID VALUES	WETTING* OBSERVATIONS	WEAR ESTIMATES			
			ASTM E303	(With H ₂ O)	(Vol. %)			
TT-P-115D, TY,II	ASPHALT	1. ALL MATERIALS EASILY SPRAYED WITH SLIGHT PLUGGING OF DC732 MIXES.	60/59/59/59	SOME BEADING	30		A1	FROM SKID DATA AND VISUAL OBSERVATION, ALL COATINGS ARE STILL EFFECTIVELY PRESENT. NO DEGRA- DATION HERE FOR MATERIALS SHOWING ASPHALT DEGRADATION IN FRICTION TESTS.
TT-P-115D/4044	ASPHALT		54/55/54/55	SOME BEADING	20		A2	
TT-P-115D/92-009	ASPHALT		59/58/58/59	SOME BEADING	30		A3	
MODIFIED TT-P/FC - 321	ASPHALT		62/61/60/60	SOME BEADING	30		A4	
MODIFIED TT-P/DC 732	ASPHALT	2. ASPHALT VERY COM- PACTED SO PENETRA- TION NOT GOOD.	68/68/66/68	SOME BEADING	30		A5	
MODIFIED TT-P/92-009	ASPHALT		60/62/60/61	NO OBVIOUS BEADING	30		A6	
MODIFIED TT-P/4044	ASPHALT		62/61/62/62	SOME BEADING	20		A7	
AROTANE 190MS0/4044	ASPHALT		45/45/43/43	SOME BEADING	20		A8	
AROTANE 190MS0/FREKOTE	ASPHALT	3. CONCRETE VERY WEATHERED.	40/40/38/38	NO OBVIOUS BEADING	20		A9	
PETROSET AT	ASPHALT		57/57/53/56	NO OBVIOUS BEADING	20 (?) -	PENETRATION MADE WEAR ESTIMATE DIFFICULT	A10	
MOD. TT-P/4044	CONCRETE	4. PETROSET AT PENE- TRATION SLOW.	TOO ROUGH TO MEASURE	BEADING NOT OBVIOUS	10		C1	
MOD. TT-P/DC 732	CONCRETE			GOOD BEADING	5		C2	
AROTANE 190MS0/4044	CONCRETE			BEADING NOT OBVIOUS	20		C3	
PETROSET AT	CONCRETE			BEADING NOT OBVIOUS	20 (?) -	PENETRATION MADE WEAR ESTIMATE DIFFICULT	C4	

APPLICATION							
LOCATION: ARAPAHOE BETWEEN FOLSOM AND 28th STREETS							
OUTSIDE, EAST BOUND LANE AND (FOR CONCRETE) PARKING LOT ENTRANCE							
DATE: 8/1/74							
AREA: 10cm WIDE STRIPES ACROSS LANE AND ACROSS ENTRANCE PAD							
RATE: TO GIVE 0.010 CM THICK FILM							
METHOD: "JET-PACK", SPRAY CANS, SURFACE NOT SNEPT OR CLEANED							
TEMPERATURES: ASPHALT 33C / CONCRETE 28C							

INTERIM PERIOD							
EST. RAIN FALL: 15cm							
TEMPERATURES: MOSTLY 27C+							
TRAFFIC: ASPHALT: 204000 VEHICLE PASSES							
CONCRETE : 20000 VEHICLE PASSES							

INSPECTION							
DATE: 9/4/74				REFERENCE			
TEMPERATURE OF ASPHALT: 20C				SKID VALUES			
				65/65/66/64			

*WETTING (BEADING) OBSERVATIONS PROBABLY PESSIMISTIC DUE TO
ACCUMULATED DIRT ON THESE REAL SURFACES

5.3 Photos

Figure 4-6 shows four photos during strip application on 8/1/74, 4-6a, 4-6b for the asphalt and 4-6c and 4-6d on the concrete entrance slab.

Figure 4-7 shows the asphalt stripes at the end of 35 days. Note the non-wetting following skid testing (rectangular areas) in stripes A8, A9 and A10.

Figure 4-8 shows the asphalt stripes following an additional 40-day period. The paint formulation stripes are still about 50 percent present.

5.4 Conclusion

Long-term wear was least for the paint formulations. While the wear rate was somewhat higher than hoped, the results were encouraging in view of the non-optimum application technique and the type of traffic flow (considerable stop-start movement) at this location.

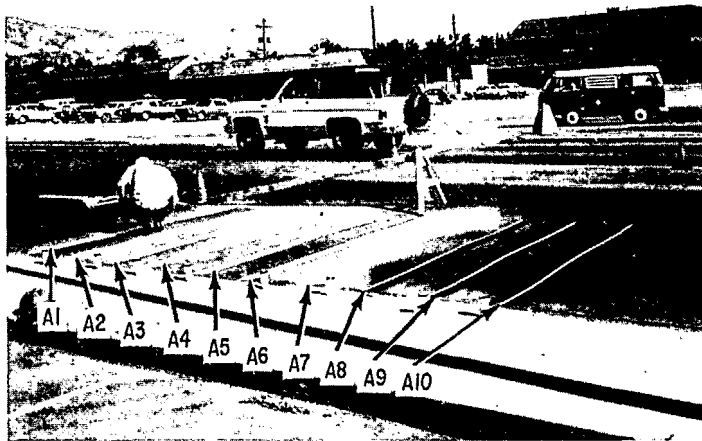
A complete environmental impact summary is given in Chapter 5. However, from these wear data, the amount of solid wear products can be estimated.

- From Reference 6 and the definition of a "standard" tire from ASTM E 249-66, the volumetric wear rate for rubber $\approx 3 \times 10^{-5} \text{ cm}^3$ of rubber/m travel. For 2×10^5 vehicle passes (see data above) or 8×10^5 tire passes, we have 24 cm^3 of rubber lost/m travel. Assuming asphalt wear is about the same, the amount of *rubber/asphalt debris* in a one-meter length of the test section after 200,000 vehicle passes $\approx 50 \text{ cm}^3$.
- For a coating thickness of 0.01 cm, we have about 300 cm^3 of material in a one-meter lane length. From the data for an average wear of 25 percent, *the solid coating removed* after 200,000 vehicle passes $\approx 75 \text{ cm}^3$.

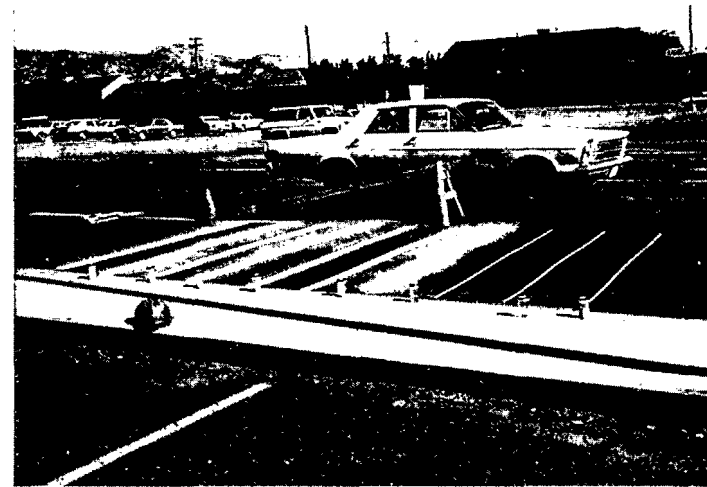
We conclude that the amount of solid debris from the coating is not much greater than that from normal rubber/asphalt highway wear.

6.0 FRICTION AND DEGRADATION TESTS

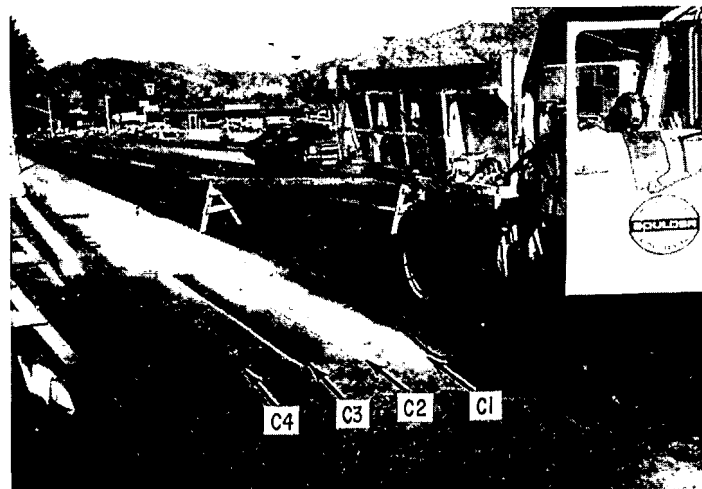
Over a four-month period, a large number of materials and formulations were applied to asphalt and concrete surfaces



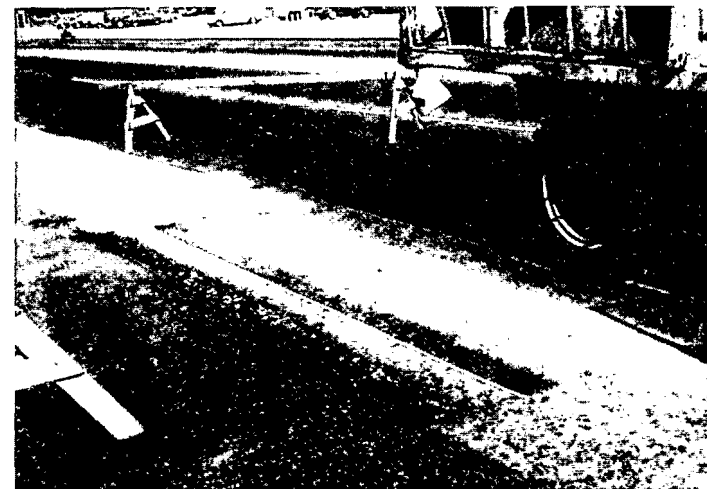
(a)



(b)

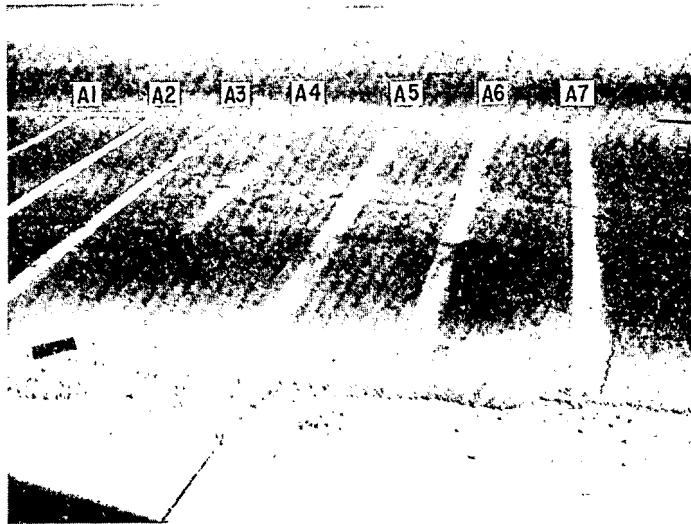


(c)

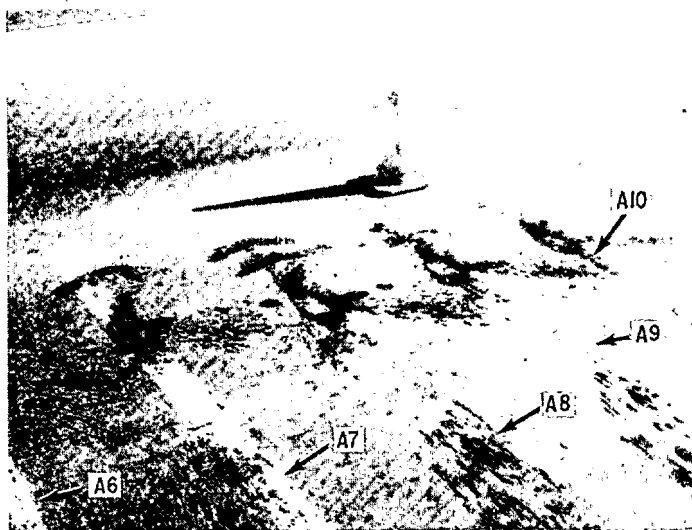


(d)

Figure 4-6 Application of Prospective Materials for Phase II Wear Tests



MATERIALS

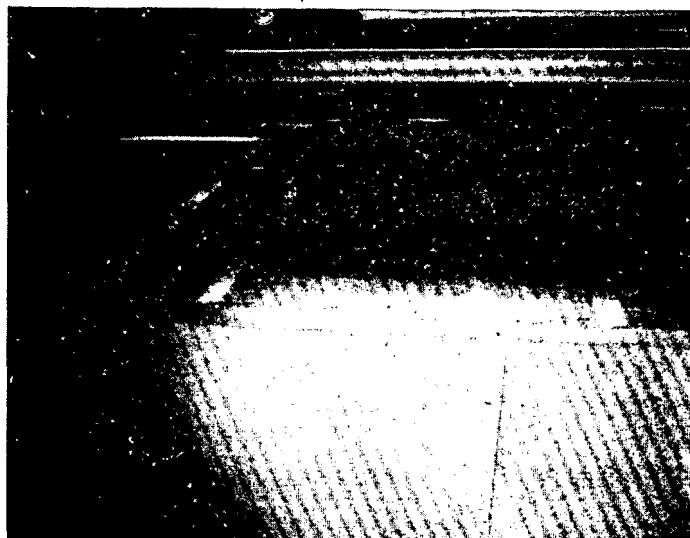


MATERIALS

Figure 4-7 Results of Phase II Wear Tests on Asphalt After 35 Days



(a)



(b)

Figure 4-8 Results of Phase II Wear Tests on Asphalt
After 75 Days

on BBRC premises. While the primary goal was the determination of coating friction characteristics on real surfaces, very useful data on coating and/or substrate degradation were also obtained during these outdoor exposure tests. These data were vital to establish that the coatings would not, in themselves, create a skid hazard.

6.1 Procedure

All coatings were applied by spray techniques to give 0.01 cm dried films. The site location, test area sizes and section numbering systems are defined in Appendix C, Figures C-2 and C-4.

6.2 Numerical Data and Observations

The numerical skid value measurements and degradation observations are presented in Table 4-9. The comments are self-explanatory. A quantitative discussion of the skid numbers is given in 6.4 below. In general, friction coefficient is proportional to the values and values below 45 are unacceptable from a safety standpoint. The variation of skid numbers with time is an indication of change.

Note the coatings that, even in this zero traffic situation, disappear or degrade with time (Formula 125, Cartaretin F-4, for example). Note also the materials that have unacceptable skid values (Arothane, Cartaretin F-4, Arolon) suggesting water solubility, since the skid tests are run with water on the surfaces. Note further that some of the unformulated silicone resins (DC92-009 and DC 732) are unacceptably slippery. Finally, note the components (Silanox 101, Arolon, Arothane and Cartaretin F-4) which degrade asphalt, indicating oleophilic solution of the asphaltic binder.

6.3 Photos

Photos indicating the test layout and some of the points made above are presented below.

Figure 4-9 (first asphalt tests) showing:

A-9a: Lot #1

A-9b: Lot #3 showing degradation already in progress

A-9c: Lot #5

A-9d: Lot #7

Table 4-9
FRICTION (ASTM E303) AND DEGRADATION TESTS

MATERIAL (SECTION)	SUBSTRATE/ DATE MTL. APP.	UNCOATED SUBSTRATE SKID VALUES				SKID TEST DATA						NOTES		ESTIMATED CONTACT ANGLES' H ₂ O
ALL MATERIALS APPLIED AT A RATE EQUAL TO A 0.010 cm. THICK FILM			TEST ONE			TEST TWO			TEST THREE			CONTACT ANGLES MEASURED 7/2/74	9/3/74	
			DATE	TEMP (°C)	VALUES	DATE	TEMP (°C)	VALUES	DATE	TEMP (°C)	VALUES			
A S P H A L T														
FIRST ASPHALT TESTS														
DRI-SIL 73 (#1)	ASPHALT/ 6/21/74	72/75/76/75	6/24	38	78/76/76/75	7/15	40	68/68/70/69	9/3	15	74/77/76/77	(86°)	60°	
AROLON 376/F-125 (#3)	ASPHALT/ 6/21/74	73/74/75/77			37/36/37/36			42/40/42/40			62/62/60/58	ASPHALT BADLY DEGRADED	30°	
DRI-SIL 73/SILANOX 101 (#5)	ASPHALT/ 6/21/74	68/72/72/70			87/86/86/84			ASPHALT DEGRADED			83/84/82/84	YELLOW COLOR SHOWS DEGRADATION (104°)	70°	
AROTHANE 190MS0/FREKOTE (#7A)	ASPHALT/ 6/21/74	76/75/75/73			49/52/51/49			43/43/40/40			57/58/56/56	ASPHALT RUINED (55°)	30°	
AROTHANE 190MS0/DRI-Sil (#9)	ASPHALT/ 6/21/74	75/76/75/76			59/60/60/59			36/33/33/33			65/66/65/63	ASPHALT RUINED (63°)	30°	
CARTARETIN F-4 (#11A)	ASPHALT/ 6/21/74	77/76/77/77			NOT MEASURED STILL TACKY			COATING DEGRADED			ASPHALT RUINED	ASPHALT AND COATING RUINED		
DRI-SIL 73/FREKOTE (#13)	ASPHALT/ 6/21/74	73/75/75/77			73/73/70/70			73/77/75/76			70/70/70/69	LOOKS O.K. (90°)	30°	
DRI-SIL 73/SILANOX 101 (#11B)	ASPHALT / 6/24/74	77/76/77/77						ASPHALT DEGRADED			83/83/83/82	SAME AS #5	90°	
PETROSET AT (#7B)	ASPHALT/ 7/3/74	76/75/75/73						65/65/67/67			77/77/78/78	LOOKS O.K.	60°	
CONCRETE TESTS														
PETROSET AT (#1)	CONCRETE/ 7/11/74	72/72/72	7/15	32	57/53/53/50	7/30	30	63/63/63/63	9/3	15	71/70/70/70		30°	
DRI-SIL 73 (#2)	CONCRETE/ 7/11/74				67/65/65/64			62/60/60/60			76/74/73/75		60°	
DRI-SIL 73/ DC 732 (#3)	CONCRETE/ 7/11/74				67/67/68/68			58/56/57/56			64/63/65/65		90°	
FORMULA 125 (#4)	CONCRETE/ 7/11/74				76/79/75/76			68/69/69/69			74/73/73/73	F-125 GONE BY 9/3/74	30°	
TT-P-115D/DC 732 (#5)	CONCRETE/ 7/11/74	78/78/78/77			79/78/82/78			76/77/76/77			83/83/83/85		90°	
TT-P-115D/4044 (#6)	CONCRETE/ 7/11/74				93/93/90/91			96/94/96/96			100/100/98/95		60°	
190MS0/FREKOTE 33 (#7)	CONCRETE/ 7/12/74				67/66/67/65			43/43/43/43			70/68/68/68		30°	
190MS0/4044 (#8)	CONCRETE/ 7/12/74	73/75/74/73			35/35/34/34			43/40/40/41			41/40/38/40		30°	
CARTARETIN F-4 (#9)	CONCRETE/ 7/12/74				19/17/20/20						60/61/63/62	F-4 GONE BY 9/3/74	30°	

*DEGRADATION OF ASPHALT CHARACTERIZED BY LIFTING AND PEELING OF SURFACE

Table 4-9 FRICTION (ASTM E303) AND DEGRADATION TESTS (Continued)

MATERIAL (SECTION)	SUBSTRATE/ DATE MIL. APP.	UNCOATED SUBSTRATE SKID VALUES	SKID TEST DATA									NOTES	ESTIMATED CONTACT ANGLES H ₂ O 9/3/74
ALL MATERIALS APPLIED AT A RATE EQUAL TO A 0.010 cm THICK FILM			TEST ONE			TEST TWO			TEST THREE				
			DATE	TEMP °C	VALUES	DATE	TEMP °C	VALUES	DATE	TEMP °C	VALUES		
SECOND ASPHALT TESTS													
DRI-SIL 73/DC 732 (#2)	ASPHALT / 7/16/74	73/74/74/74	7/17	39	73/77/78/79	7/30	30	66/68/68/69	9/3	15	66/65/67/66		30°
TT-P-115D/DC 732 (#4)	ASPHALT / 7/16/74	76/78/78/78			68/67/67/66			68/68/69/70			70/70/70/70		90°
TT-P-115D/4044 (#6)	ASPHALT / 7/16/74	73/74/76/75			88/88/88/88			85/85/86/85			85/84/84/85		60°
190MS0/FREKOTE 33 (#8)	ASPHALT / 7/16/74	65/63/63/62			43/44/43/43			34/33/33/34			40/38/38/39		60°
MOD. TT-P/ 4044 (#10)	ASPHALT / 7/16/74	67/71/68/73			84/86/84/84			83/85/87/87			96/98/98/96		30°
DC 92-009 (#12)	ASPHALT / 7/16/74	73/73/75/76			45/46/45/46			34/35/34/35			39/36/36/35		90°
MOD. TT-P/DC 92-009 (NE)	ASPHALT / 7/29/74							59/61/62/62			58/58/55/60		90°
MOD. TT-P/DC 732 (SE)	ASPHALT / 7/29/74							54/54/54/53			60/61/60/61		90°
MOD. TT-P/FC 321 (2-4)	ASPHALT / 7/30/74							87/89/89/88	9/18	27	81/84/88/88		60°
G31, 2x (#14)	ASPHALT / 9/19/74	78/80/80/80	9/20	26	67/69/68/68								
G31 THIN (#14)	ASPHALT / 9/19/74	78/80/80/80			79/78/79/80								
XYLAN 2052 (#14)	ASPHALT / 9/19/74	78/80/80/80			59/56/56/56								
FC-321 (#1)	ASPHALT / 10/9/74	72/75/76/75	10/9	25	63/63/60/63								
FREKOTE 33 (#2)	ASPHALT / 10/9/74	73/75/74/77			73/72/73/73								
DC 732 (#3)	ASPHALT / 10/9/74	68/72/72/70			46/44/44/44								

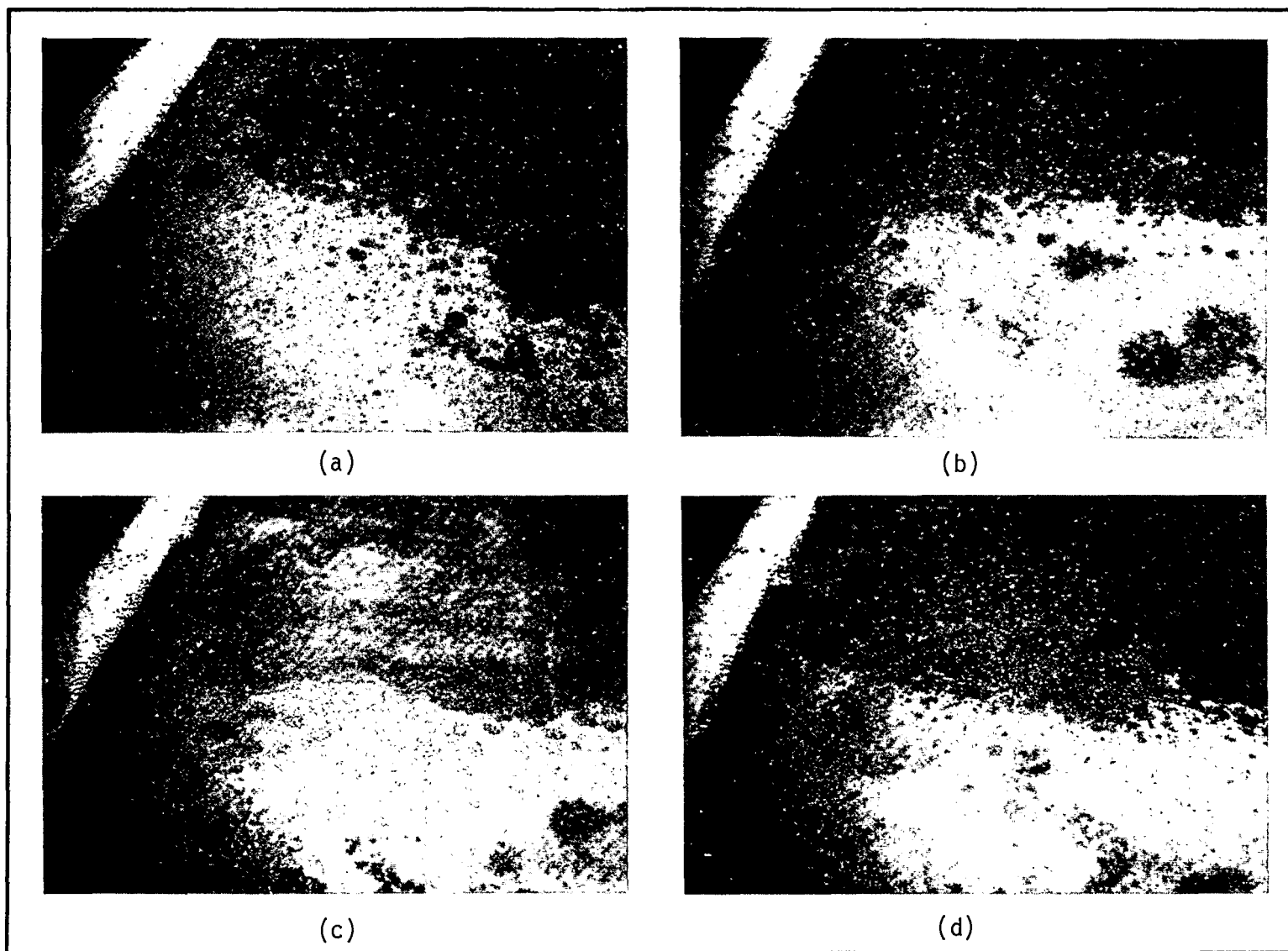


Figure 4-9 Tests of Various Materials on BBRC Asphalt Parking Lot

Figure 4-10 (first asphalt tests) showing:

A-10a: Lot #9

A-10b: Lot #11A -- Note poor wetting by the coating

A-10c: Lot #11B with degradation starting

A-10d: Lot #13

Figure A-11 showing:

A-11a: An overall view of the asphalt area of Phase II

A-11b: Severely degraded Cartaretin F-4 (the narrow strip) and more advanced degradation in adjacent 11B due to Silanox 101

A-11c: The relative appearance of STD TT-P-115D and

A-11d: modified TT-P-115D with the smooth coatings obtained

Figure 4-12 showing:

A-12a: Severely degraded asphalt due to Arolon 376

A-12b: Severely degraded asphalt due to Arothane 190M50

A-12c: Severely degraded asphalt again due to an Arothane formulation

Figure 4-13 showing various views of the concrete sidewalk coatings immediately after spraying.

6.4 Skid Value Interpretation

Figure 4-14 shows a photo of the ASTM E303 portable skid tester used in making the skid value measurements. The unit is basically a pendulum with a rubber foot that slides a given distance across the (wet) test surface and swings up an amount inversely proportional to the frictional energy dissipated.

Figure 4-15 shows two correlations for friction coefficient as a function of skid value scale reading. The BBRC derivation was from a simple balance of potential energy loss (during the tester swing) against frictional energy gain. The Colorado Department of Highways' correlation is from their

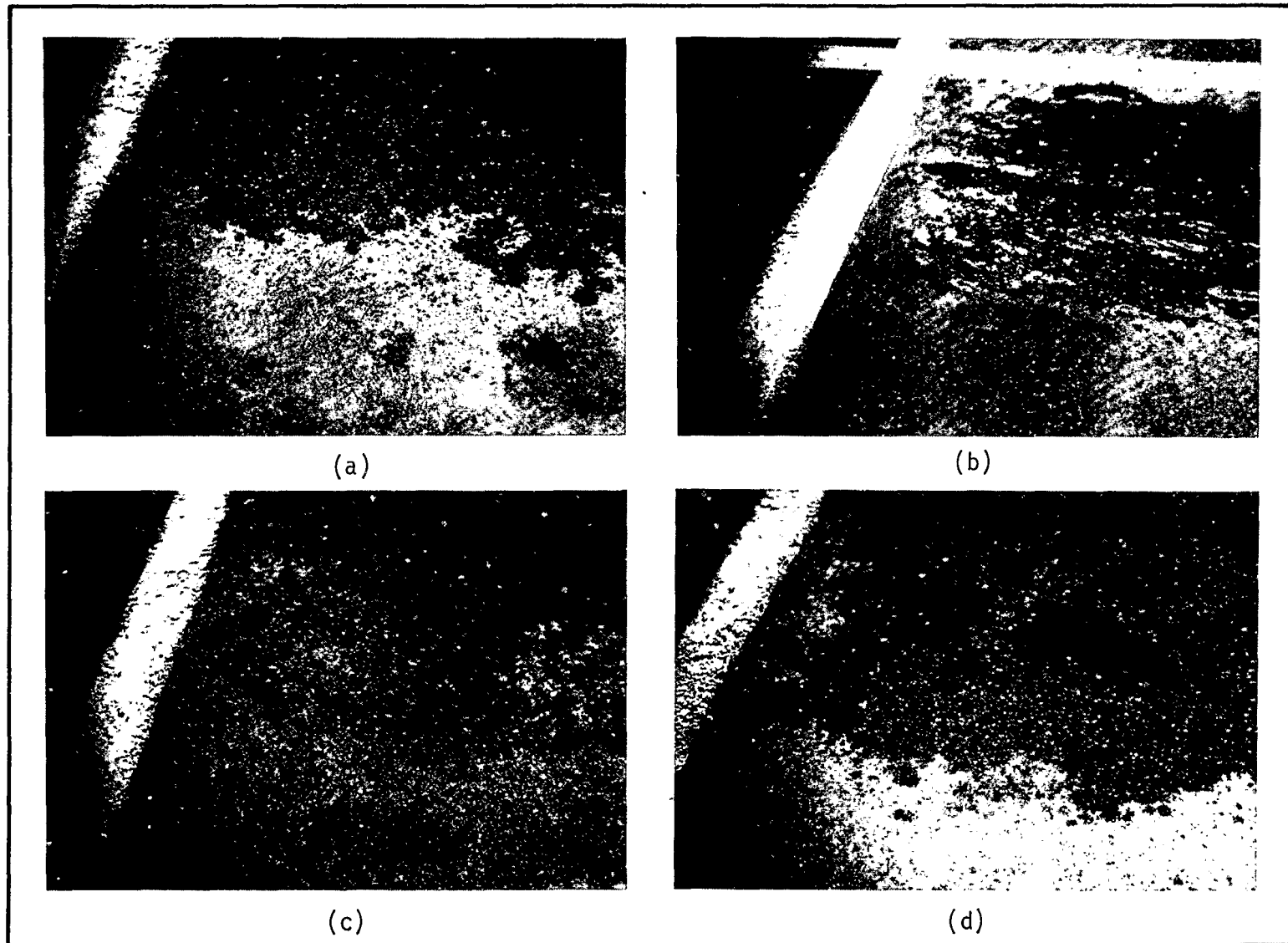


Figure 4-10 Tests of Various Materials on BBRC Asphalt Parking Lot

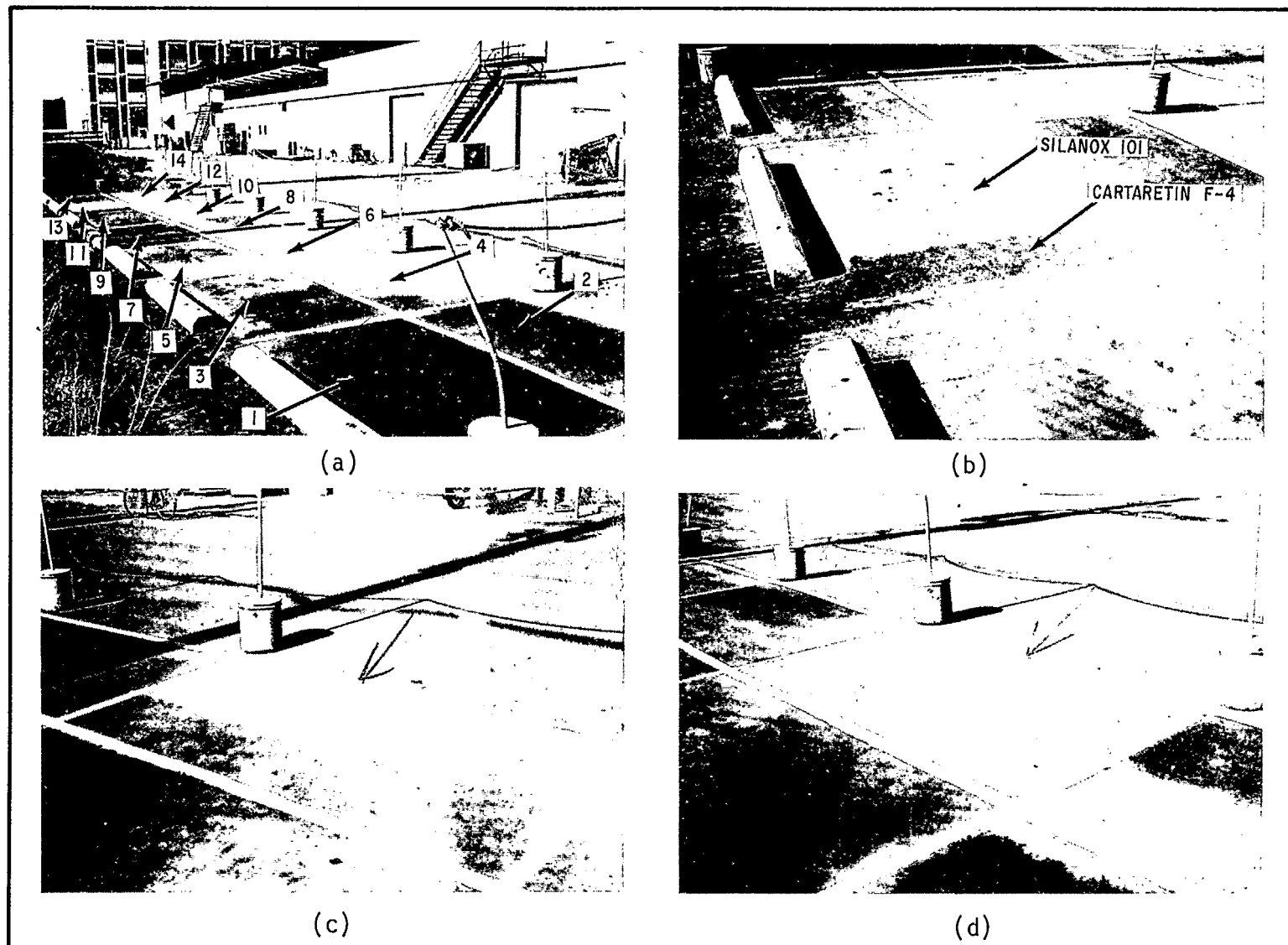
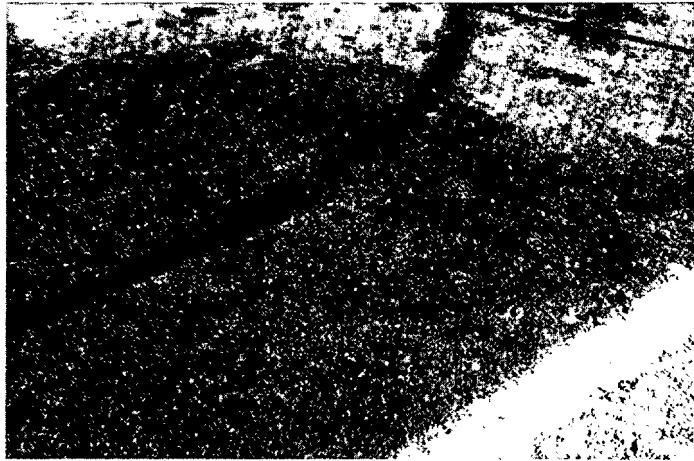
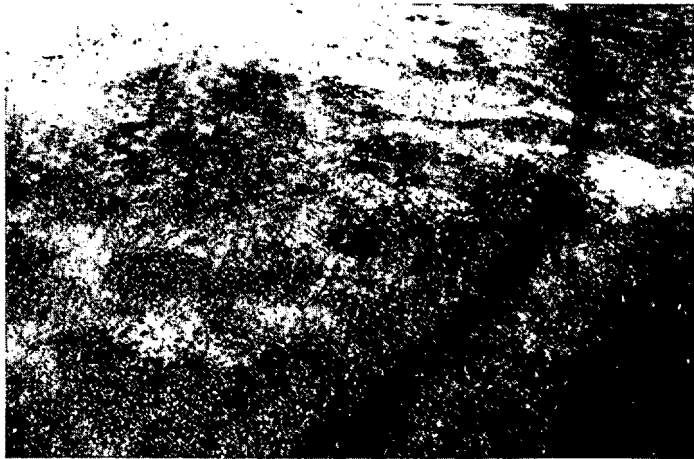


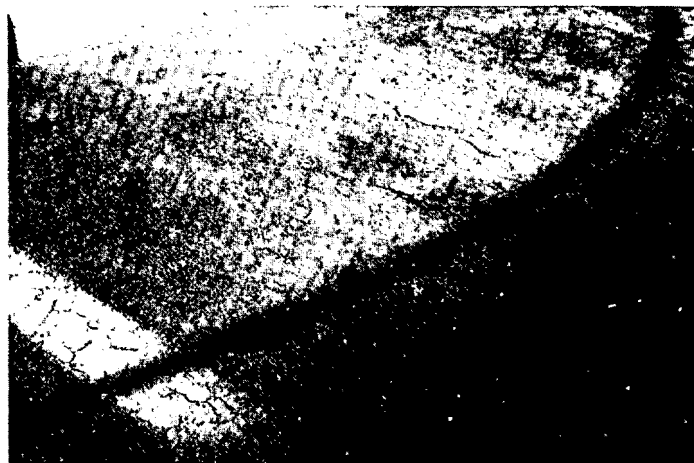
Figure 4-11 Tests of Various Materials on BBRC Asphalt Parking Lot



(a)



(b)

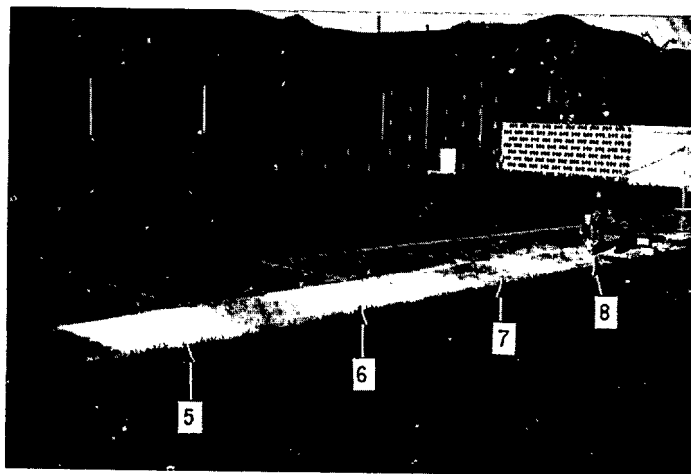


(c)

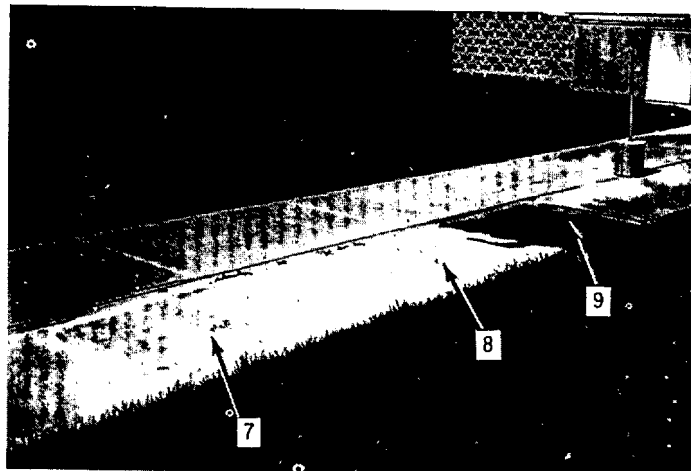
Figure 4-12 Severely Degraded Test Sections on BBRC Asphalt Parking Lot



(a)



(b)



(c)

Figure 4-13 Tests of Various Materials on BBRC Concrete Sidewalk



Figure 4-14 ASTM E303-69 Portable Skid Tester

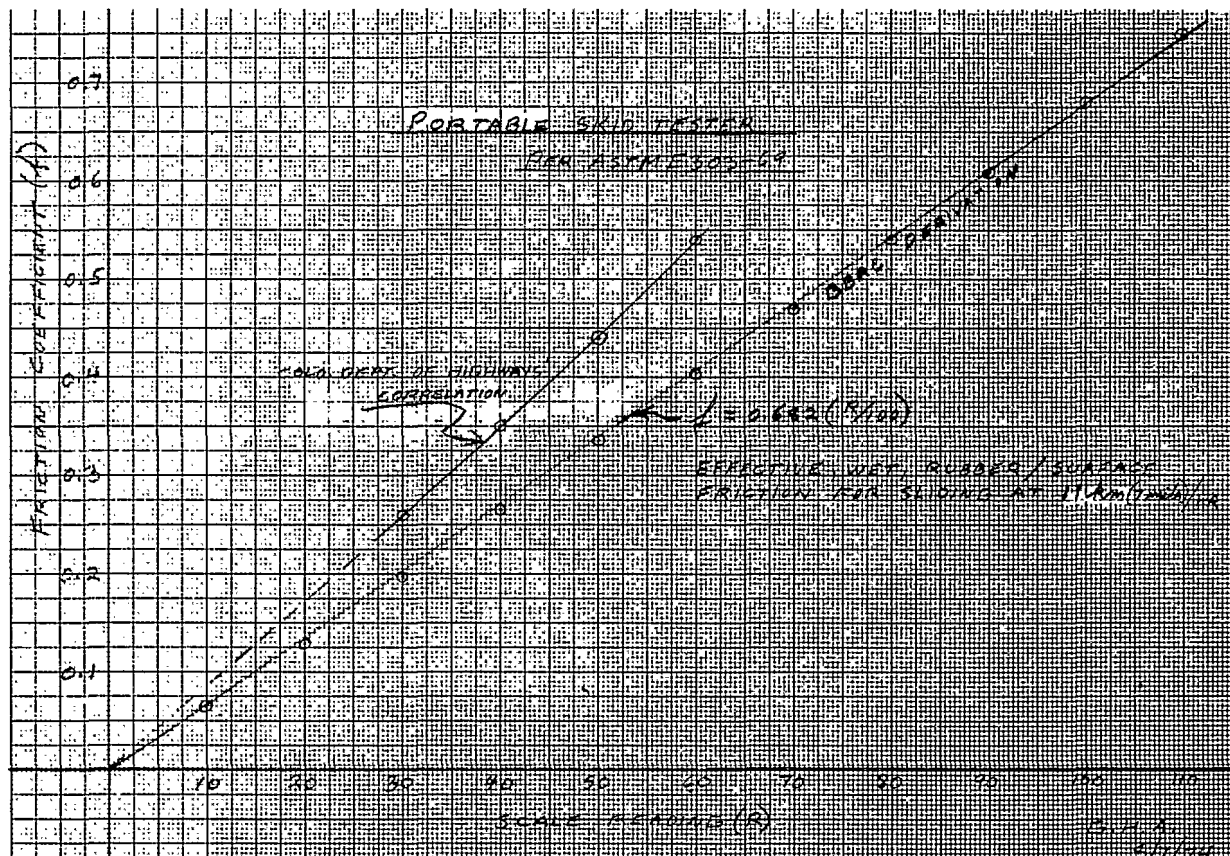


Figure 4-15 Correlation of Friction Coefficient (f) with Skid Value (R) from Portable Skid Tester

data files. The cited speed is that at which the tester foot first contacts the surface. Due to the uncertainty of the correct correlation, tabular data has been left as "skid values". If it is desired to convert the data to friction coefficients, it is suggested that the BBRC correlation be used. The Colorado Highways' correlation is based on a comparison with grooved tire data whereas the skid tester foot is not grooved.

The other reason for leaving the skid values as such is shown in Table 4-10. The skid values are, through use of the table, directly related to highway safety -- a matter of vital interest to any state Highway Department.

Finally, it should be noted that even the poorest coatings have a friction coefficient of about 0.25 compared to 0.06 to 0.12 for ice (Figure 1 of Reference 54 in Appendix A).

6.5 Conclusion

The results of the friction and degradation tests showed that, in general

- Water soluble materials are unacceptably slippery.
- Very highly hydrophobic materials, in their pure form, also provide too little traction.
- Highly oleophilic materials and surface-active agents badly degrade asphalt.

The skid-number tests and associated degradation observations proved to be one of the most effective screening techniques employed in this program.

7.0 ULTRAVIOLET STABILITY TESTS

The ultraviolet stability test, which was conducted primarily because of indications that hydrophobic silicones are especially sensitive to this degradation mechanism (Reference 7), is described below.

7.1 Theory

Per Reference 7, the Si-O bond is especially vulnerable to rupture from ultraviolet radiation. Since silicones have this bond and are frequently useful hydrophobes, this type of evaluation was felt to be necessary. Furthermore, although it is not pointed out specifically in Reference 7, C-O bonds

Table 4-10
SUGGESTED VALUES OF 'SKID RESISTANCE' FOR USE
WITH THE PORTABLE TESTER

CATEGORY	TYPE OF SITE	'SKID-RESISTANCE' ON WET SURFACE	STANDARD OF SKIDDING RESISTANCE REPRESENTED
A	MOST DIFFICULT SITES SUCH AS: (i) ROUNDABOUTS (ii) BENDS WITH RADIUS LESS THAN 500 ft. ON DERESTRICTED ROADS (iii) GRADIENTS, 1 in 20 OR STEEPER, OF LENGTH GREATER THAN 100 yd (iv) APPROACH TO TRAFFIC LIGHTS ON DERESTRICTED ROADS	ABOVE 65	GOOD': FULFILLING THE REQUIREMENTS EVEN OF FAST TRAFFIC, AND MAKING IT MOST UNLIKELY THAT THE ROAD WILL BE THE SCENE OF REPEATED SKIDDING ACCIDENTS
B*	GENERAL REQUIREMENTS, i.e. ROADS AND CONDITIONS NOT COVERED BY CATEGORIES A AND C	ABOVE 55	GENERALLY SATISFACTORY : MEETING ALL BUT THE MOST DIFFICULT CONDITONS ENCOUNTERED ON THE ROADS
C*	EASY SITES, e.g. STRAIGHT ROADS, WITH EASY GRADIENTS AND CURVES, AND WITHOUT JUNCTIONS, AND FREE FROM ANY FEATURES, SUCH AS MIXED TRAFFIC, ESPECIALLY LIABLE TO CREATE CONDITIONS OF EMERGENCY	ABOVE 45	SATISFACTORY ONLY IN FAVORABLE CIRCUMSTANCES'
D	ALL SITES	BELOW 45	POTENTIALLY SLIPPERY'

* ON SMOOTH-LOOKING OR FINE-TEXTURED ROADS IN THESE CATEGORIES, VEHICLES HAVING SMOOTH TYRES MAY NOT FIND THE 'SKID-RESISTANCE' ADEQUATE. FOR SUCH ROADS ACCIDENT STUDIES SHOULD ALSO BE MADE TO ENSURE THAT THERE ARE NO INDICATIONS OF DIFFICULTIES DUE TO SKIDDING UNDER WET CONDITIONS.

CHART FROM 'ROAD NOTE NO. 27 OF THE (BRITISH) ROAD RESEARCH LABORATORY

(as in ethers and alcohols), with a bond energy of 75 Kcal/mole, should be even more vulnerable to rupture than the Si-O bond.* For a 75 Kcal/mole bond, energy radiation at $\approx 3800 \text{ \AA}$ should be most disruptive and this is near the intensity peak of solar ultraviolet radiation (see Table 4-11).

Assuming a solar constant of $140 \text{ milliwatts/cm}^2$, that an average of 13 percent of this reaches ground level and that 5 percent of this is in the ultraviolet wavelength region ($2900 \text{ \AA} - 4000 \text{ \AA}$); normal exposure in real time would be $0.91 \text{ milliwatts/cm}^2$. To test for an equivalent of 150 days exposure, an energy input of about $9.1 \text{ milliwatts/cm}^2$ for 15 days is required. This translates to $1.2 \text{ by } 10^4 \text{ J/cm}^2$ total dosage. It must be emphasized that this is for 150 24-hour days and for a normal winter season represents an *overtest* by a *factor* of about *four*.**

7.2 Procedure

Films of the sample coatings were applied (by pouring for this test) to about one-half of one side of glass microscope slides. A preliminary 39-hour water soak was performed and contact angles were obtained to aid in the test decision concerning the materials to be irradiated.

The samples were mounted on a temperature-controlled platen and irradiated for 16 days to a total dose of $1.3 \text{ by } 10^4 \text{ joules/cm}^2$. Due to sample placement, the variation over the sample area was less than ± 10 percent. Periodic measurements of water contact angle and irradiation intensity were made during the exposure period.

At the end of the test, additional water soaking, qualitative ice release on some samples and sample coating conditions were used to evaluate ultraviolet degradation on a go, no-go basis.

*Note that it is not the magnitude of the bond strength that is important in this case. It is the resonant frequency which controls ultraviolet degradation sensitivity, as is completely discussed in Reference 7.

**With respect to dose rate this is an even greater overtest when compared to ASTM D795/1148 which is discussed in 5.4 of Chapter 6.

Table 4-11
ULTRAVIOLET DEGRADATION SCREENING

MATERIAL CONSIDERED	BEFORE TEST WATER CONTACT ANGLES (DEGREES)		TEST DECISION	WATER CONTACT ANGLES AFTER EXPOSURE (DEGREES)			SAMPLE CONDITION AFTER 16 HOUR WATER SOAK	QUALITATIVE ICE ADHESION STRENGTH	CONCLUSION																
	7/1/74	7/3/74 AFTER 39 HRS. AT 100% R.H.		2 DAYS EXPOSURE	4 DAYS EXPOSURE	16 DAYS EXPOSURE																			
	START IRRADIATION - 7/11/74																								
DRI-SIL 73	*90/96,89	*91,91,90	NO																						
DC 92-009	92,92,96	94,92,97	YES	92,94,94	100,99,102	95,98,94	NON-WETTED, TOUGH		NO DEGRADATION																
SS-4044	88,9189	91,90,92	YES	88,88,88	90,96,94	89,86,88	NON-WETTED, TOUGH		NO DEGRADATION																
FREKOTE 33	100,101,105	99,93,99	YES	92,89,87	92,91,92	102,98,100	NON-WETTED, TOUGH		NO DEGRADATION																
RTV 11(CATALYZED)	90,88,93	97,102,101	NO(POT LIFE TOO SHORT)																						
FORMULA 125	13		NO																						
DRI-SIL 73/SILANOX 101	*112,115,115	*116,112,105	YES	106,110,110	104,110,110	111,115,114	WETTED, TOUGH	COATING REMOVED	NOT USEFUL OR DEGRADED																
TT-P-115D, TY II	83,85,87	69,63,75	YES	66,70,70	68,68,60	61,65,60	WETTED, VERY TOUGH	HARD RELEASE	NOT USEFUL, BUT NO DEGRADATION																
AROTHANE 190M50	*93,93,91	*86,89,89	YES	91,90,90	81,87,89	84,82,82	NON-WET, VERY TOUGH	EASY RELEASE	NO DEGRADATION																
FC-321	115,109,106	101,104,102	YES	103,103,103	105,108,108	114,112,111	WETTED, VERY SOFT	COATING REMOVED	DEGRADED																
FC-210	*41,37,26		NO																						
DC 732	93,86,92	89,83,83	YES	96,96,96	90,89,89	102,102,100	NON-WETTED, TOUGH	EASY RELEASE	NO DEGRADATION																
(WATER WETTED SAMPLE = *)																									
<div><div>SAMPLE TEMPERATURE: 29C ± 1C IRRADIATION INTENSITY: 7/11/74: 9.3 mW/cm² 7/15/74: 9.4 mW/cm² 7/27/74: 9.4 mW/cm² TOTAL DOSE: 1.3 x 10⁴ J/cm²</div><div><table><tr><th colspan="2">RELATIVE INTENSITY</th></tr><tr><th>WAVE LENGTH-Å</th><th>INTENSITY</th></tr><tr><td>3100</td><td>0.08</td></tr><tr><td>3300</td><td>0.32</td></tr><tr><td>3500</td><td>0.66</td></tr><tr><td>3700</td><td>1.00</td></tr><tr><td>3900</td><td>0.72</td></tr><tr><td>4100</td><td>0.30</td></tr></table></div></div>										RELATIVE INTENSITY		WAVE LENGTH-Å	INTENSITY	3100	0.08	3300	0.32	3500	0.66	3700	1.00	3900	0.72	4100	0.30
RELATIVE INTENSITY																									
WAVE LENGTH-Å	INTENSITY																								
3100	0.08																								
3300	0.32																								
3500	0.66																								
3700	1.00																								
3900	0.72																								
4100	0.30																								

7.3 Numerical Data

All data and test conditions are summarized in Table 4-11. The judgment factors are self-explanatory. The Dri-Sil 73 is presumed to be satisfactory based on vendor exposure data and the toughness of the Dri-Sil 73/Silanox 101 combination film despite the presence of the Silanox 101, which is known to be sensitive to outdoor exposure from the data in Section 6 above.

The virtual destruction of the FC-321 was somewhat surprising but may be the reason for 3M's opinion of non-suitability for this material in the subject application (see Chapter 3).

7.4 Photos

Figure 4-16 shows:

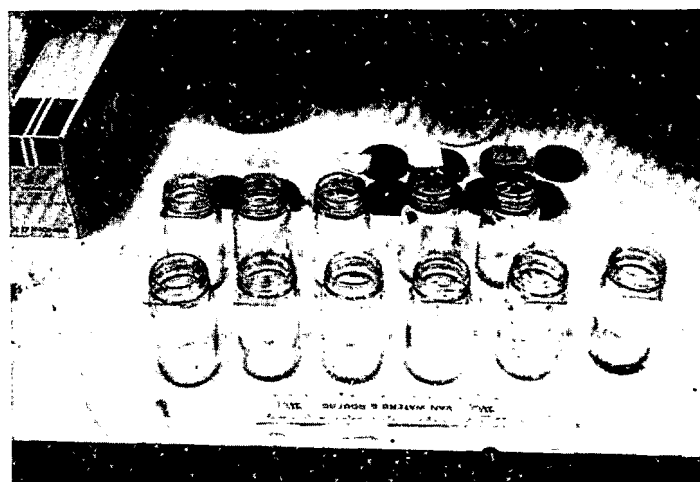
- 4-16a: The samples applied to the glass plates
- 4-16b: The general test setup showing the enclosed irradiation cabinet with blower tubes to remove ozone, the temperature controller for the sample platen and the ultraviolet source power supply
- 4-16c: The irradiation cabinet open and the platen in a horizontal position during contact angle measurement

7.5 Conclusion

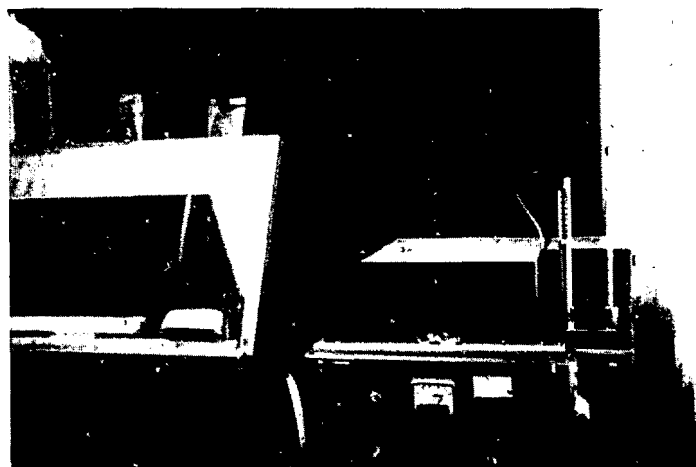
Materials, such as DC92-009 and DC 732, which were suspected of being sensitive to ultraviolet-radiation degradation, were in fact found to be stable while one material (FC-321) from a class usually considered quite stable (i.e., fluorinated compounds) was in fact found to be extremely sensitive. This tends to confirm the frequency, rather than absolute energy, dependency mentioned in Reference 7. Note that additional ultraviolet exposure data for the three road-tested coatings are presented in Chapter 6.

8.0 SOLUBILITY DATA

Basic solubility data are presented in Sections 1 and 2 above. A further discussion of coating solubility and solvents required for application is given in the Environmental Impact section of Chapter 5.



(a)



(b)



(c)

Figure 4-16 Ultraviolet Stability Test Samples and Apparatus

In any event the basic criteria in this area are:

- Negligible or very low water solubility for the applied coatings as demonstrated by the contact angle test series (Section 2 above), weather endurance (Section 6 above and Chapter 6) and quantitative solubility from environmental-hazard-related tests (Section 9 below and Chapter 6).
- Negligible solubility of oil in the coating to avoid degradation of asphalt surfaces as detected during the tests described above in Section 6.

9.0 ENVIRONMENTAL IMPACT TESTS

Environmental impact tests on components showing the most promise from the above tests are reported below. As discussed in the Recommendations and Summary, Chapter 1, hydrophobic coatings -- no matter how efficient -- cannot be justified unless their environmental impact compared to conventional deicing chemicals is substantially less.

9.1 Procedure

The test coatings were sprayed on one side of 7.6 cm by 15.2 cm stainless steel plates. The plates were cured overnight at ambient temperature and water contact angles were measured.

The plates were soaked for 48 hours in one liter of distilled water per plate. Note that this is equivalent to the concentration of the coatings' water-soluble material in only an 8.6 cm (3.4-inch) deep rain, which is much less precipitation than would be expected over a winter season. The water samples were then analyzed per USEPA methods. The coatings were re-examined for contact angle and physical characteristics.

9.2 Numerical Data and Observations

The data are summarized in Table 4-12. Most of the items are self-explanatory and indicate quite low pollution potential for most of the coatings. Again, the FC-321, which looked rather promising in the ice adhesion tests, softened (degraded) during the extended water-soaking period. The Frekote 33 was the biggest surprise since other tests had indicated good water repellency.

Table 4-12
ENVIRONMENTAL SCREENING TEST RESULTS

MATERIAL {SAMPLE NUMBER}	FILM AREA (cm ²) /WEIGHT (gm)		WATER CONTACT ANGLE-DEGREE	WATER ANGLE-DEGREE	FILM	pH	SOLIDS (gms)	BOD mg/LITER	COD mg/LITER	WEIGHT % OF FILM DISSOLVED
	AFTER 48 HOUR SOAK									
			(7/29/74)	(8/23/74)		ENVIRONMENTAL DATA				
							(HAUSER REPORT 74-314) (REF. 60)			
BLANK	116	- - -				6.37	-0.0006			
190M50 (E1)	116	0.76	86,89,86	82,82,84	VERY TOUGH EXC. ADHESION	6.10	+0.0063	3	11	0.8
FREKOTE 33 (E2)	116	0.07	98,99,101	99,103,102	TOUGH GOOD ADHESION	5.95	+0.0040	11	42	5.7
TT-P-115D,TY. II (E3)	116	1.67	98,95,75	89,89,89	VERY HARD GOOD ADHESION	6.60	+0.0137	4	15	0.8
MODIFIED TT-P-115D TY, II (E4)	116	1.73	94,96,98	96,99,96	HARD FAIR ADHESION	6.40	+0.0103	8	32	0.6
FC -321 (E5)	116	1.01	107,107,112	101,105,105	SOFT,GOOD ADHESION	6.73	+0.0023	9	34	0.2
4044 (E6)	116	0.30	92,90,91	87,83,83	TOUGH,GOOD ADHESION	6.10	+0.0020	4	16	0.7
DC 732 (E8)	116	0.05	107,108,109	106,103,103	STILL TACKY	5.90	+0.0023	3	16	4.6
92-009 (E7)	116	0.41	96,95,96	99,103,102	TOUGH,POOR COHESION	5.90	+0.0007	2	5	0.2
TT-P-115D/DC 732 (E9)	116	2.06	102,106,109	101,106,114	TOUGH, GOOD ADHESION	6.98	+0.0140	5	15	0.7
190M50 / FREKOTE (E10)	116	0.52	100,98,98	96,85,95	TOUGH,HARD EXC. ADHESION	6.90	+0.0013	14	55	0.3
PETROSET AT (E11)	116	1.42			STILL TACKY EXC. ADHESION	6.10	+0.0183	22	84	1.3
*75th STREET SEWAGE TREATMENT PLANT, 1/2/74	{INFLUENT}							170	378	
	{EFFLUENT}							10	49	
*WATER TREATMENT PLANT, 1/2/74	{INFLUENT}							0.5	1.0	
	{EFFLUENT}							0.4	0.9	

*REFERENCE 60 SUPPLEMENT

9.3 Conclusion

Most of the coatings exhibit quite low environmental contamination potential. This is borne out by the numerical data, the comparative numbers cited at the bottom of the table and the discussion of the test data (Reference 60) included in Appendix A.

As regards total runoff water solids content, consider a one-meter highway lane (3.66 m wide) coated with DC 732. Per meter of length, there is 366 cm³ of material (for the 0.01 cm thickness used). For the total life of the coating, if it were *all* DC 732, the runoff water could contain no more than about 18 grams of dissolved material per meter of length. The application rate for salt cited in the problem statement of the Recommendations and Summary, Chapter 1, gives 560 grams of salt per meter of length, or more than 25 times the amount of material possible from a DC 732 coating.* Noting that most of the coating materials give 20 percent or less solids dissolved than DC 732 (per the last column of Table 4-12), it is concluded that most of the coatings have truly negligible contamination potential once applied to the roadway.

*Note that weights are being compared, not necessarily environmental impact.

Chapter 5

PHASE III APPLICATION STUDY

1.0 INTRODUCTION

In this chapter are presented the various considerations employed in the selection, application and environmental evaluation of the coatings to be used in the Phase IV highway tests. Specific topics are material rating factors, the exact composition of the solutions as applied, a summary of material data required to compute application rates, a complete cost summary comparison including updated factors for salt, the formulae used to determine spray rates and application vehicle speed and data on spray techniques and calibration methods. Finally, a complete environmental impact discussion including wear debris, coating solubility considerations and hazards during application is presented.

2.0 RATING FACTORS AND FORMULATION SELECTION

Not all materials investigated were explicitly rated. Certain materials and/or combinations were deleted for the following (typical) reasons:

- Cationic Surface Active Agents. These materials showed no tendency to be hydrophobic. Although they are attractive from a cost standpoint and may exhibit a "residual" release effect, extensive tests with one such material (Cartaretin F-4) indicated very rapid degradation of asphalt and very high BOD/COD demand due to high water solubility and its chemical nature (i.e., amines and amides create high demand levels).
- Cost. While cost alone was not used as a factor in arbitrary deletion, high cost combined with less than outstanding wear resistance (Nyebar F) or likely high material and application costs combined with very limited availability (the NRL sample) caused elimination.
- Toxicity. Toxicity "as received" was not heavily weighted since limited use and/or solvent replacement

could reduce the hazard. However, one binder (TT-P-85D) contains sufficient lead in the dried film (per shipping label) to present a health-hazard increase over the existing high levels found in road dust (Reference 69) if used as a road coating rather than the current 7 cm (3-inch) wide stripes.

2.1 The Ratings

As noted in Table 5-1, the ratings are divided into four groups. These groups are so diverse that it was felt a single composite rating factor would be more confusing than useful. The ratings are so devised that higher numbers or "+" signs are favorable. The detailed rating system is defined below.

- Ice Adhesion

> 9 kg/cm² = 0

6-9 kg/cm² = 1

3-6 kg/cm² = 2

< 3 kg/cm² = 3

Removal of the coating or an upward trend per successive release is indicated by "-".

- Road Friction

< 45 skid value = 0

45-55 skid value = 1

55-65 skid value = 2

> 65 skid value = 3

- Water Contact Angle

< 65° = 0

65°- 80° = 1

,80°-100° = 2

>100° = 3

Table 5-1
MATERIAL/FORMULATION RATING(1) SHEET

MATERIAL AND/OR FORMULATION	FUNCTIONAL FACTORS		JUDGMENT FACTORS		USEFUL LIFE FACTORS			APPLICATION COST FACTORS			ROAD TEST CONCLUSION	EXACT FORMULA COST ¢/m ²
	ICE ADHESION	ROAD FRICTION	H ₂ O CONTACT ANGLE	BOD/COD LEVEL	MATERIAL OR SUBSTRATE DEGRADATION	UV STABILITY	WEAR ⁽²⁾ AND / OR HARDNESS	BASE OF APPLICATION	CURRENT AVAILABILITY	MATERIAL COST		
FC-321	2	2	3	1	+	-	1(H)	3	2	0	OUT. COST, WEAR, STABILITY	590
FREKOTE 33	2	3	3	1	+	+	2(H)	3	1	0	OUT. COST, AVAIL.	
G31, 2X G31, THIN	3 2	3 3	2 2	2 1 (1)	+(SHORT + (TERM)	+	1(H) 1(H)	0 0	1 1	0 2	PRICE IN DETAIL	336 96
DC 92-009	3	0	3	2	+	+	1(H)	2	2	0	OUT. FRICTION, WEAR	
TT-P-115D	1	2	2	2	+	+	3(W)	1	2	3	PRICE IN DETAIL	20
TT-P-115D/DC 732	1(-)	3	3	2	+	+	2(H)	1	2	2	OUT. ADHESION, COST	
TT-P-115 D/SS-4044	1	2-3	3	2	+	+	2(W)	1	2	1	OUT. ADHESION, COST	
TT-P-115D/ DC 92-009	2	2	3	2	+	+	2(W)	1	2	2	PRICE IN DETAIL	66
TT-P-115 D/ FC-321	2(-)	2(1)	3	1	+	-	3(H)	1	2	1	OUT. ADHESION, STABILITY	
TT-P-115 D/FREKOTE 33	1		3	1	+	+	2(H)	1	1	0	OUT. ADHESION, COST	
MOD TT-P-115 D/ FC-321		3	2(1)	1	+	-	2(W)	1	1	1	OUT. STABILITY, COST, INFERRED AD.	
MOD TT-P-115 D	0(-)		1	1	+	+	3(H)	1	1	2	OUT. ADHESION	
① MOD. TT -P-115 D/DC 732	3	2-3	3	1	+	+	3(W)	1	1	2	PRICE IN DETAIL	37
MOD TT-P-115 D/DC 92-009	3(1)	2	3	1	+	+	2(W)	1	1	2	PRICE IN DETAIL	55
MOD TT-P-115D/SS-4044	0(-)	2-3	3	1	+	+	3(W)	1	1	1	OUT. ADHESION	
DC 732	3	0	3	2	+	+	1(H)	2	2	1	OUT. WEAR, COST	
AROTHANE 190MS0	2(-)		2	2	-A	+	2(H)	2	2	3	OUT. DEGRADATION, ADHESION	
AROTHANE 190MS0/FREKOTE	2(-)	0-3	1	0	-A	+	2(W)	2	1	3	OUT. FRICTION DEGRADATION	
AROTHANE 190MS0/SS-4044	2	0	2	2	-A	+	2(W)	2	2	2	OUT. FRICTION, DEGRADATION	
AROTHANE 190MS0/DRI-SIL 73	1(1)	0-2	2	1(1)	-A	+	1(H)	2	2	2	OUT. FRICTION, DEGRADATION	
DRI-SIL 73	1(-)	3	2	1	+	?	3(H)	3	2	2	OUT. ADHESION	
DRI-SIL/SILANOX 101	0(-)	3	3	1	-A,M	?	2(H)	1	2	2	OUT. ADHESION, DEGRADATION	
DRI-SIL73/FREKOTE 33	1(1)	3	3(1)	1	+	?	2(H)	2	1	2	PRICE IN DETAIL	87
② DRI-SIL 73/DC 732	2	2-3	3	1	+	?	1(H)	2	2	2	PRICE IN DETAIL	68
DRI-SIL 73/DC 92-009	1		3	1	+	?	1(H)	2	2	2	OUT. ADHESION, WEAR	
③ PETROSET AT	0	2-3	0	0	+	?	3(W)	3	2	3	PRICE IN DETAIL, TEST	7
XYLAN 2052	2(1)	2	0-1	0(1)	+(SHORT TERM)	+(1)	1(H)	1	2	0	OUT. WEAR, COST	

- (1) = INFERRED FROM OTHER DATA
- W = ESTIMATE BASED ON WEAR DATA, H = ESTIMATE BASED ON QUALITATIVE SCRATCH TEST
- ICE ADHESION AND ROAD FRICTION WERE CONSIDERED FIRST IN THE SELECTION OF MATERIALS A, B AND C BUT SEE ALSO SECTION 2.2 OF THIS CHAPTER.

In general, the angles given are those that were measured following the ice adhesion tests or other screening tests.

- BOD/COD Level

Rated on the basis of reported COD levels for the materials or major components as follows:

> 50 mg/liter = 0

20-50 mg/liter = 1

< 20 mg/liter = 2

- Material or Substrate Degradation

"+" = No degradation

"-" = Degradation

M = Material

A = Asphalt substrate

- UV Stability

"+" = Stable

"-" = Degraded

- Wear and/or Hardness

Quantitative rating based on the road tests and on scratch resistance (usually after exposure to 100 percent relative humidity for 24 hours).

1 = Very soft or high wear

2 = Moderate

3 = Low wear or very hard (*not* the same as tough)

- Ease of Application

Based on a scale of 0 (most difficult) to 3 (easy) by positive considerations of (a) standard spray

truck application, (b) low settle-out rate, (c) long pot life and (d) ease of equipment clean-up.

- Availability

A rating of the availability *and* delivery time currently estimated with:

0 = Not currently available

1 = Available but not a stock item

2 = Delivery available from stock

- Material Cost

A rating of the cost of the material (\$) to cover a one square-meter area with solids equivalent to a thickness of 0.010 cm.

$> 300 \text{ } \$/\text{m}^2 = 0$

$100\text{-}300 \text{ } \$/\text{m}^2 = 1$

$30\text{-}100 \text{ } \$/\text{m}^2 = 2$

$< 30 \text{ } \$/\text{m}^2 = 3$

2.2 Discussion

With one exception (Scotchgard FC-321), ten coatings appeared promising enough to price in detail (pricing factors are given in Sections 3 and 4 below). These costs are given at the right edge of Table 5-1. At the left edge are given the letter designations for the three formulations selected for highway evaluation tests. Formulation B appears to violate most of the basic ground rules. However, per the ~~contact~~ report included in Appendix B, it is one of only two materials studied which was reported to be directly applicable for this study. It should also be mentioned that, had the program ground rules permitted consideration of (and funds for) incorporation of materials into resurfacing mixtures, other materials such as Viscospin B would certainly have been rated.

3.0 COMPOSITION SUMMARY

The exact compositions of the coatings rated in Table 5-1 are given in Table 5-2. These compositions were determined on a

Table 5-2
COMPOSITIONS OF RATED COATINGS

DESIGNATION	MAJOR MTL./AMOUNT	2ND. MTL./AMT.	OTHER COMPONENTS/AMOUNTS			FORMULATION OF TTP-115D	
						COMPONENT	AMOUNT POUNDS/100 GALS.
FC-321	FC-321/50 cm ³	VMP/25cm ³				SHELL TOLU-SOL 19EC	340
FREKOTE 33	FREKOTE 33 / 100%					METHANOL/WATER (95/5)	2
G 31, 2X G 31, THIN	SS-4044 / 33 gm DRI-SIL 73 / 49 gm	DRI-SIL 73 / 8 gm RTV-11/7 gm	RTV 11/3 gm CAT./1 gm	CATALYST/.04 gm		CLORAFIN 40	35
DC 92-009	DC 92-009 / 50 cm ³	VMP / 50 cm ³				VELSICOL XL-37	35
TT-P-115D	TT-P-115D / 100 cm ³	VMP / 50 cm ³				BENTONE-38	5
TT-P-115D/DC 732	TT-P-115D / 80 cm ³	VMP / 40 cm ³	DC 732/13.7 gm	VMP/31 cm ³	ISOPROP/3cm ³	SOYA LECITHIN	8
TT-P-115D/SS-4044	TT-P-115D / 80 cm ³	VMP / 40 cm ³	4044/11 cm ³			PLIOLITE VTL	107
TT-P-115D/DC 92-009	TT-P-115D / 80 cm ³	92-009 / 20 cm ³	VMP/55cm ³			TITANOX 2061	150
TT-P-115D/FC-321	TT-P-115D / 80 cm ³	FC-321 / 20 cm ³	VMP/40 cm ³			DURAMITE	210
TT-P- 115D/FREKOTE 33	TT-P-115D / 80 cm ³	FREKOTE / 15 cm ³	VMP/40 cm ³			ASBESTINE X	53
MOD. TT-P-115D/FC-321	MIT-P-115D/ 80 cm ³	FC-321 / 20 cm ³	VMP/40 cm ³			CELITE 110	83
MOD. TT-P-115D	MIT-P-115D / 80 cm ³	VMP / 40 cm ³				MINERALITE 3X	59
(A) MOD. TT-P-115D/DC 732	MIT-P-115D / 80 cm ³	DC 732 / 14 gm	VMP/71 cm ³	ISOPROP/3 cm ³			
MOD. TT-P-115 D/92-009	MIT-P-115D / 80 cm ³	92-009 / 15 cm ³	VMP/ 50 cm ³				
MOD. TT-P-115 D/ SS-4044	MIT-P-115D / 80 cm ³	VMP / 40 cm ³	4044/10 cm ³				
DC 732	DC 732 / 14 gm	VMP / 31 cm ³	ISOPROP/3 cm ³				
AROTHANE 190M50	190M50 / 85 cm ³	VMP / 51 cm ³					
AROTHANE 190M50/FREKOTE 33	190M50 / 85 cm ³	FREKOTE / 22 cm ³	VMP/49 cm ³				
AROTHANE 190M50/ SS-4044	190M50 / 85 cm ³	4044 / 14 cm ³	VMP/49 cm ³				
AROTHANE 190M50/DRI-SIL 73	190M50 / 85 cm ³	DRI-SIL / 57 cm ³	VMP/114 cm ³				
DRI-SIL 73	DRI-SIL 73 / 92 cm ³	VMP / 46 cm ³					
DRI-SIL 73/SILANOX 101	DRI-SIL 73 / 100 cm ³	S-101 / 6 gm					
DRI-SIL 73/FREKOTE 33	DRI-SIL 73 / 100 cm ³	FREKOTE / 57 cm ³	VMP/50 cm ³				
(C) DRI-SIL 73/DC 732	DRI-SIL 73 / 100 cm ³	DC 732 / 13 gm	VMP/80 cm ³	ISOPROP/3 cm ³			
DRI-SIL 73/DC 92-009	DRI-SIL 73 / 100 cm ³	92-009 / 100 cm ³	VMP/100 cm ³				
(B) PETROSET AT	PETROSET / 100 cm ³	WATER / 200 cm ³					
XYLAN 2052	XYLAN 2052 / 100%						

somewhat trial-and-error basis according to what was required for efficient spraying. The composition of Fed. Spec. paint TT-P-115D, Type II, is given at the right-hand side. The modified version (MOD. TT-P-115D) omitted the Titanox. Note that during this program, it was discovered that isopropanol is capable of stabilizing DC 732 in solution for extended periods of time (six months or more). Finally, it must be emphasized -- as pointed out in the Recommendations section -- that the compositions are not necessarily optimized with regard to either major/minor component ratio or solvent type.

4.0 COST/COVERAGE SUMMARY

The data required to compute the required coverage rates (for a 0.010 cm film of non-volatiles) and material costs are presented in Table 5-3 for the more seriously considered materials. The formulae used in computing coverage rate (A) and unit material cost ($\$/m^2$) are noted on the table. It would appear that the Arothane (which degrades asphaltic surfaces) might merit special study as a concrete sealant in view of its low cost.

As a sample computation for mixtures, consider formulation A -- the MOD. TT-P-115D/DC 732 mixture. From the formulation table, Table 5-2, we have:

- MOD. TT-P-115D 80 cm³
- VMP Naphtha 71 cm³
- Isopropanol 3 cm³
- Total Volume \approx 150 cm³
- DC 732 14 gm

From the cost/coverage table, Table 5-3:

- Non-volatile solids content of MOD. TT-P-115D plus DC 732 = 14 gm + 80 cm³ (0.81 g/cm³) = 78.8 gms
- Estimated film density ρ = 1.1 g/cm³ (average from table)

For a 0.01 cm thick film, coverage rate $A = \frac{0.1\rho}{p}$

Table 5-3
BASIC MATERIAL COST/COVERAGE DATA

MATERIAL	VENDOR'S QUOTED COST	AS RECEIVED DENSITY- gm/cm ³	NON-VOLATILE CONTENT kg./l	ESTIMATED FILM DENSITY gm / cm ³	COVERAGE ⁽¹⁾ l/m ²	COST ⁽²⁾ ¢/m ²	⁽³⁾		
	C		P	P	A				
TT-P-115D, TYPE II	\$6.00 / GAL. (158¢/l)	1.18	0.85	1.1	0.129	20			
MOD. TT-P-115D, TYPE II	\$5.00 / GAL. (131¢/l)	1.14	0.81	1.1	0.136	18	(3)		
PETROSET AT	\$2.00 / GAL. (53¢/l)	1.0	0.63	0.8	0.127	7	(3)		
DRI-SIL 73	\$12.00 / GAL. (316¢/l)	0.923	0.66	1.1	0.166	52	(3)		
DC 92-009	\$50.00 / GAL. (1320¢/l)	0.800	0.28	1.1	0.393	520			
AROTHANE 190M50	\$3.33 / GAL. (88¢/l)	0.891	0.57	0.9	0.157	14			
FC-321	\$18.00 / LB. SOLIDS (1180¢/l)	1.13	0.30	1.5	0.50	590			
RIV 11	\$8.00 / LB. (2000¢/l)	1.15	1.13	1.2	0.106	212			
FREKOTE 33	\$10.00 / GAL. (264¢/l)	1.19	0.006	1.2	20	5300	(CONCENTRATE, IF AVAILABLE, WOULD COST ABOUT 1/2)		
SS - 4044	\$29.00 / GAL. (765¢/l)	0.78	0.103	1.0	0.97	740			
DC 732	\$3.50 / LB. (756¢/l)	1.0	0.98	1.0	0.102	77	(3)		
XYLAN 2052	\$60.00 / GAL. (1585¢/l)	1.2	0.40	2.16	0.54	860			
CARTARETIN F-4	(50¢/LB. SOLIDS (42¢/l)	1.06	0.38	1.0	0.263	11			
NYEBAR F	3750¢ / l	1.7	0.030	1.5	5	18700	(CONCENTRATE, IF AVAILABLE, WOULD COST ABOUT 1/2)		
FOR 0.010 cm FILM; A = 0.1P/P AND ¢/m ² = CA									
VMP NAPHTHA	\$2.00 / GAL. (53¢/l)								
ISOPROPANOL	\$8.00 / GAL. (211¢/l)								
(1) FOR A 0.010 cm THICK FILM USING UNDILUTED MATERIAL AS RECEIVED.									
(2) FOR A 0.010 cm THICK FILM USING UNDILUTED MATERIAL AS RECEIVED WITHOUT NECESSARY SOLVENTS.									
(3) BASED ON ACTUAL DELIVERED COST.									

Where

A is in ℓ/m^2

ρ is in g/cm^3

P is in kg/ℓ

And

Cost/unit area = C A

Where

C is in $\text{\$/}\ell$

Therefore, for the above example

$$A = \frac{(0.1)(1.1)}{\frac{0.0788}{0.150}} = 0.209 \ell/m^2$$

$$\begin{aligned} \text{Cost/Area} &= \frac{0.209}{0.150} [131(0.080) + 53(0.071) + 211(0.003) \\ &\quad + 756(0.014)] \\ &= 35 \text{ \$/m}^2 \end{aligned}$$

5.0 APPLICATION FORMULA

The following BBRC-derived formula was used in applying the coatings to the highways in Phase IV and in determining the application cost for sprayable hydrophobic mixtures.

$$V = 0.06 R_s / f W A$$

Where

V = Required spray vehicle speed, km/hr

R_s = Spray rate, ℓ/minute

W = Lane width, meters

A = Application rate, ℓ/m^2

f = The fraction (if less than one) of a lane treated in one pass

The use of this formula is illustrated in following sections.

6.0 COST SUMMARIES AND COMPARISONS

Material costs have been given in Section 4 above. An estimate of application costs for these materials and the cost of salting is presented below in Sections 6.1 and 6.2, respectively.

6.1 Hydrophobic Material Application Cost

From spraying work in Phase II and in Phase III (see Section 7 below), it is known that all the hydrophobic materials can be applied by a standard roadway distributor.

According to Reference 65, standard distributors are capable of a wide range of application. For this cost analysis, assume 0.28 l/m^2 -- the highest rate used for applying the coatings tested in Phase IV (see Section 7.2). Also assume a spray rate of 18.9 l/minute (5 gallons/minute) which, according to Reference 65, is on the low side. Both assumptions result in a conservative (high) cost. Using the formula from Section 5 and a lane width of 3.66 m (12 feet) we have:

$$\begin{aligned} V &= 0.06 (18.9) / 1 (3.66) (0.28) \\ &= 1.11 \text{ km/hr (0.69 mph)} \end{aligned}$$

For the current operating expense of \$36/hr (Reference 65), the cost is:

$$\text{Cost} = 3600 / 1.24 (1000) (3.66) = \underline{0.9 \text{ ¢/m}^2}$$

or less than one cent per m^2 to apply the hydrophobic coatings.

6.2 Salting Cost

To be able to estimate the cost effectiveness of the hydrophobic coatings, the data on Reference 4 (page 36) for the possible cost savings from replacing salt with a non-corrosive, environmentally-benign material must be updated to the 1975 level used for hydrophobic materials.

Per Reference 65, the current cost of a commonly-used sand/salt mixture (4:1 ratio) is \$0.0127/kg (\$11.50/ton). From Reference 70, 8.2 billion kg (9 million tons) of salt are used per year. This gives a sand and salt material cost of

$$\underline{\text{M.C.}} = 0.0127 (5) (8.2 \times 10^9) = \underline{\$0.5 \text{ billion/year}}$$

From the data of Reference 4, the application cost is estimated to be equal to the material cost or

$$\underline{A.C. = \$0.5 \text{ billion/year}}$$

The vehicle corrosion damage due to salt (and other chemical deicers) is cited as 2 billion dollars per year under very conservative assumptions (Reference 70). Therefore

$$\underline{V.C.D. = \$2 \text{ billion/year}}$$

Also from Reference 70, bridge deck damages in the United States related to salt were estimated to exceed \$0.5 billion/year.

$$\underline{B.D. = \$0.5 \text{ billion/year}}$$

Maintenance cost for cities, counties and townships for spring clean-up of sand (per Reference 71) (including catch basin and sewer cleaning) has been cited as \$.011/kg (\$10/ton) of sand. Based on the commonly-used sand/salt mixture (4:1 ratio) (Reference 65), cleaning cost on an annual basis is estimated 1971-1975 from Reference 68):

$$C.C. = 0.011(1.42)(4)(8.2 \times 10^9) \approx \$0.5 \text{ billion/year}$$

The above totals give a yearly cost of about \$4 billion per year with no consideration of pollution damage or road repair costs. Using the mileage analysis of Reference 4, page 36, (i.e., assume approximately 1 million miles of 30 foot wide roadway to be treated) the allowable applied cost of a hydrophobic coating becomes 28¢/m² which is in the range of some of the coatings investigated here (see Table 5-3).

$$\text{ALLOWABLE APPLIED COST} = 4 \times 10^9 / 1 \times 10^6 (30)(5280)(.09) \approx 28¢/\text{m}^2$$

7.0 SPRAY TECHNIQUES AND CALIBRATION

During the evaluation and screening tests conducted during Phase II (Chapter 4), various application methods for the coating solutions were studied. These are discussed in 7.1 below. The application methods selected for the Phase IV road tests and equipment calibration techniques are reported in Section 7.2.

7.1 Spray Application Techniques

During the Phase II screening tests reported in Chapter 4, many different methods were employed to apply the materials to the test substrates. These included dipping, pouring, painting

and spraying. Since spraying is the only economical application method usable on real-life highway surfaces, the various spray methods evaluated are reported below.

- Conventional air spraying. This method uses air (or a propellant) to draw the fluid from a reservoir and propel the partially vaporized mixture. Various types employed included artist's air brushes, SPRAY-ON brand "Jet-Pack" units and a conventional Binks Model 630 spray gun. In addition to low volumetric capacity, the primary deficiency noted was a tendency for these types of units to vaporize solvent at an excessive rate. The mixtures reached the surface to be treated too dry for even spreading and good penetration into roadway-type substrates.
- Airless (Electric) Spray Units. Although not used at BBRC, the Project Leader (George Ahlborn) studied the use of one such unit (Burgess Model VS-860) in applying the coatings to small (4 m by 4 m or so) surfaces. Although too slow for roadway use, good wetting and penetration on concrete was achieved.
- Compressor-Powered Airless Spraying. This method was selected for application of the coatings in the Phase IV highway tests. Adequate flow rates were possible with very little solvent vaporization before reaching the roadway surface.

7.2 Application and Equipment Calibration

Application of the coatings was performed using two Model ST 7000 Wagner "Spraytech" airless spray units with four #2680 spray tips. The tips were mounted vertically 46 cm above the roadway surface to give a 1.83 m-wide spray pattern (1/2 lane width). Photos are given in Chapter 6.

The spray tips were calibrated using the actual formulations to be supplied. The results were, for four tips operating together:

Formulation A: 9.46 liters/minute

Formulation B: 8.78 liters/minute

Formulation C: 9.46 liters/minute

Including about 10 percent extra for spray losses, the actual application rates were (see Section 4.0 above for sample computation):

Formulation A: 0.226 liters/m²

Formulation B: 0.282 liters/m²

Formulation C: 0.282 liters/m²

Using the formula given in Section 5.0 above, the required vehicle speeds were:

Formulation A = $0.06 (9.46) / 1/2 (3.66) (0.226)$
= 1.37 km/hr (75 ft/min)

Formulation B = $0.06 (8.78) / 1/2 (3.66) (0.282)$
= 1.02 km/hr (56 ft/min)

Formulation C = $0.06 (9.46) / 1/2 (3.66) (0.282)$
= 1.10 km/hr (60 ft/min)

The available truck at BBRC used to carry the compressors, barrels of treatment formulations, the spray tips and operators was not capable of the required low speeds. Accordingly, an International Harvester Tractor Model 340 was rented to pull the truck. The tractor tachometer was calibrated against speed to achieve the required road speed for each formulation.

8.0 ENVIRONMENTAL IMPACT SUMMARY

The expected environmental impact data for the hydrophobic coatings are presented in various sections of this report. The summary, in three categories, is presented below.

8.1 Solid Wear Debris

In Section 5.4 of Chapter 4, the wear rate of the screen-tested hydrophobic coatings was shown to be about equal to the combined tire/asphalt wear rate under the same traffic loads.

Using the same road area as assumed for cost comparison purposes in Reference 4, page 36, we now consider that this area ($1.43 \times 10^{10} \text{ m}^2$) is coated with a hydrophobic material 0.01 cm

thick. If all this material were worn off, we would have 1.5 billion kg of chemically inert debris. The inert character of this debris is verified by Reference 60 data and the known inert character of silicones and of the traffic paint components. Comparing this with the nine billion kg of salt cited in Reference 4, we must conclude that, even if hydrophobic coatings were applied at the same rate traffic paints are applied (about four times the application rate considered here), the hydrophobic coatings would present much less of a quantitative threat than does the salt currently employed. By the same reasoning, coating debris during the winter is only one-half the tire-rubber/asphalt debris generated in the United States during one year (again assuming four times the coating application rate used above and assuming per Reference 4 that only 27 percent of the total highways require deicing).

8.2 Water Soluble Matter

In Section 9 of Chapter 3, data were presented showing that a maximum of about 5 percent (and, more typically, less than 1 percent) of the coatings were water soluble after application. These data were confirmed by hot water extraction tests in Phase IV (see Chapter 6). As was also pointed out in Chapter 4, this water soluble matter (in a more concentrated form than would be expected in real life) had low oxygen demand levels and a pH (acidity) within ± 0.5 units of neutrality compared to the control sample.

On the same basis as that used in 8.1 above, the total water soluble matter that can be obtained from these coatings can at most be 0.05 (1.5×10^9) or 75 million kg for the entire United States. This is literally negligible compared to the more than nine billion kg of salt used per year.

8.3 Air-Borne Contamination

For the two formulations showing promise, this is a real problem during the application of the coatings to the road.

Consider the application of Formulation A to one lane-kilometer of highway. This involves covering 1000 (3.66) or 3660 m² of surface. From 7.2 above, 3660 (0.226) or about 830 liters of solution is required. During application and shortly thereafter the following are evaporated into the atmosphere:

- VMP Naphtha: 393 liters

- Isopropanol: 17 liters
- Shell Tolu-Sol 19EC: 242 liters

A copy of EPA Regulation Number 7 (as adopted by the State of Colorado) is included in Appendix B. Per the requirements of Section G, Paragraphs 2, 3 and 10, the solvents cited above (equivalent to about 900 pounds) would have to be vaporized over at least a two-hour period. However, this only holds if the concentration of aromatics in the Tolu-Sol (which can vary significantly from batch to batch) is such that the concentration of aromatics in the total solvent mixture is less than 20 percent. If the concentration of aromatics in the mixture exceeds 20 percent, the entire mixture must be classified as being photochemically reactive and the rate at which the mixture may be vaporized becomes impracticably low.

Time did not permit BBRC to experiment with paint formulations (one ground rule was the use of commercial materials). It is certain that photochemically reactive volatiles could be eliminated. However, for the classes of materials showing promise for *application to existing roadways*, some organic solvent appears to be a necessity. This remains a problem area for this concept. Certainly, a flammability problem also exists. However, in the application of Formulations A and C to highways in Phase IV, reasonable precautions and the very rapid dissipation of the solvents (requiring only a few minutes in a two km/hr wind) created no problems.

Chapter 6

PHASE IV HIGHWAY APPLICATION AND TESTING

1.0 INTRODUCTION

In this chapter are presented the program efforts during the application of three coatings to selected sites, visual observations of these sites during the winter of 1974-1975 and post-winter field and laboratory evaluations. The three coatings applied have been completely defined in Chapter 5.

2.0 SITE SELECTION AND DESCRIPTION

The decisions involved in test site selection, the geographical location of the sites and a precise definition of the coatings' application configuration are given in this section.

2.1 Site Selection Decisions

Site selection factors and related decisions are given below.

- Snow Fall. Per Reference 22, the entire State of Colorado historically experiences snow falls in excess of 50 cm (20 inches) per winter season and the area near Boulder, Colorado, typically has snow ground cover over 60 days per winter season. More precise data could not be obtained due to the recent discontinuance of season weather records by local agencies (see "Other Contacts" in Chapter 3). Personal observations by the Project Leader indicated severe icing conditions in prior winter seasons periodically in the Boulder area.
- Nearness to BBRC. It was originally proposed that at least some of the test sections be located in the high mountain country near Boulder, Colorado. Three practical factors eliminated this concept:
 1. Extensive travel and cost would be required to check such areas. Rapid checking by aerial flights was ruled out due to dangerous flying conditions in these areas and further contacts with aerial photographic experts and airports indicating a two-day notice before flights could

be made. In this region, the two days are long enough to completely change the surface conditions.

2. The (justifiable) refusal by the Colorado Department of Highways to allow application of the coatings to areas of extreme hazard (such as curved mountain roads) until the coatings had proved not to be skid hazards in themselves in less hazardous locations.
 3. For safety reasons, the Highway Department is required to salt/sand or plow roadway areas as soon as hazardous conditions become evident. Efforts were made to avoid such treatment of the coated sections but this was not always possible. Accordingly, areas near BBRC were selected to permit rapid (prior to sanding or plowing) and frequent inspection with a minimum of expense.
- Road Surfaces. It was considered a necessity that locations include:
 1. Both concrete and asphalt in adjacent sections at one high traffic location.
 2. Worn asphalt in a high traffic location.
 3. Asphalt in a low traffic location.
 4. Asphalt in a zero traffic location.
 5. Concrete in a zero traffic location.

This exceeded the original scope but was felt necessary to gain the maximum useful data.

- Proximity of Sites. Due to the expense of recording weather data at five sites (some \$8000 for two sites), it was considered a requirement that the sites not only be near BBRC but also in close proximity to each other to be able to assume that at least temperatures would be relatively constant from site to site.
- History. To satisfy ourselves and the Colorado Department of Highways that the coatings were not safety hazards, it was necessary to have at least two locations for which the headquarters of the

Colorado Department of Highways could provide accident records. These accident reports are included in Appendix D and will be referred to in a later section.

2.2 Geographical Site Location

The geographical locations of the sites are defined in Appendix C, Figures C-1 and C-2.

Figure C-1 of Appendix C shows the relationship of the local area to the Denver Metropolitan area in north-eastern Colorado.

In Figure C-2 of Appendix C are indicated all the test areas employed in the program. Indicated areas are:

- The wear-test location used in Phase II (Chapter 4).
- BBRC, where the Phase II friction and degradation tests were made (Chapter 4) and where the zero-traffic asphalt and concrete sites used in Phase IV were located.
- The asphalt-only location (medium wear, high traffic) required in Phase IV (Highway 7).
- The high-traffic-load location with adjacent sections of new asphalt and medium-worn concrete (Highway 36).
- The very worn low-traffic-load asphalt section used in Phase IV (East Pearl Street).

Note that hereafter in this chapter the above locations will be referred to simply as

- BBRC asphalt and concrete
- Highway 7
- Highway 36 asphalt and Highway 36 concrete
- East Pearl Street

Except for the BBRC sites, all test sections were located on straight and level sites where traffic could move at relatively constant speeds.

2.3 Site Application Configuration

The exact location, configuration and size of the treated areas are defined in the figures in Appendix C. None of the figures are drawn to scale.

Figure C-4 defines the areas used at BBRC.

Figure C-5 shows the Highway 7 coating placement and the estimated traffic load based on October, 1974 data.

Figure C-6 gives the same information for Highway 36 concrete, and Figure C-7 gives the data for Highway 36 asphalt. (Coating A in Figure C-7 was about 100 m west of coating C in Figure C-6.)

Figure C-8 shows the East Pearl Street location.

3.0 APPLICATION OF COATINGS

The application of the coatings is described in this section. The application rates for formulations A, B and C, their exact composition, the equipment employed and its calibration and application considerations have been defined in Chapter 5. Presented below are the details of the actual coating operation.

3.1 Coating Application Factors

Various factors and procedural considerations were:

- The three formulations were mixed about two weeks prior to application. Very little separation for even the paint formulation was observed.
- Application dates were controlled by projected weather forecasts from the U. S. National Weather Service in Denver. Dates and approximate application times are summarized in the tabular observations summary sheets in Section 4 below.
- No surface pre-treatment (washing, sweeping, etc.) was employed nor was any post-treatment used.
- Formulations were applied in the time sequence "C, B, A" since the drying times decrease in the same sequence. This minimized tracking of one coating over following sections.

- Adjacent-lane traffic was stopped during the few minutes of actual spraying for each 1.83 m (1/2 lane) strip of each section to eliminate any hazard from the naphtha vapors.
- Treated highway sections were opened to traffic about two hours after the last section was coated.
- Skid test data were taken shortly after application. These data and final skid values are given in Section 5 below.

3.2 Conditions and Observations

Prevailing conditions and observations during the actual applications are summarized below:

- BBRC Phase II and IV Concrete:
 - Date: 7/11 - 7/12/74
 - Temperature: Pavement + 37 C
 - Wind: Nil
- Highway 36 Concrete:
 - Date: 12/12/74
 - Temperature: Air + 10 C; Pavement + 14 C
 - Wind: 10-20 km/hour
 - Petroset "fisheyed"
 - Drying faster on concrete than asphalt
- Highway 36 Asphalt:
 - Date: 12/12/74
 - Temperature: Air + 10 C; Pavement + 14 C
 - Wind: 10-20 km/hour
 - Petroset "fisheyed"

- Highway 7 Asphalt:
 - Date: 12/13/74
 - Temperature: Air + 4 C; Pavement + 6 C
 - Wind: 4-6 km/hour
- East Pearl Street:
 - Date: 1/14/75
 - Temperature: Air + 7 C
 - Wind: Gusts to 35 km/hour
 - Coatings somewhat uneven due to wind
- BBRC Phase IV Asphalt:
 - Date: 2/13/75
 - Temperature: Air + 10 C; Pavement + 15 C
 - Wind: Nil

3.3 Photos

Figure 6-1 shows the application truck with compressors, spray tips and barrels containing the coating formulations during application on Highway 36.

Figure 6-2 shows the entire application rig including the tractor found necessary to achieve the low road speed required due to our low total spray rate.

Figure 6-3 shows the control possible at the highway edge (thus little overspray and vegetation damage on roads with narrow shoulders).

Figure 6-4 shows the first one-half lane strip of formulation B immediately after its application to Highway 36. The section of Formula C that had been applied earlier in the day can be seen in the background.

Figure 6-5 shows a closeup of the second one-half lane strip of Formulation A being applied to Highway 36 concrete. Note the small amount of drifting vapors with this technique and

the even spray pattern ("fisheyes" in completed strip were due to lack of pre-cleaning).

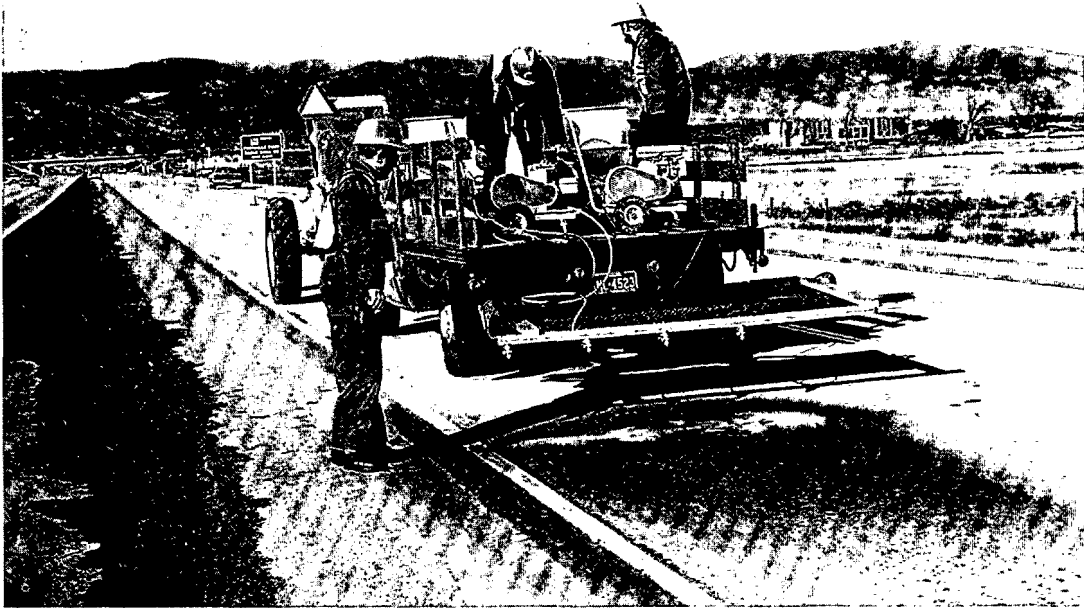


Figure 6-1 Application Truck on Highway 36 Showing Barrels of Coating Formulations and Spray Apparatus

Figure 6-6 shows a complete lane with Highway Department traffic control in background.

By early February, 1975, it became apparent that definitive results might not be obtained from the Highway and street sites. Accordingly, the three formulations were applied to asphalt at BBRC, using air rather than airless spraying.

Figure 6-7 shows:

- a and b: Formulation A on BBRC Phase IV asphalt. Note poor condition of asphalt. Note also uneven spray pattern due to use of air type spray gun in this case (see discussion of spray methods in Chapter 5).



Figure 6-2 Entire Application Rig During Coating of Highway 36

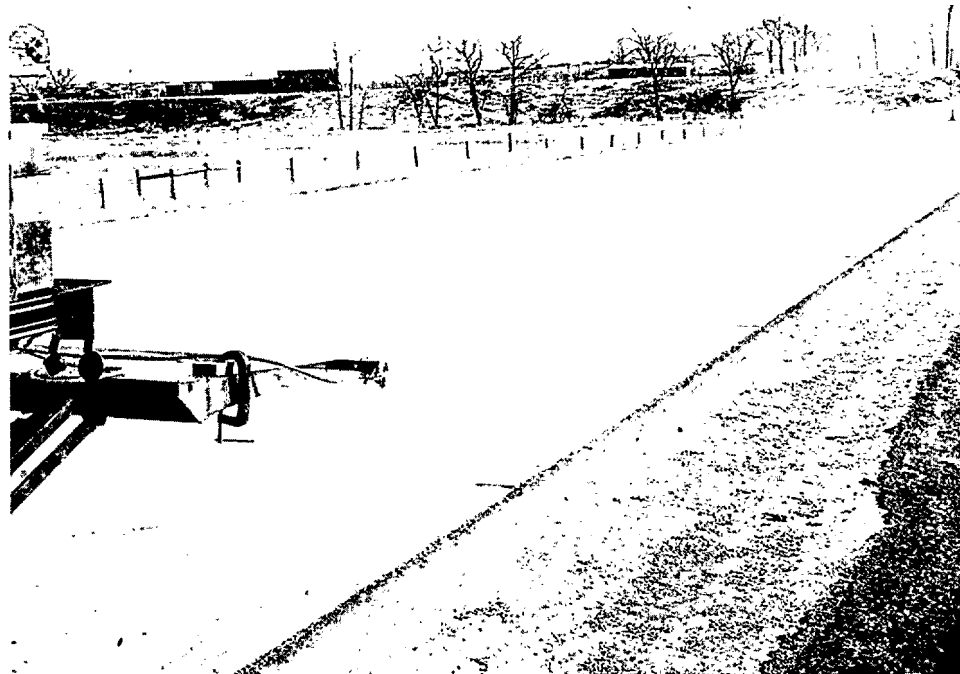


Figure 6-3 Application of Coating on Highway 36 Concrete Showing Level of Edge Control Obtainable



Figure 6-4 First One-Half Lane Strip of Formulation B Applied to Highway 36 Concrete

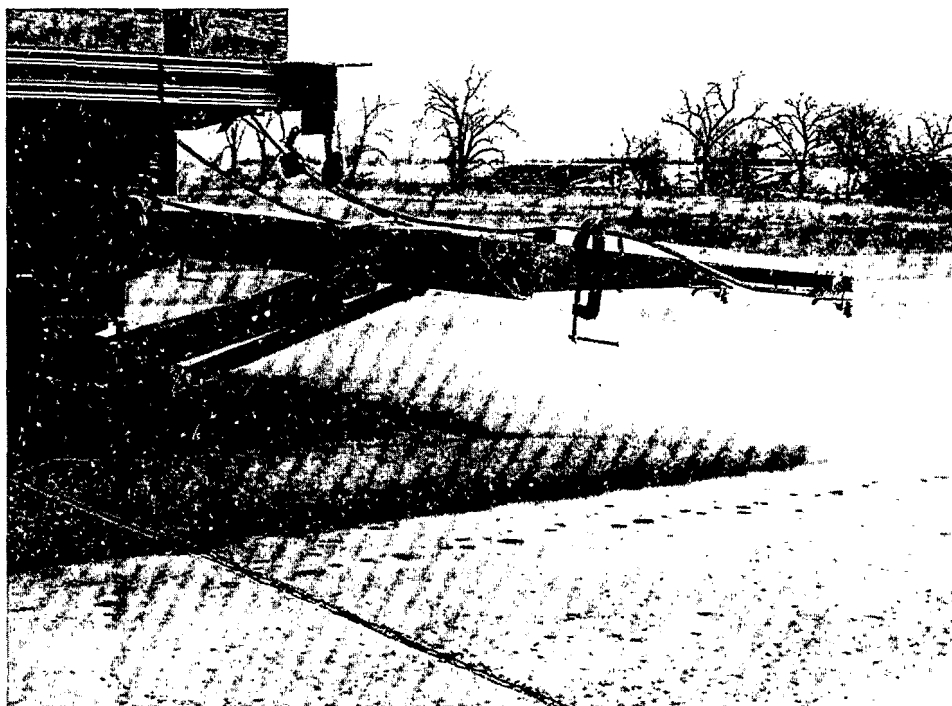


Figure 6-5 Application of Formulation A to Highway 36 Concrete Showing Even Spray Pattern and Small Amount of Vapor Drift

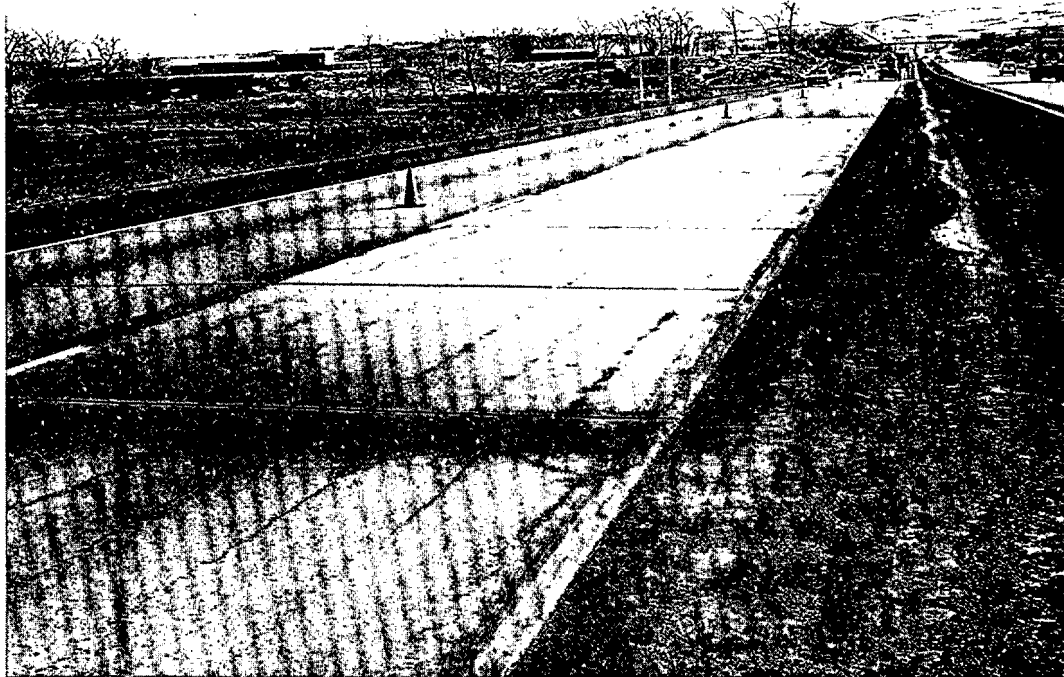


Figure 6-6 Completed Application of Formulation B to Highway 36 Concrete

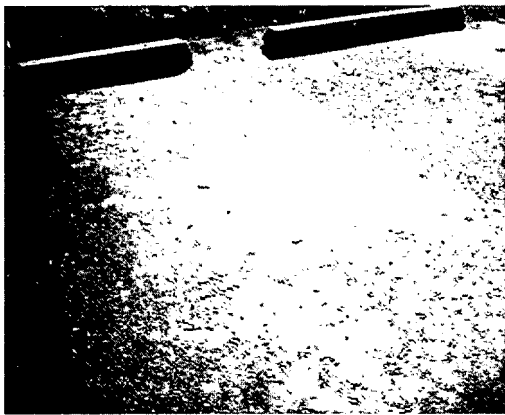
c and d: Formulation B on BBRC Phase IV asphalt. Same comments as above.

e and f: Formulation C on BBRC Phase IV asphalt. Same comments as above.

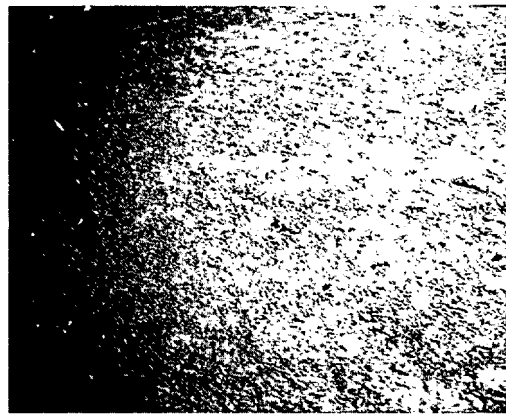
3.4 Conclusion

Due to the planning in Phase III, less difficulty than expected was experienced in applying the coatings. Of general note are:

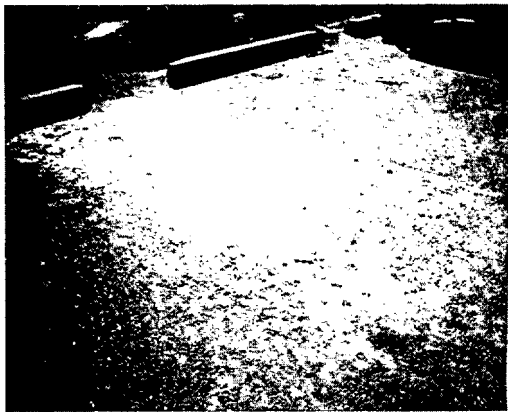
- Application was quite even except for the Pearl Street location where winds were quite high.
- The superiority of airless versus air gun spraying was again demonstrated.
- Formulation A (Paint/DC 732) sprayed with the least "fog".



(a)



(b)



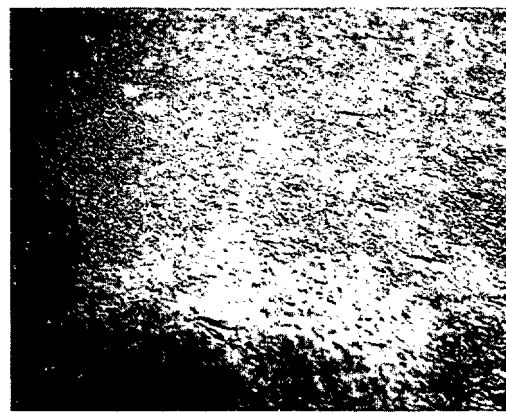
(c)



(d)



(e)



(f)

Figure 6-7 Formulations A, B and C on BBRC Asphalt Test Site

- The Petroset AT (Formulation B), being a water-based mixture, went on the most unevenly but evened out to virtual invisibility on asphalt in a few hours.
- Formulation C (Dri-Sil 73/DC 732) had the most "fog" but this could be corrected by cutting solvent quantity.
- Adverse weather the week before application resulted in the Highway 36 and 7 surfaces being slightly damp during application. However, this is probably a typical real-life condition so the test was considered valid in this respect.

It must be noted that a standard distributor could not be used due to the small quantity of material being applied (less than 100 liters per formulation per day). Use of standard equipment would permit faster, more uniform application.

4.0 WINTER OF 1974-1975 OBSERVATIONS

Visual observations, temperature and precipitation data and qualitative ice release data for the six outdoor test sites during the winter season are presented below.

4.1 Measurement Methods

- Visual observations of release effectiveness were made by at least two observers. Where there was disagreement, this is indicated.
- Precipitation amounts were based on broadcast data and at-the-site measurement.
- Temperatures were measured at the site during the visual measurements using an Omega Engineering Model T-151C41 electronic temperature indicator.
- Qualitative estimates of wind velocity were also made.

It was regrettable that so much of the data were qualitative. However, especially in the case of weather factors, even the nearest station at Jeffco Airport (about ten miles from the sites) could not supply the quantitative data desired.

4.2 Observations and Data

Table 6-1 presents the recorded weather data for every date observations were made at any site. Note that the precipitation

Table 6-1
WEATHER DATA DURING ROAD TEST PERIOD

Date	Temperatures		Precipitation		Precipitation Type
	Surface	Air	Amount		
12-23-74	-1C		5	cm (2 in)	Dry, fluffy snow, light wind
1-9-75			2.5	cm (1 in)	Dry, powder snow, light wind
1-21-75	5C	- 3C	7.5	cm (3 in)	Moderately wet snow, no wind
2-4-75	2C		Trace		Freezing rain, some sleet
2-14 to 2-17-75	-2C		25	cm (10 in)	Wet snow, light wind
2-18-75	-1C	-10C	None		
3-7-75	6C	- 2C	None		
3-10-75	2C	- 3C	Trace		Wet snow, no wind
3-12-75	2C	- 4C	None		
3-13-75	1C	- 4C	None		
3-26 to 3-28-75	-1C	- 7C	20	cm (8 in)	Heavy wet snow, no wind
4-1-75	2C	- 6C	10	cm (4 in)	Heavy wet snow, moderate wind
to					
4-3-75	-2C	-12C	20	cm (Acc) (10 in)	Heavy wet snow, moderate wind

amounts, up through mid-March, were far below seasonal normals (see 2.1, above). In addition, surface and air temperatures were above normal during this period. Note also the large differences between air and ground temperatures and the rare periods when surface temperatures would allow ice formation. By the end of March when weather conditions became more favorable for test purposes, the coatings on Highways 36 and 7 were over three months old and no longer effective, especially in the wear tracks. (However, see Section 5.3 data below.)

Table 6-2 presents the observations for Highway 36. The data for the concrete section were particularly disappointing. Several causes for this were:

- Heavy sanding. Due to the presence of an exit at the end of the section, the interests of safety overrode test considerations.
- Concrete was rather old and weathered, which probably accelerated the coating wear.
- Concrete, being far more porous than asphalt (see Reference 54), may well require higher application rates than used here or a lower solvent ratio to reduce penetration.

The asphalt section on Highway 36 yielded some data. Up to about one month of age, all coatings appeared to have a beneficial effect. After nearly two months, Formulation C remained effective.

Table 6-3 gives the data for Highway 7 and Pearl Street. On Highway 7:

- Early observations indicated release from Formulations A and B.
- At about two months after application Section A was still showing qualitative ice release.
- At three months, Formulation C was demonstrating lower adhesion than the untreated roadway.
- At no time did qualitative car braking tests indicate a skid danger from the coatings applied.

The Pearl Street location yielded little data. The asphalt here was quite worn. This, combined with the high winds

Table 6-2
ROAD TEST OBSERVATIONS ON HIGHWAY 36

DATE	CONCRETE (APPLIED 12-12-74 ~1-1/2 Hrs)	ASPHALT (APPLIED 12-12-74 ~1-1/2 Hrs)
12-23-74 1500	Heavy traffic and light wind did not allow the snow to accumulate on the pavement. No meaningful data could be taken.	Heavy traffic and light wind did not allow the snow to accumulate on the pavement. No meaningful data could be taken.
1-9-75	Very light, dry snow, light wind. No meaningful data could be taken.	Very light, dry snow, light wind. No meaningful data could be taken.
1-21-75	No difference could be distinguished between treated and untreated sections of roadway.	Noticeable difference could be observed between treated and untreated areas, especially Section C. All treated sections appeared freer of snow and slush than untreated sections.
2-4-75	No differences could be observed. This site had been heavily sanded.	Section C appeared to have less snow and ice accumulated than the other areas, both treated and untreated. All sections had been heavily sanded.
2-14 to 2-17-75	This was a heavy wet snow. When the area was first observed, the snow cover was too thick to tell if anything was happening on the pavement. When the area was next observed, it had been plowed and all that remained was slush.	This was a heavy wet snow. When the area was first observed, the snow cover was too thick to tell if anything was happening on the pavement. When the area was next observed, it had been plowed and all that remained was slush.
3-10-75	No ice had formed. The area was very heavily sanded. No difference could be seen between coated and uncoated surfaces. It appeared that the coatings were very nearly gone.	Not as heavily sanded as the concrete. The traffic paths were about equally dry on treated and untreated sections
3-26 to 3-28-75	Highway 36 will no longer be reported. The extreme heavy sanding on this road, the wear of coatings (applied in the high speed lane) and the high traffic density (resulting in very rapid "wear through" of snow or ice in all sections) have made further observations non-productive.	

Table 6-3
ROAD TEST OBSERVATIONS ON HIGHWAY 7 AND PEARL STREET

DATE	HIGHWAY 7	PEARL STREET
	ASPHALT (APPLIED 12-13-74 ~2 Hrs)	ASPHALT (APPLIED 1-14-75 ~1-1/2 Hrs)
12-23-74 0800	Light powder snow, ~1.25 cm (1/2 in.) on curbside lane. Traffic on inside lane had removed most of the snow from treated and untreated areas. Sections of A and B, on the outside, (curbside) lane showed more snow displacement and more bare surface, than untreated areas. No difference could be distinguished between Section C and untreated sections.	
1530	About 2.5 cm (1 in.) of dry snow had accumulated. The inside lane was still bare due to heavy traffic. No difference could be distinguished between treated and untreated areas in outside lane. Drove across test areas at 30 to 35 m.p.h. alternately applying brakes lightly and accelerating to regain speed. Could tell no difference between treated and untreated sections.	
12-24-74 0800	The snow had packed in the outside lane, the inside lane was clear. No difference in snow pack could be distinguished.	
1330	The packed snow appeared to be releasing from the surface and breaking up on Sections A and B. This could not be seen on Section C.	
1-9-75	The light wind kept most of the snow off the pavement, but a slight difference could be seen between Sections A and B and untreated areas. No difference could be seen on Section C.	
1-21-75	No difference could be seen, in the packed snow, between the treated and the untreated sections, however the packed snow on Section C did seem darker in color than other sections.	More snow on treated sections. This street has light traffic normally, so the snow had more of a chance to accumulate. It appeared that the coating was insulating the snow from the ground warmth.
2-4-75	Section A released ice more easily than untreated sections. This was determined by chipping the ice away on both treated and untreated areas. There was no difference in "slipperiness" between treated areas. "Slipperiness" was determined by driving a car across the area and lightly applying the brakes, then accelerating.	None of the sections, treated or untreated were iced. No difference in "slipperiness" could be determined. Slipperiness" was determined by driving a car across the area and lightly braking and accelerating.
2-14-75	No differences in accumulated ice slush. Chipping at the ice on Section A did indicate low adhesion. No other observations could be made due to the heavy sanding.	No ice had formed at this site.
3-10-75	Some possible differences could be observed. Test site was very heavily sanded.	This site was not checked.
3-26-75	Section C appeared definitely clearer of packed snow than untreated areas.	This site will no longer be checked. Heavy sand-truck traffic from city of Boulder yard makes effects difficult to judge.
4-1-75	No observations made.	

during application, probably resulted in poor penetration of the coatings and a short life. However, one dramatic effect was the ability of the coatings to apparently insulate the ground from the colder air above.

Although the three formulations applied to asphalt at BBRC were not as well applied as they were on the highways (since air, rather than airless spraying, was employed), they did yield positive results. Table 6-4 gives these observations for the BBRC, Phase IV, asphalt and concrete sites (see Appendix C, Figure C-4). For asphalt, note:

- The insulating effect again.
- On 3/13/75 and 4/2/75, the dramatically easier release for the coatings.

The concrete site was notable for reconfirmation of the insulation effect and the good conditions of the coatings after nine months of weathering.

4.3 Photos

Photos could not be obtained at the highway sites. When differences were visually apparent, light level and direction were such that sufficient photographic contrast could not be recorded. It is suspected that aerial photos might have worked but, as explained in Section 2.1 of this chapter, the two-day notice required for flights is much too long a delay.

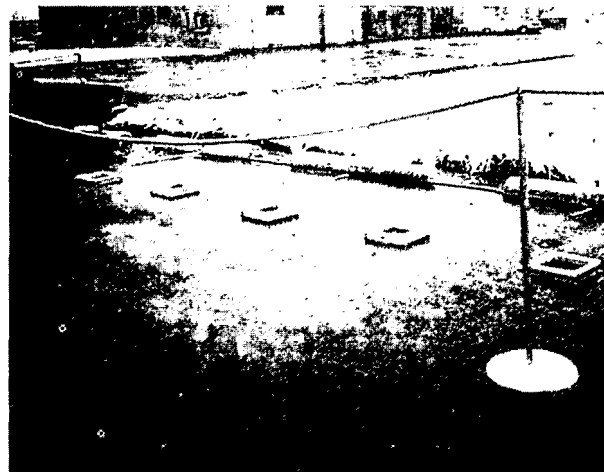
Figure 6-8 shows the BBRC Phase IV asphalt after a brief snow on 3/10/75. Figure 6-8a is an overall view showing the frames used to hold water for ice release tests placed on treated and untreated sections of the site. Figures 6-8b, c and d are formulations A, B and C, respectively. Note the beading up of water near the frames in b and c and the excellent beading over the entire area of d.

Figure 6-9 illustrates the release data of 3/13/75 on the BBRC Phase IV asphalt. 6-9a and b are formulations A and C and 6-9c and d are adjacent, untreated areas. Note the release of large chunks of ice in a and b and the inability to force release in c and d.

Figure 6-10 illustrates the ice release observed on 4/2/75 on the BBRC Phase IV asphalt. 6-10a, b and c are formulations A, B and C, respectively. Note the release of large ice chunks in all three cases.

Table 6-4
OBSERVATIONS ON BBRC ASPHALT AND CONCRETE SITES

DATE	BBRC PARKING LOT (APPLIED TO ASPHALT 2-13-75 ~4 Hrs)	BBRC	BBRC CONCRETE SIDEWALK (APPLIED 7-11-74 ~4 Hrs)
2/18/75 0730	Most of the snow had melted off of the parking area the previous day. Water was poured on treated and untreated areas. The ice was tapped and chipped to check release. The treated sections appeared to release the ice more easily than the untreated sections.	9/3/74	Lots 1,4,7,8,9 had poor beading. Lots 3,5 had good beading Lots 2 and 6 beading was medium
2-27-75	Wooden frames were placed on treated (#1, #3, #5) and untreated (#2 and #4) areas to contain water. When ice is formed the areas will be checked for ice/pavement adhesion.		
3-7-75	Frames had film of ice. No data taken.		
3-10-75	Ice not formed, but beading evident near frames on sections A and B.		
3-12-75	Ice frozen to pavement only in frame #2 (untreated area). Ice would not release with screwdriver. The ice was nearly frozen to pavement in frame #4 (untreated area). The frames on the treated surfaces all had 1-5mm of water at the bottom. This could indicate the insulating effect of the coating again.		
3-13-75	Ice frozen in all frames except #3. Number 3 frozen along one edge. The ice in frame #1 released in large chunks only at ice/pavement interface. Ice in frame #2 (untreated) could only be chipped away in small shards. It would not release at ice/pavement interface. The ice in #3 released only at ice/pavement interface. The ice in frame #4 was frozen to the untreated pavement and could not be broken away. The ice in frame #5 released at the ice/pavement interface, but not as easily as in frames 1 and 3.		
4-1-75			The two sections treated with traffic paint mixtures were covered with snow, while the remainder of the sidewalk was wet. This could indicate, once more, the insulating effect of the coatings containing paint.
	Sections A and C released excellently at the ice/pavement interface. Section B also released excellently, but was partly melted at ice/pavement interface. The untreated sections would not release from the pavement. The frames could not be chipped away without damage to the frames.		



(a)



(b)

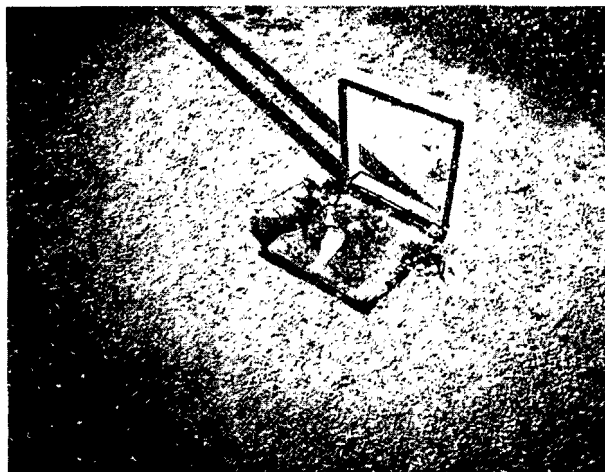


(c)

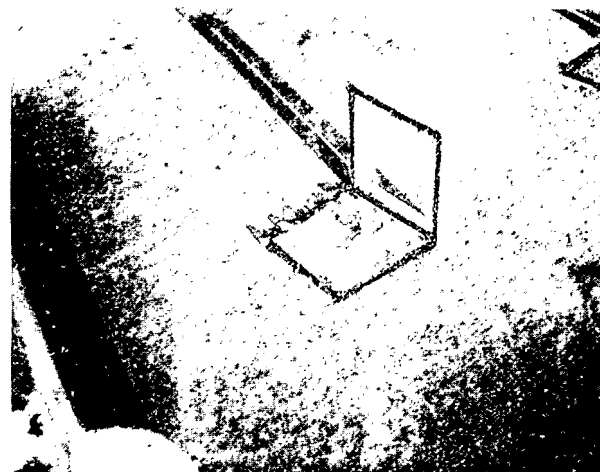


(d)

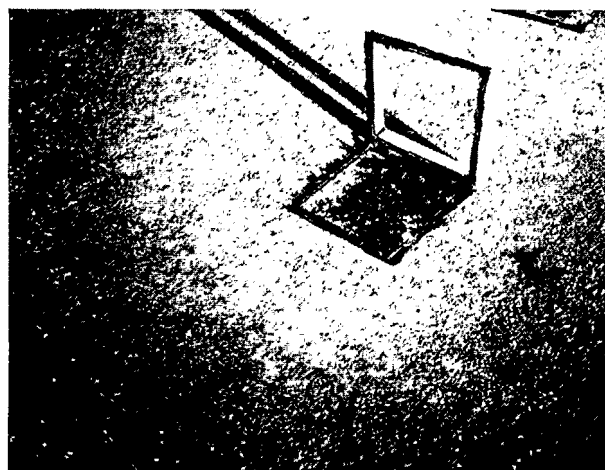
Figure 6-8 BBRC Asphalt Test Site, Showing Overall View and Hydrophobicity of Tested Formulations A, B and C



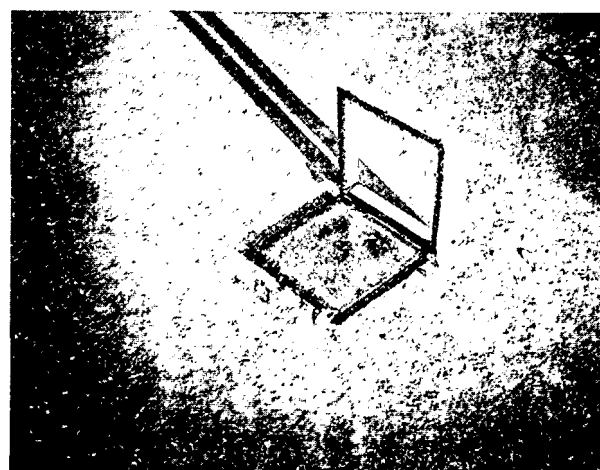
(a)



(b)



(c)



(d)

Figure 6-9 BBRC Asphalt Test Site Showing Release of Ice From Sections Treated with Formulations A and C Compared to Lack of Release From Untreated Sections



(a)



(b)



(c)

Figure 6-10 BBRC Asphalt Test Site Showing Release of Ice From Sections Treated with Formulations A, B and C

Figure 6-11 gives four views of the BBRC concrete sidewalk during April 1975. Figure 6-11a dramatically shows the insulation effect on sections 5 and 6 coated with paint formulations (see Chapter 4). Figure 6-11b shows the beading of water on section 3 (Formulation C). 6-11c and d illustrate the degradation of two Arothane formulations due to nine months of weather exposure. The darker areas are the coating in the process of peeling off.

4.4 Conclusion

A complete discussion of the Phase IV data are given in Section 6.0 below.

As regards the observations in this section:

- Formulation A appears to release ice most easily but all three demonstrate the ability on unworn (but weathered) asphalt.
- Formulation C appears to have the longest effective wear life on asphalt.
- Formulation C's effectiveness seems to improve with (exposure) age. This is reasonable since both components cure by exposure to water.
- The insulating effect, especially of the paint formulation (A), is quite pronounced. This might be a helpful feature on such structures as bridge decks.
- Coatings applied on concrete seem to have shorter wear life than on asphalt. Higher application rates may be needed on concrete.
- Formulation B was still *visible* on Highway 36 asphalt as of July 1975.

5.0 POST-TEST DATA

As a means of clarifying and quantifying some aspect of the Phase IV highway and lot test work, a number of post-test examinations and tests were performed. These data, including (a) wear-life computations, (b) skid-test measurements, (c) ice adhesion tests on core samples and (d) environmental contamination tests (including more severe ultraviolet and water solubility evaluation than performed in Phase II -- Chapter 4), are presented below.

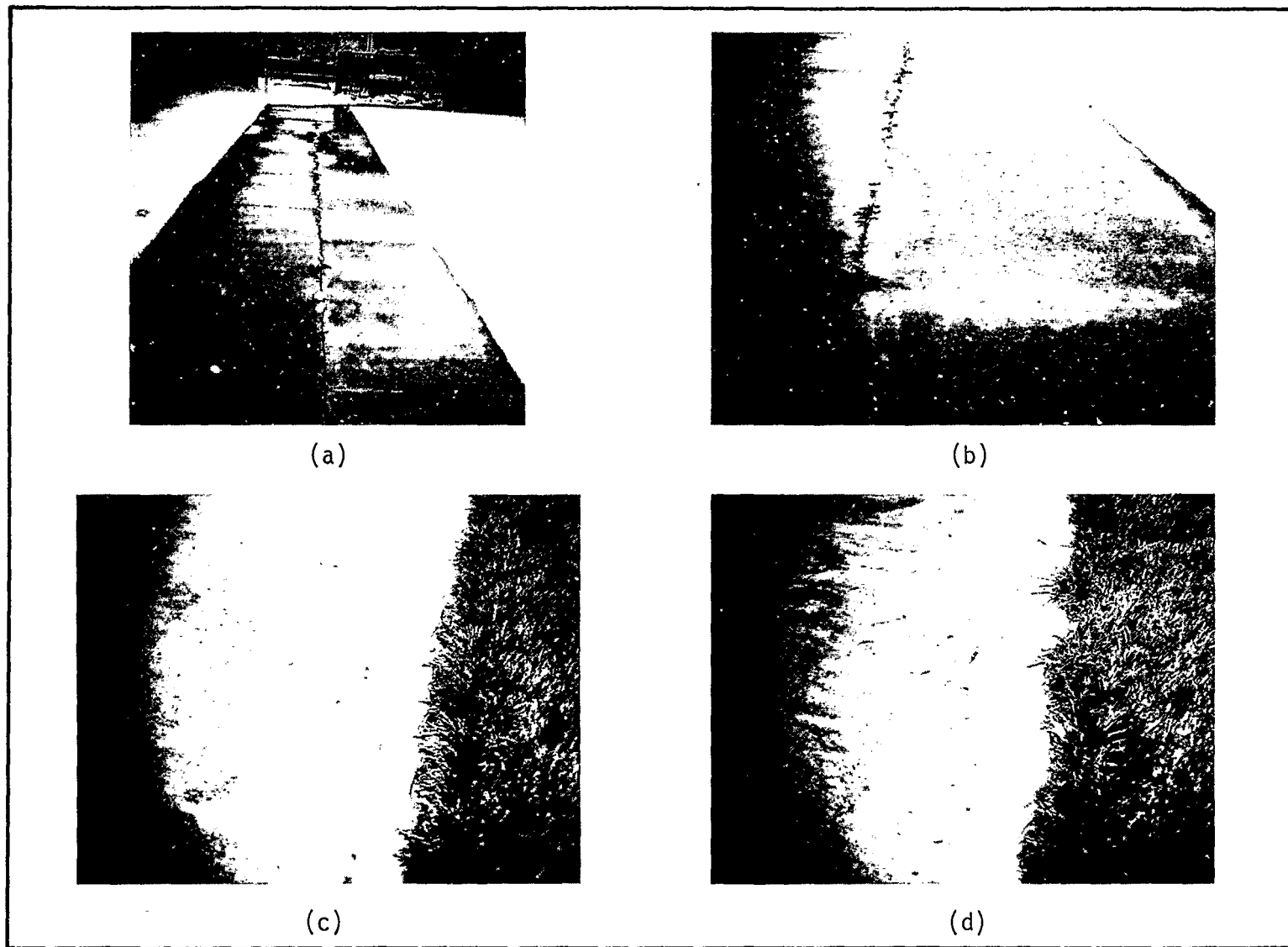


Figure 6-11 BBRC Concrete Test Site Showing Insulating Effect, Hydrophobicity and Degradation of Various Coating Materials

5.1 Wear-Life Estimates

In Section 5 of Chapter 4, visual observations indicated 20 to 30 percent wear after about 200,000 vehicle passes for coatings similar to the three tested here. The observations were based on estimates of the amount of the wear stripes gone (across the lane width) and the wetting/non-wetting of these stripes during skid tests. The following are based on high traffic load areas. See Appendix C for the traffic loads per lane day.

Because of heavy sanding on Highway 36 concrete (see Section 4.2), little can be said about the efficacy of the ice-release coatings. We can only say that all three coatings were gone after 500,000 vehicle passes.

On Highway 36 (asphalt):

- All three formulations appeared effective after 192,000 vehicle passes.
- Formulation C appeared effective after 270,000 vehicle passes.

On Highway 7 (asphalt):

- All three formulations were effective after 75,000 vehicle passes.
- Formulation A exhibited qualitative ice release after 151,000 vehicle passes.
- Formulation C appeared effective after 228,000 vehicle passes.

As indicated in connection with the tests discussed in 5.3 below, dirt and sand ground into the coatings had a more detrimental effect than was originally expected.

5.2 Skid Test Measurements

As shown in Chapter 4, Section 6, friction tests with the portable skid tester are a good indication of the presence of the coatings. Skid test measurements were made at the six locations discussed in this chapter, both a few days after application and on April 14, 1975, when further sub-zero weather appeared unlikely. These data are presented below.

5.2.1 Numerical Data

The numerical data are presented in Table 6-5. Values for the uncoated surface are given in the "control" column. Note the following from the values:

- On Highway 36 concrete, only formulation C is still present along the edge.
- On Highway 36 asphalt, all formulations are apparently still present along the edge judging by beading. Skid values for formulation C also indicated the presence of the coating.
- On Highway 7, beading again indicates the presence of all three at the edge with the presence of formulation A also being evidenced by its skid values.
- On East Pearl Street, the coatings were no longer visible and only one check was made, which showed that formulation A was in fact gone.
- On BBRC parking lot 2, the coatings were, of course, still present with the formulations again showing their trends with age (i.e., formulation A, remaining relatively constant, formulation B becoming less slippery -- the Petroset being a water-based slow-cure material -- and formulation C becoming somewhat more slippery).
- The concrete sidewalk also used in Phase II (Chapter 4) shows the same trends as the BBRC asphalt, although the baseline value has shifted. The formulations are indicated with A and C still showing excellent hydrophobicity after nine months of weathering.

In the high traffic areas, all coatings are gone. As is shown here and well recognized by highway experts (Reference 50, for example), worn highways are more slippery than new surfaces. Also, worn surfaces seem to retain the coatings less well than newer ones. It is hypothesized that concrete becomes more porous and has less strength while asphalt becomes compacted so the coatings cannot penetrate sufficiently to strongly adhere.

Table 6-5
PHASE IV HIGHWAY SKID TEST VALUES

TEST SECTION			TEST CONDITIONS			SKID TEST VALUES				
AREA	SUBSTRATE AND CONDITION	DATE COATING APPLIED	DATE TESTED	TEMP. (C)		CONTROL	FORMULATION A	FORMULATION B	FORMULATION C	
				AIR	ROAD					
HIGHWAY 36	CONCRETE/WORN	12/12/74	12/18/74	4	7	62/62/62/62	50/50/50/50(B)	63/62/63/62(B)	50/49/50/50(B)	
			4/14/75	6	10	65/65/63	71/72/70-EDGE 77/77/76-CENTER	64/64/62-EDGE	54/54/55-EDGE(B)	
HIGHWAY 36	ASPHALT/NEW	12/12/74	12/18/74	4	7	66/65/66/66	53/52/52/51(B)	53/52/52/52(B)	60/58/56/59(B)	
			4/14/75	6	10	75/75/77-EDGE 71/70/70-CENTER	73/73/73-EDGE(B) 70/69/70-CENTER	73/73/74-EDGE(B) 73/70/71-CENTER	69/69/70-EDGE(B) 71/71/71-CENTER	
HIGHWAY 7	ASPHALT/VARIABLE	12/13/74	12/18/74	6	10	70/70/70/73	50/49/49/52(B)	60/60/60/60(B)	56/65/59/56(B)	
			4/14/75	10	14		60/61/60-EDGE(B) 73/72/72-CENTER	75/73/73-EDGE(B) 71/71/71-CENTER	70/70/70-EDGE(B) 73/74/74-CENTER	
EAST PEARL ST.	ASPHALT/VERY WORN	1/14/75	1/17/75	6	10	54/58/60/60	44/45/48/47(B)	52/54/55/55(B)	43/49/50/48(B)	
			4/14/75	11	15		53/59/60-CENTER			COATINGS APPARENTLY GONE
BBRC PARKING LOT 2	ASPHALT/WORN	2/13/75	2/21/75	0	12	74/74/75	63/63/63/63(B)	71/71/71/70(B)	76/77/77/78(B)	
			4/14/75	8	22		63/62/63(B)	75/75/74(B)	72/73/74(B)	
CONCRETE WALK AT BBRC	CONCRETE/GOOD	7/11/74	7/15/74	26	32	73/75/74/73	79/78/82	53/53/50	67/67/68	
			4/14/75	8	20	66/66/65	57/60/61 (HIGH 3)	77/77/76	54/54/54 (HIGH 3)	

NOTES: B=BEADING OF WATER NOTED DURING TEST

CENTER = CENTER OF TESTED LANE IN WEAR TRACKS

5.2.2 Conclusion

Perhaps most important to note is the visible presence of these thin coatings in some areas over four months of traffic. This certainly implies low wear over a normal winter period.

5.3 Ice Adhesion Tests

On 24 March 1975, 10 cm diameter core samples were taken from selected locations on Highway 7 and from the BBRC asphalt lot and subjected to laboratory ice adhesion testing.

A photograph of the cores is shown in Figure 6-12. Note that even after washing the surfaces (Reference 59) considerable ground-in road dirt is still visible on the formulation A samples (second from left). Dirt is composed mainly of clays and silicates -- both of which are hydrophilic. This illustrates another practical problem in the evaluation of hydrophobic coatings for real-life use on highways.



Figure 6-12 Ice Adhesion Test Core Samples
from Highway 7 and BBRC Asphalt

5.3.1 Numerical Data

The data are summarized in Table 6-6. These tests, as well as the core tests in Phase II (References 57, 58 and 59 in Appendix A), were conducted by soaking the cores in water at ambient temperature for two hours before testing. The porosity of a particular core sample may account for the variability in results.

Table 6-6
PHASE IV HIGHWAY CORE ICE ADHESION TEST RESULTS

SITE / LOCATION	ADHESION VALUES - kg/cm^2	DATE CORES TAKEN	FORMULATION	\bar{X} kg/cm^2	R kg/cm^2	SIGNIFICANCE OF DIFF. FROM CONTROL
HIGHWAY 7: CONTROL	10.8/11.6/13.5/11.6/14.8/11.6	3/24/75	----	12.35	4.01	----
MIDDLE	14.0/10.3/13.2/10.9/16.7/10.7	3/24/75	A	12.69	6.47	----
EDGE	6.42/3.92/7.07/5.77/13.2	3/24/75	A	7.30	9.28	99%
MIDDLE	13.4/8.82/13.3/9.38/15.6/10.2	3/24/75	B	11.83	6.82	----
EDGE	14.1/11.3/11.7/13.5/11.8/9.38	3/24/75	B	12.00	4.71	----
MIDDLE	8.68/9.17/11.1/10.6/14.2/15.2	3/24/75	C	11.54	6.54	----
EDGE	7.14/8.26/8.68/9.10/12.1/11.6	3/24/75	C	9.53	4.99	98%
BBRC PARKING :CONTROL LOT 2	15.3/9.87/12.6/12.2/12.7/14.1	3/24/75	----	12.88	5.48	----
	8.54/7.21/7.03/8.19/11.6/11.3	3/24/75	A	9.00	4.57	99%
	11.2/9.52/14.1/13.7/11.8/10.3	3/24/75	B	11.80	4.57	----
	11.8/9.94/13.6/10.8/11.9/15.0	3/24/75	C	12.20	5.06	----

NOTES: • DATA FROM HAUSER RPT. 75-168 (REF. 59)

• TESTS RUN AT -5C

• SIGNIFICANCE VALUES FROM LORD'S TEST IN DESIGNING ENGINEERING EXPERIMENTS; Lipsom et al.; Ann Arbor 1968 (REF. 66)

For the Highway 7 data, note that both formulations A and C still show a significant (from Lord's test in Reference 66) reduction in ice adhesion compared to the control at the edge of the highway. This in spite of the large amount of ground-in dirt and sand on these cores.

For BBRC lot 2, formulation A shows a significant reduction. Formulation C gave values over twice (12.20 kg/cm^2 versus 5.2 kg/cm^2) those obtained in Phase II (Chapter 4). This may be due to the combined factors of a less-than-ideal application technique for this site (see photos in 3.3 above and discussion of 7.1 of Chapter 5) and/or the demonstrated ability of formulation C to improve with time. The fact remains that all three indicated easy release in the real-life tests described in Section 4 above.

5.4 Additional Environmental Data

On 21 February 1975, soil samples were taken on the north side of the East Pearl Street location adjacent to the treated sections. This site was chosen since the coatings had been applied only one month previously and visual observation indicated rather rapid removal of the coatings. Thus, this was felt to be a worst-case situation as regards runoff soil contamination.

The environmental impact of the coatings has been summarized in 8.2 of Chapter 5. Solubility data were presented in Section 9 and ultraviolet degradation data were presented in Section 7 of Chapter 4. The data presented below are an extension and confirmation of the above.

5.4.1 Test Methods

The test methods and procedures are completely described in Reference 61 included in its entirety in Appendix A.

5.4.2 Data

A total of twelve samples was taken, four adjacent to each of the three formulation sections.

In all cases, analysis of the soil samples using Pyrolysis Gas Chromatographic Techniques failed to detect any of the formulations in the soil. As a means of using larger soil samples, Pyrolysis Infrared Spectroscopy was performed. Again, no detectable amounts of the formulations' components were found in any of the soil samples. As discussed in Section 9

of Chapter 4, these results are not surprising. The solubility of the coatings after application to the roads is so low that a full-lane width of material would yield -- at most -- 18 grams of material per meter of length if concentrated along the roadside at one time.

In a final attempt to further define the maximum solubility/pollution potential of the three formulations, films of the three formulations 0.05 cm thick were cured (37.8 C for 7 days) on Teflon sheets. One sheet of each formulation was exposed to ultraviolet radiation per ASTM D 795/1148. The exposed and unexposed films were then extracted for 24 hours at 70 C to 80 C (a very severe condition which would certainly remove all water-soluble material). The weights lost by the samples were determined and the non-volatile residues, obtained by evaporating the extracting solutions, were weighed. From Reference 61, these data are as follows in Table 6-7.

Table 6-7
HIGH TEMPERATURE WATER EXTRACTION DATA

Sample	Weight Lost Percent Of Film Weight	Residue Weight - Percent Of Film Weight
Formulation A, Unexposed	1.3	0.6
Formulation A, Exposed	9.0	6.6
Formulation B, Unexposed	4.6	0.5
Formulation B, Exposed	16.7	7.8
Formulation C, Unexposed	3.8*	6.0*
Formulation C, Exposed	5.4	1.7

*Anomalous Results - Residue Weight Cannot Exceed Weight Loss

5.4.3 Discussion of Water Extraction Data

Comparing the data in Table 6-7 with that presented in Section 9, Chapter 4, we find that the unexposed weight losses are comparable. This is true especially in view of the much

thicker (5X) films used here, which are thus harder to cure. Furthermore, except for the anomalous results of unexposed formulation C, the weight losses and residue weight differences between the formulations seem consistent with the facts that formulations A and B contain volatile water-soluble materials while formulation C cures with water.

The data for the exposed films would seem to indicate severe ultraviolet degradation. This is in conflict with the data reported in Section 7 of Chapter 4 and with the stability of the formulations exposed for 9 months on the BBRC sidewalk which are discussed in this chapter. The degradation in the present tests is difficult to understand since the dose (per Hauser Laboratories) was 2.28×10^3 joules/cm² in this exposure while the dose in Phase II testing was 1.3×10^4 joules/cm² -- a factor of nearly 6 higher.

It appears that the discrepancies in the data can be explained by the differences between the two test techniques. In the Phase II tests, great care was taken to eliminate ultraviolet generated ozone from the vicinity of the films being irradiated, whereas in the ASTM test, ozone attack of the films is possible because the ASTM cabinet is not vented.

Of more importance, the temperatures of the tested films were significantly different in the two different tests. In our Phase II work, the temperature was held to 29 ± 1 C while during the exposure detailed here the films went to a temperature of 51 C.

From the Arrhenius equation (see any physical chemistry text), it can easily be shown that for two different temperatures:

$$R_{T_1/T_2} = e^{\frac{\Delta H}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)} \quad (1)$$

Where

R = Reaction rate ratio at T_1 , and T_2 , absolute temperatures in K. ($K = C + 273.2$)

ΔH = Reaction enthalpy change, cal/mole

R = Universal gas constant, 1.987 cal/°K-mole

Thus equation (1) becomes

$$R_{51/29} = e^{1.13 \times 10^{-4} \Delta H} \quad (2)$$

If we consider that, for example, the Si-O bond is being broken, $\Delta H \approx 80,000$ and $R = 8435$. For a more normal reaction mechanism, assume $\Delta H \approx 20,000$ and $R = 9.6$. In any event, the sample temperature difference in the two exposures has a very pronounced effect and probably explains the degradation in these coating film exposures.

5.4.4 Conclusion

On the basis of soil sample testing and high-temperature water extraction, the environmental pollution effect of the three road-tested formulations appears negligible.

Even as partially degraded by high-temperature ultraviolet exposure, the *total* non-volatile water-soluble matter is still about the same as the maximum assumed in the Environmental Impact Summary given in Section 8 of Chapter 5.

6.0 PHASE IV CONCLUSIONS

The results of the Phase IV highway evaluation tests of three formulations have been presented above. Two of these show considerable promise with the primary problem areas being wear life and pollution of the atmosphere during application.

None of the three present a severe safety (skid) hazard in themselves (see also the accident reports in Appendix D).

While the Petroset AT did not demonstrate notable ice adhesion mitigation on the highways, it did show such properties in the asphalt parking lot tests.

Both the Petroset AT and formulation C indicated durability on the asphalt road surfaces and suggested protection of the asphalt as well.

All formulations tested show low environmental pollution potentials.

Applied cost of the tested (as well as those untested, see Chapter 5) formulations are near the cost of salt/sand mixtures when automotive corrosion and bridge damage factors are included.

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APPENDIX A
HOUSER LABORATORIES REPORTS
(Reference 54)

CHARACTERISTICS OF HIGHWAY PAVEMENTS & COATINGS

A Materials Study For
Ball Brothers Research Corporation
Boulder, Colorado

Report No. 5506-74-6
March 18, 1974

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CHARACTERISTICS OF HIGHWAY PAVEMENTS AND COATINGS

1. Introduction

The objective of this study was to provide a background of information relating to prospects for using adhesive materials to prevent ice adhesion to highways. This study was performed on BBRC subcontract #01460 dated February 5, 1974, directed by Mr. George H. Ahlborn.

2. Properties of Concrete Paving

Practically all concrete paving consists of Portland cement plus aggregate. The American Association of State Highway Officials Specification M85 is concerned with Portland cement, and Specification M134 is concerned with air-entrained Portland cement.

Type I	General purpose
Type II	For use in areas of moderate sulfate concentration in the soil or ground waters
Type III	For use where high early strength is required
Type IV	For use when a low heat of hydration is required (very large bulk of concrete, seldom used for paving)
Type V	For use where high sulfate concentration may exist in the soil or ground waters.

The chemical specifications for the five types of Portland cement are noted in Table 1.

During the setting reaction, Portland cement is very alkaline, a pH about 13 is common, and some alkalinity remains after cure. For this reason, organic esters may be

Table 1. Chemical Requirements of Portland Cement,
Specification M85.

	Type I	Type II	Type III	Type IV ^a	Type V ^a
Silicon dioxide (SiO ₂), min, per cent.....	21.0
Aluminum oxide (Al ₂ O ₃), max, per cent.....	6.0
Ferric oxide (Fe ₂ O ₃), max, per cent.....	6.0	6.5
Magnesium oxide (MgO), max, per cent.....	5.0	5.0	5.0	5.0	4.0
Sulfur trioxide (SO ₃), max, per cent:					
When 3CaO·Al ₂ O ₃ is 8 per cent or less.....	2.5	2.5	3.0	2.3	2.3
When 3CaO·Al ₂ O ₃ is more than 8 per cent.....	3.0	4.0
Loss on ignition, max, per cent.....	3.0	3.0	3.0	2.5	3.0
Insoluble residue, max, per cent.....	0.75	0.75	0.75	0.75	0.75
Sodium and potassium oxide (Na ₂ O + 0.658 K ₂ O), max, per cent.....	0.6	0.6	0.6	0.6	0.6
Tricalcium silicate (3CaO·SiO ₂), ^c max, per cent.....	50	35
Dicalcium silicate (2CaO·SiO ₂), ^c min, per cent.....	40
Tricalcium aluminate (3CaO·Al ₂ O ₃), ^c max, per cent.....	8	15 ^d	7	5
Tetracalcium aluminoferrite plus twice the tricalcium alumi- nate ^c (4CaO·Al ₂ O ₃ ·Fe ₂ O ₃ + 2(3CaO·Al ₂ O ₃), or solid solution (4CaO·Al ₂ O ₃ ·Fe ₂ O ₃ + 2CaO·Fe ₂ O ₃), as ap- plicable, max, per cent.....	20.0

^a See Note 1.

^b This requirement applies only when the engineer specifies "low-alki cement." Such cement should be specified only when alkali-reactive aggregates are to be used in the concrete. The maximum value of 0.6% may be reduced when the experience of the engineer indicates that such action is desirable.

^c The expressing of chemical limitations by means of calculated assumed compounds does not necessarily mean that the oxides are actually or entirely present as such compounds.

When the ratio of percentages of aluminum oxide to ferric oxide is 0.64 or more, the percentages of tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite shall be calculated from the chemical analysis as follows:

$$\text{Tricalcium silicate} = (4.071 \times \text{per cent CaO}) - (7.600 \times \text{per cent SiO}_2) - (6.718 \times \text{per cent Al}_2\text{O}_3) - (1.430 \times \text{per cent Fe}_2\text{O}_3) - (2.852 \times \text{per cent SO}_3)$$

$$\text{Dicalcium silicate} = (2.867 \times \text{per cent SiO}_2) - (0.7544 \times \text{per cent CaO})$$

$$\text{Tricalcium aluminate} = (2.650 \times \text{per cent Al}_2\text{O}_3) - (1.692 \times \text{per cent Fe}_2\text{O}_3)$$

$$\text{Tetracalcium aluminoferrite} = 3.043 \times \text{per cent Fe}_2\text{O}_3$$

When the alumina-ferric oxide ratio is less than 0.64, a calcium aluminoferrite solid solution (expressed as $\text{C}_2\text{AF} + \text{C}_2\text{F}$) is formed. Contents of this solid solution and of tricalcium silicate shall be calculated by the following formulas:

$$\text{as } (\text{C}_2\text{AF} + \text{C}_2\text{F}) = (2.100 \times \text{per cent Al}_2\text{O}_3) + (1.702 \times \text{per cent Fe}_2\text{O}_3)$$

$$\text{Tricalcium silicate} = (4.071 \times \text{per cent CaO}) - (7.600 \times \text{per cent SiO}_2) - (4.479 \times \text{per cent Al}_2\text{O}_3) - (2.859 \times \text{per cent Fe}_2\text{O}_3) - (2.852 \times \text{per cent SO}_3)$$

No tricalcium aluminate will be present in cements of this composition. Dicalcium silicate shall be calculated as previously shown.

In the calculation of C_2A , the values of Al_2O_3 and Fe_2O_3 determined to the nearest 0.01 per cent shall be used. Values for C_2A and for the sum of $\text{C}_2\text{AF} + 2\text{C}_2\text{A}$ shall be reported to the nearest 0.1 per cent. Values for other compounds shall be reported to the nearest 1 per cent.

^d When moderate sulfate resistance is required for type III cement, tricalcium aluminate may be limited to 8 per cent. When high sulfate resistance is required, the tricalcium aluminate may be limited to 5 per cent.

subject to saponification in presence of concrete. For example, oil-base paints have always had decomposition problems on concrete surfaces.

Portland cement is easily attacked by weak or strong acids. Hydrochloric acid is often a recommended treatment to prepare Portland cement concrete surfaces for bonding to other materials. The acid treatment removes any loosely bonded lime materials at the surface of the concrete. Sometimes any excess acid is neutralized with a trisodium phosphate wash, but often the great reservoir of alkalinity in the concrete is used for self-neutralization.

The physical requirements of cement per M85 Specifications are noted in Table 2.

Table 2. Physical Requirements and Strength Properties
of Portland Cement, Specification M85.

	Type I	Type II	Type III	Type IV (1)	Type V (1)
Fineness, specific surface, sq. cm. per g. (alternate methods): (2)					
Turbidimeter test:					
Average value, min.....	1,600	1,600	1,600	1,600
Min. value, any one sample.....	1,600	1,600	1,600	1,600
Air permeability test:					
Average value, min.....	2,800	2,800	2,800	2,800
Min. value any one sample.....	2,600	2,600	2,600	2,600
Soundness:					
Autoclave expansion, max., per cent.....	0.50	0.50	0.50	0.50	0.50
Time of setting (alternate methods): (3)					
Gillmore test:					
Initial set, min., not less than.....	60	60	60	60	60
Final set, hr., not more than.....	10	10	10	10	10
Vicat test (T131):					
Set, min., not less than.....	45	45	45	45	45
Air content: (5)					
Air content of mortar prepared and tested in accordance with Method T 137, max. per cent by volume.....	12.0	12.0	12.0	12.0	12.0
Tensile strength, psi.: (4)					
The average tensile strength of not less than three standard mortar briquets, prepared in accordance with Method T132, shall be equal to or higher than the values specified for the ages indicated below:					
1 day in moist air.....	275
1 day in moist air, 2 days in water.....	150	125	375
1 day in moist air, 6 days in water.....	275	250	175	250
1 day in moist air, 27 days in water.....	350	325	300	325
Compressive strength, psi.: (4)					
The average compressive strength of not less than three mor- tar cubes, prepared in accordance with Method T 106, shall be equal to or higher than the values specified for the ages indicated below:					
1 day in moist air.....	1,700
1 day in moist air, 2 days in water.....	1,200	1,000	3,000
1 day in moist air, 6 days in water.....	2,100	1,800	800	1,500
1 day in moist air, 27 days in water.....	3,500	3,500	2,000	3,000
False set, final penetration, min. percent (6)	50	50	50	50	50

- (1) See Note, Section 1.
- (2) Either of the two alternate fineness methods may be used at the option of the test laboratory. However in case of dispute or when the sample fails to meet the requirements of the Blaine meter, the Wagner turbidimeter shall be used, and the requirements in Table II for this method shall govern.
- (3) The purchaser should specify the type of setting time test required. In case he does not so specify, or in case of dispute, the requirement of the Vicat test only shall govern.
- (4) The purchaser shall specify the type of strength test desired. In case he does not so specify the requirements of the compressive strength test only shall govern. Unless otherwise specified, the strength tests for Types I and II cement will be made only at 3 and 7 days. The strength at any age shall be higher than the strength at the next preceding age.
- (5) Cement producing an air content of mortar between 12 and 16 percent may be accepted at the discretion of the purchaser when it is to be used in air-entraining concrete and the air content of this concrete is controlled at the mixer.
- (6) This requirement applies only when specifically requested.

The air-entraining Portland cement of AASHTO Specification M134 may be of three

types:

- | | |
|----------|--|
| Type IA | For general concrete construction |
| Type IIA | For use in general construction when moderate heat of hydration and/or sulphate resistance is needed |

Type IIIA For use where high early strength is required.

The chemical requirements for air-entraining cements are almost identical to those of Types I, II and III of Table I. The physical requirements are noted below in Table 3.

Table 3. Physical Requirements and Strength Properties
of Portland Cement, Specification M134

	Type IA	Type IIA	Type IIIA
Fineness, specific surface, sq. cm. per g. (alternate methods) (1):			
Turbidimeter test:			
Average value, min.:	1,600	1,600
Min. value, any one sample	1,500	1,500
Air permeability test:			
Average value, min.:	2,800	2,800
Min. value, any one sample	2,600	2,600
Soundness:			
Autoclave expansion, max., per cent	0.50	0.50	0.50
Time of setting (alternate methods) (2):			
Gillmore test:			
Initial set, min., not less than	60	60	60
Final set, hr., not more than	10	10	10
Vicat test (T 131):			
Set, min., not less than	45	45	45
Air content of mortar, prepared and tested in accordance with Method T 137, per cent by volume	19 ±3	19 ±3	19 ±3
Tensile strength, psi. (3):			
The average tensile strength of not less than three standard mortar briquettes, prepared in accordance with Method T 132, shall be equal or higher than the values specified for the ages indicated below:			
1 day in moist air	275
1 day in moist air, 2 days in water	150	125	375
1 day in moist air, 6 days in water	275	250	(3)
1 day in moist air, 27 days in water	350	325
Compressive strength, psi. (3):			
The compressive strength of mortar cubes, composed of 1 part cement and 2.75 parts graded standard sand, by weight, prepared and tested in accordance with Method T 106, shall be equal to or higher than the values specified for the ages indicated below:			
1 day in moist air	1,300
1 day in moist air, 2 days in water	900	750	2,500
1 day in moist air, 6 days in water	1,500	1,400
1 day in moist air, 27 days in water	2,800	2,800
False set, final penetration, min., per cent (4)	50	50	50

- (1) Either of the two alternate fineness methods may be used at the option of the testing laboratory. However, in case of dispute, or when the sample fails to meet the requirements of the Blaine meter, the Wagner turbidimeter shall be used, and the requirements in Table II for this method shall govern.
- (2) The purchaser should specify the type of setting time test required. In case he does not so specify, or in the case of dispute, the requirements of the Vicat test only shall govern.
- (3) The purchaser shall specify the type of strength test desired. In case he does not so specify the requirements of the compressive strength test only shall govern. Unless otherwise specified the strength tests for Types IA and IIA cement will be made only at 3 and 7 days. The strength at any age shall be higher than the strength of the next preceding age.
- (4) This requirement applies only when specifically requested.

A typical mix of Portland cement with aggregate for highway paving is noted in Table 4.

**Table 4. Typical Portland Cement Paving Mix
and Aggregate Grading**

1. Approximate Mix Proportions by Volume (Dry loose measure):

1 part cement (type IA)
2 parts sand (2 NS grading)
3-3/4 parts gravel, stone, or slab (4A & 10A or 6A)
Estimated cement content: 5.5 sacks/cu.yd.

2. Fine and Coarse Aggregates:

2 NS Gradation		Sieve Size, inch	Coarse Aggregate Gradation		
Sieve Size No.	Percent Passing		Percent Passing	4A*	10A*
3/8 in.	100	2-1/2	100	--	--
4	95-100	2	95-100	100	--
8	65-95	1-1/2	65-90	95-100	100
16	35-75	1	10-40	60-90	95-100
30	20-55	1/2	0-20	25-55	35-65
50	10-30	3/8	0-5	--	--
100	0-10	No. 4	0-8	0-8	0-8

*4A and 10A, when used in a concrete mix, are combined 50-50

The void content of an air-entraining concrete as in Table 4 is approximately 19 percent (Table 3) of the 14 percent cement volume in the cured concrete, or about 2.7 percent of the paving. If a Type I cement is used (maximum 12 percent air) the void content of the paving should not exceed 1.7 percent.

Voids in the concrete are intended to improve freeze-thaw resistance, but they also make the paving slightly permeable to water and they provide a grip for mechanical adhesion of ice on the surface.

Water has a high affinity for untreated concrete, virtually a zero contact angle. The highly polar characteristics of the concrete and the aggregate minerals are responsibility for this hydrophilic characteristic.

The friction coefficient of Portland cement paving is dependent upon vehicle speed, tire tread, and surface wetness. Figure 1 presents one of several friction coefficient studies reported in the "Proceedings of the First International Skid Prevention Conference".

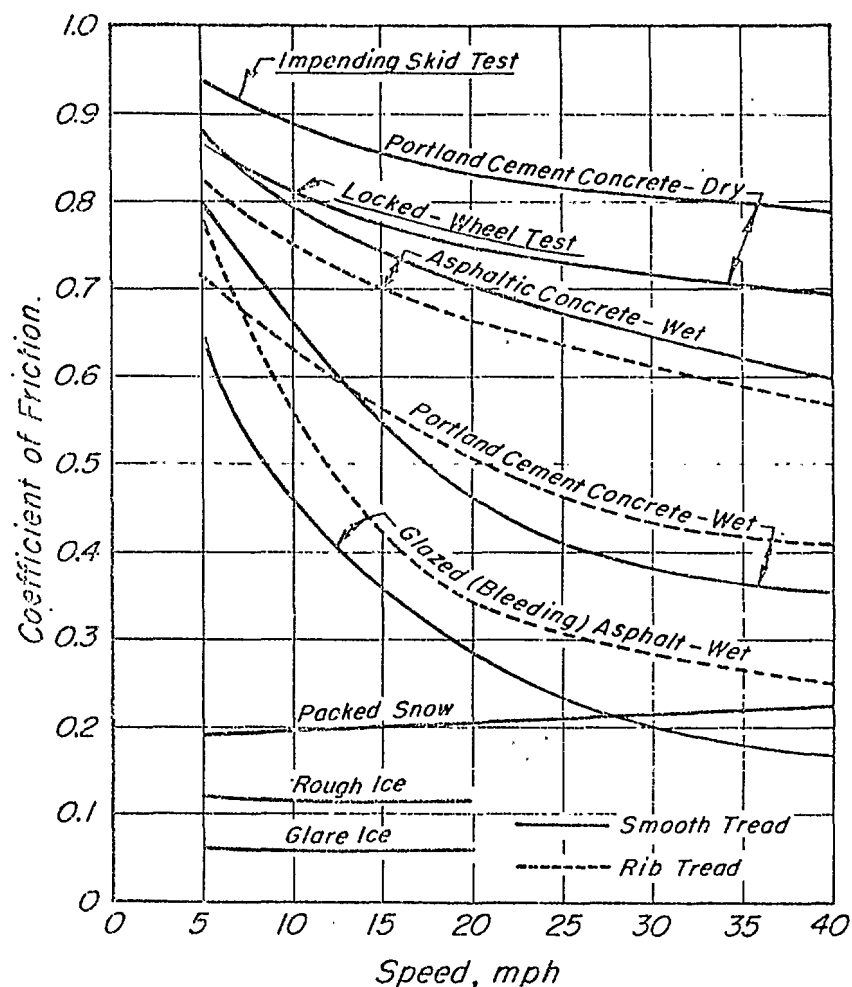


Figure 1. Coefficients of friction for rib tread and smooth tread tires on various surfaces in wet and dry conditions, ref. Moyer, "A Review of the Variables Affecting Pavement Slipperiness".

The dramatic differences between the friction coefficient of dry Portland cement paving and glare ice are major concern in this study of methods for preventing ice adhesion. Glare ice, for example, has a friction coefficient about 0.06 at 20 mph, and an impending skid on dry concrete has a friction coefficient of 0.85 at the same speed.

The surface of a concrete paving changes with time and use. Spills of lubricating oil, transfer of tire rubber and wear by traffic can significantly decrease the friction coefficient of the concrete. Figure 2 presents some friction data

for traffic lanes (well worn) and passing lanes (less traveled) for both concrete Portland cement and bituminous paving.

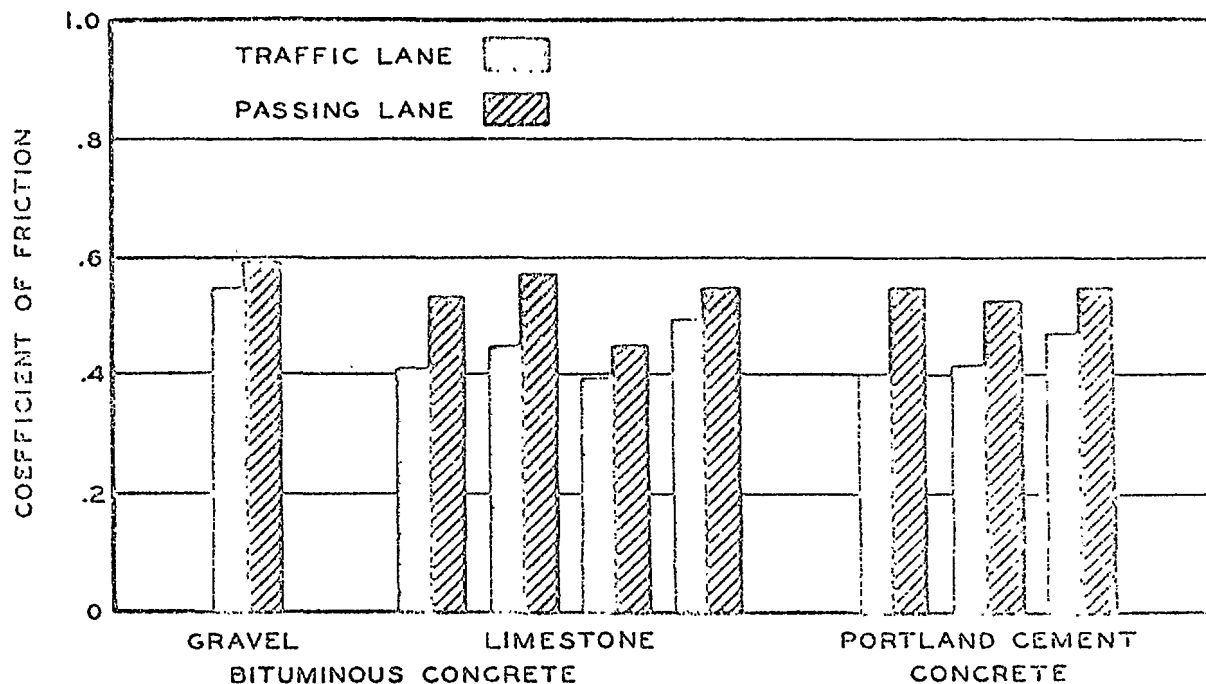


Figure 2. Effects of traffic wear and residues on the friction coefficients of bituminous and portland cement paving.

3. Properties of Bituminous Paving

Like concrete highways, bituminous paving consists of a binder phase and a particulate phase. Bituminous highways use asphaltic materials as binder (about 6 weight percent) and natural aggregates as economical and efficient filler. Many of the highway specifications and publications refer to asphalt/aggregate pavement as bituminous concrete.

Asphalts for highway paving are specified by AASHO designation M20-63 in five different grades. These petroleum derivatives are largely polycyclic compounds of variable molecular weight, hence the five different grades of hardness and ductility of the "asphalt

cement". The five grades refer to the hardness as measured by a special needle penetration test, and specifications are noted in Table 5.

Table 5. Asphalt Requirements for AASHO Specification M20.

Penetration grade	40-50		60-70		85-100		120-150		200-300	
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Penetration at 77 F., 100 g., 5 sec...	40	50	60	70	85	100	120	150	200	300
Flash point, Cleveland open cup, F.....	450	...	450	...	450	...	425	...	350	...
Ductility at 77 F., 5 cm. per min., cm.....	100	...	100	...	100	...	100
Solubility in carbon tetrachloride percent.....	99	...	99	...	99	...	99	...	99	...
Ash, percent.....	...	1.0	...	1.0	...	1.0	...	1.0	...	1.0
Thin-film oven test, $\frac{1}{8}$ in., 325 F., 5 hour
Loss on heating, percent.....8080	...	1.0	...	1.3	...	1.5
Penetration, of residue, percent of original.....	58	...	54	...	50	...	46	...	40	...
Ductility of residue at 77 F., 5 cm. per min., cm.....	50	...	75	...	100	...	100	...
Spot test (when and as specified, see Note 1) with:										
Standard naphtha solvent.....	Negative for all grades									
Naphtha-xylene solvent, _____ percent xylene.....	Negative for all grades									
Heptane-xylene solvent, _____ percent xylene.....	Negative for all grades									

NOTE 1.—The use of the spot test is optional. When it is specified, the engineer shall indicate whether the standard naphtha solvent, the naphtha-xylene solvent, or the heptane-xylene solvent will be used in determining compliance with the requirement, and also, in the case of the xylene solvents, the percentage of xylene to be used.

Aggregates may be either natural gravel or crushed rock. Frictional characteristics of the highway may be highly dependent upon both the type and the amount of aggregates used.

The asphaltic cements are generally resistant to chemical reaction at normal highway temperatures, but they are highly susceptible to softening or dissolution by solvents. Lubricating oils, automotive fuels, paint thinners and chlorinated solvents can rapidly damage the surface of the asphaltic cement if present in large quantities.

The amount of solvent used in a highway striping paint is generally insufficient to cause any degradation of the asphalt, and the solvent rapidly evaporates to leave the highway surface in its original condition. On the other hand, oil spills on the highway can cause slickness for a fairly long period of time.

Anti-ice adhesion treatments in relatively volatile solvents should cause no chemical deterioration of bituminous highway paving.

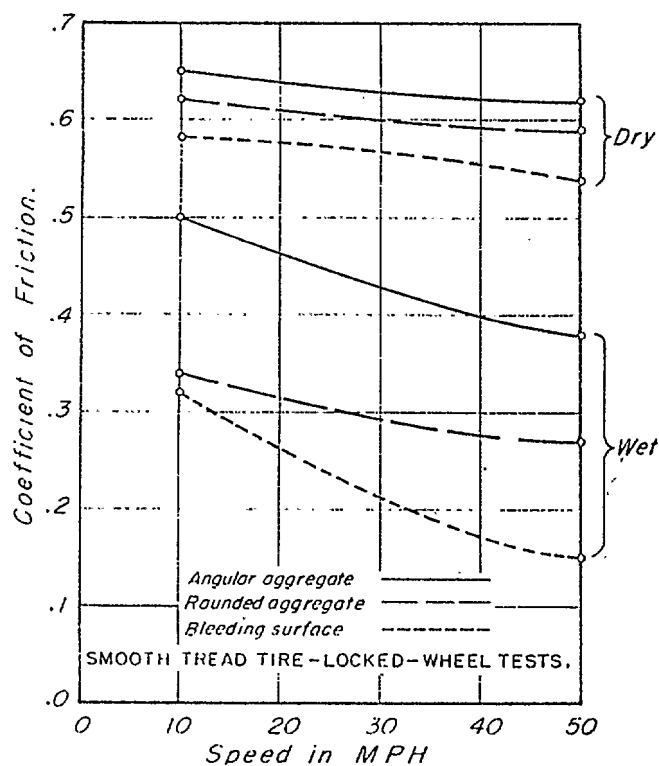
Compared to Portland cement paving, bituminous highways are rather soft. The asphaltic portion of the paving surface wears away with traffic or it may become displaced by forces from vehicle tires. New surfaces are continually exposed, and as one aggregate particle may become polished by traffic, another rough aggregate particle may become exposed to the surface.

The friction coefficient of bituminous paving is of the same magnitude as friction between Portland cement paving and automobile tires. Again, the friction is dependent upon the vehicular speed, road conditions and surface wear or glazing. Figure 1 shows that the asphaltic paving may have a friction coefficient comparable to Portland cement concrete if aggregate is properly exposed, but the friction may be less if there is excess asphalt on the surface (bleeding).

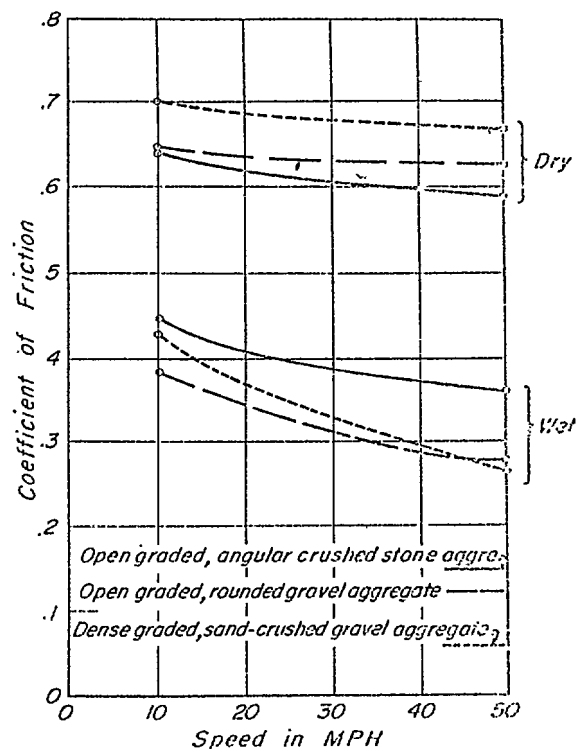
Additional friction coefficient data showing the effects of three different aggregates in bituminous paving are shown in Figure 3. The effects of aggregate polishing by traffic are demonstrated in Figure 2, where the traffic lane has about 70 percent the friction coefficient of the passing lane for both bituminous and Portland cement paving.

The surface tension of water on bituminous paving is normally high and the contact angle is near zero. The pavement surface is about 85 percent aggregate with high wettability. Slight oxidation of the organic asphalt, probably accelerated by sunlight, causes high water affinity on the remainder of the surface.

Bituminous paving is normally considered to be non-porous and relatively impermeable to water. Some publicity has been given to recent development of a permeable



A. Asphalt Seal Coat



B. Asphalt Plant Mix

Figure 3. Friction coefficients for skidding tires on bituminous pavings with different aggregates.

asphaltic paving material. In our opinion, a permeable asphalt is of real value in parking lots where water retention and absorption into the water table may help to mitigate flooding. However, a permeable asphalt could cause major subgrade problems in a highway.

An anti-ice adhesion treatment will have to stick to bituminous paving by means other than mechanical penetration and interlock.

4. Pavement Paints and Coatings

Several Federal Specifications describe the traffic paints appropriate for marking highways, but state highway departments frequently have their own specifications. Three of the Federal Specification paints are as follows:

TT-P-85	Paint, Traffic, Reflectorized for Airfield Runway Marking
TT-P-110	Paint, Traffic, Black Type I Vinyl toluene-butadiene Type II Chlorinated rubber-alkyd
TT-P-115	Paint, Traffic, Highway Type I Alkyd Type II Vinyl toluene-butadiene Type III Chlorinated rubber-alkyd

The first of these coatings may be white or yellow. The specification does not describe the chemical nature of the coating, but emphasizes performance properties. The coating is used at a rate of 105 sq. ft./gallon and the glass spheres are used at the rate of 10 pounds per gallon of paint.

Specification TT-P-110b is for marking on concrete pavement and for obliteration of white and yellow striping on bituminous pavements. It is normally used in a wet film thickness of 16 mils. Type I uses a polyvinyl toluene-butadiene copolymer, chlorinated paraffin and petroleum hydrocarbon resins in a thinner of VM & P Naphtha (boiling range 200-230°F). It dries more slowly than Type II which uses both alkyd resin and chlorinated rubber in toluene/xylene solvents. This specification describes both formulation and performance requirements of the paints.

The third traffic paint specification, TT-P-115D describes the resin type and content and the pigment content for each of the three types, in addition to performance requirements.

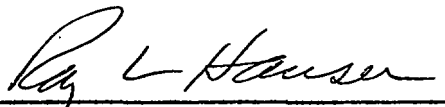
All three types of coating are intended for application in 15 mil wet film thickness.

Glass spheres may be added for reflection at the rate of six pounds per gallon of paint.

Traffic paints normally penetrate into the pores of concrete paving, or the solvent softens and impregnates molecularly a bituminous paving. When traffic paints spall off concrete paving, they often have a portion of the Portland cement concrete on the paint chip.

Olexander Hnojewy in "Highway Marking Paints" describes a method for measuring the depth of penetration of a traffic paint or primer, and the same test may be appropriate for anti-ice-adhesion materials.

In recent years, linseed oil has been touted as an impregnant for concrete to minimize water absorption and to improve freeze-thaw resistance. A linseed oil pretreatment might prevent "sponge" absorption of an expensive anti-ice compound. On the other hand, prior linseed oil treatment on the pavement might prevent the concrete from acting as a sponge and wicking medium for an inexpensive anti-ice-adhesion treatment.



Dr. Ray L. Hauser, Research Director

REFERENCES

Standard Specifications for Highway Materials, Part I, Ninth Edition (1966), American Association of State Highway Officials, AASHO, 341 National Press Building, Washington, D. C. 20004.

Proceedings, First International Skid Prevention Conference, Part I (August, 1959), Virginia Council of Highway Investigation and Research, Charlottesville, Virginia.

Olexander Hnojewy, et al, Highway Marking Paints (March, 1971), North Dakota State University, Fargo, North Dakota, PB 204 271.

(Reference 55)

June 30, 1974
Test Report No. 74-225

CLIENT:

Ball Brothers Research Corporation
P.O. Box 1062
Boulder, Colorado

Attn: Mr. George Ahlborn

P.O. #40770

MATERIALS:

Supplied by client, 15 steel plates, 1/4" thick, with coatings, identified below.

Control	No Coating
A1	Cortoretin F4
A2	Plate A2
A3	FC 321 (11)
A5	Drisil + 101
A6	Nyebar F
A7	NRL Sample
A8	Frekote 33
A9	FC 210
A10	6-31-2X
A11	92-009
A12	Drisil 73
A13	FC 210W/A43
A14	Drisil + 92-009
A15	6-31 Thin

TESTS:

Ice adhesion in shear. Two teflon rings 0.50" I.D. x 0.25" high were located on each plate, filled with water, then frozen. These specimens were allowed three to 12 hours temperature soak at approximately 10°F. Specimens were tested by attaching a 0.025" diameter, nylon jacketed, steel cable to the upper or fixed crosshead member of a tensile test machine. The cable was looped around the teflon ring where upon the specimen plate, attached to the movable crosshead member was pulled away at crosshead rate 0.50 cm/sec. (11.8 inch/minute). Load was measured by a 500 lb. Bytrex Load Cell with electronic readout to an X-Y Recorder. Tests were conducted at precisely $-12^{\circ} \pm 1^{\circ}\text{C}$. This procedure was repeated three times.

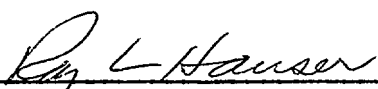
RESULTS: Coating No.	Sequence	Force (lbs.)	Shear Strength (psi)	Remarks
Control	1a	20.2	103	
	1b	15.7	79.7	
	2a	19.7	100	
	2b	22.9	116	
	3a	20.9	106	
	3b	22.9	116	
Coating A1 Disintegrated Upon Exposure to Water at Room Temperature .				
A2	1a	18.7	95.2	
	1b	19.5	99.1	
	2a	24.9	127	
	2b	19.6	99.8	
	3a	20.4	104	
	3b	13.3	67.7	
A3	1a	12.6	63.9	
	1b	11.1	56.5	
	2a	6.65	33.9	
	2b	8.05	41.0	
	3a	7.95	40.5	
	3b	7.40	37.7	
A5	1a	50 +	255 +	Part of the coating was removed from the plate during each test with nearly 100% bare metal for #3 tests .
	1b	35.7	182	
	2a	41.3	210	
	2b	68.7	350	
	3a	19.9	101	
	3b	21.2	108	

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A6	1a	11.5	58.6	
	1b	18.9	96.0	
	2a	10.8	55.0	
	2b	12.8	65.2	
	3a	11.7	59.3	
	3b	10.9	55.5	
A7	1a	20.5	104	
	1b	15.7	80.0	
	2a	6.6	33.4	
	2b	12.8	64.9	
	3a	14.5	73.6	
	3b	17.6	89.4	
A8	1a	18.6	94.7	
	1b	14.1	71.8	
	2a	9.75	49.7	
	2b	12.5	63.4	
	3a	15.0	76.1	
	3b	10.9	55.5	
A9	1a	14.0	71.3	
	1b	20.4	104	
	2a	14.3	72.8	
	2b	17.4	88.6	
	3a	11.9	60.6	
	3b	10.3	52.5	

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A10	1a	9.75	49.7	
	1b	6.90	35.1	
	2a	8.10	41.3	
	2b	7.95	40.5	
	3a	5.70	29.0	
	3b	3.60	18.3	
A11	1a	4.35	22.2	
	1b	4.30	21.9	
	2a	2.90	14.8	
	2b	3.50	17.8	
	3a	2.60	13.2	
	3b	2.80	14.3	
A12	1a	25.9	132	
	1b	20.2	103	
	2a	26.2	133	
	2b	24.0	122	
	3a	14.8	75.1	
	3b	14.5	73.8	
A13	1a	17.3	88.1	
	1b	11.4	57.8	
	2a	12.4	63.2	
	2b	7.9	40.2	
	3a	13.0	66.2	
	3b	17.5	88.9	

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A14	1a	36.4	185	
	1b	32.0	163	
	2a	21.6	110	
	2b	16.3	83.0	
	3a	21.0	107	
	3b	22.1	112	
A15	1a	15.5	78.9	
	1b	12.1	61.6	
	2a	9.80	49.9	
	2b	9.35	47.6	
	3a	10.5	53.2	
	3b	8.40	42.8	

Tests Supervised & Certified By:


 Dr. Ray L. Hauser, Research Director

(Reference 56)

September 10, 1974
Test Report No. 74-319

CLIENT: Ball Brothers Research Corporation
P.O. Box 1062
Boulder, Colorado 80302
Attention: Mr. George Ahlborn P.O. 41280

MATERIALS: Seventeen coated steel plates supplied by client.

TESTS: Ice adhesion in shear. Two teflon rings 0.50 inch I.D. x 0.25 inch high were located on each plate, filled with water, then frozen. These specimens were allowed 16 hours temperature soak at approximately 10°F. Specimens were tested by attaching a 0.025 inch diameter, nylon jacketed, steel cable to the upper or fixed crosshead member of a tensile test machine. The cable was looped around the teflon ring then the specimen plate attached to the movable crosshead member was pulled away at crosshead rate 0.50 cm/seconds (11.8 inch/minute). Load was measured by a 500 pound bytrex load cell with electronic readout to an X-Y recorder. Tests were conducted at precisely $-12^{\circ} \pm 1^{\circ}\text{C}$. This procedure was repeated until three tests had been completed at each location.

RESULTS:

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A 2	1a	2.85	14.5	
	1b	2.43	12.4	
	2a	6.85	34.9	
	2b	11.70	59.6	
	3a	3.10	15.8	
	3b	8.10	41.3	
A 3	1a	17.4	88.4	
	1b	19.3	98.0	
	2a	23.1	117	
	2b	20.5	104	
	3a	19.9	101	
	3b	21.1	107	

RESULTS:

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A 5	1a	12.9	65.7	
	1b	12.7	64.4	
	2a	21.9	112	
	2b	16.3	83.0	
	3a	17.2	87.6	
	3b	13.8	70.3	
A 6	1a	13.5	68.8	
	1b	10.2	51.9	
	2a	12.6	64.2	
	2b	22.4	114	
	3a	11.6	59.1	
	3b	23.1	117	
A 8	1a	12.4	63.2	
	1b	16.0	81.2	
	2a	11.2	56.8	
	2b	18.4	93.5	
	3a	12.7	59.1	
	3b	13.3	67.7	
A 10	1a	11.5	58.6	
	1b	9.55	48.6	
	2a	20.9	106	
	2b	11.6	58.8	
	3a	20.5	104	
	3b	19.4	98.5	

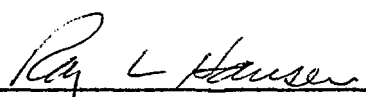
RESULTS:

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A 11	1a	20.1	102	
	1b	22.2	113	
	2a	15.4	78.4	
	2b	11.7	59.6	
	3a	22.8	116	
	3b	23.1	118	
A 13	1a	9.84	50.1	
	1b	10.5	53.5	
	2a	19.2	97.8	
	2b	25.3	129	
	3a	29.4	150	
	3b	27.4	139	
A 14	1a	13.1	66.7	
	1b	23.8	121	
	2a	18.0	91.7	
	2b	22.8	116	
	3a	25.9	132	
	3b	23.5	120	
A 18	1a	9.46	48.2	
	1b	15.0	76.4	
	2a	16.9	85.8	
	2b	16.9	86.1	
	3a	21.4	109	
	3b	18.5	94.2	

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>	<u>Remarks</u>
A 21	1a	11.6	59.1	
	1b	11.1	56.5	
	2a	20.3	103	
	2b	13.6	69.3	
	3a	22.4	114	
	3b	20.2	103	
A 23	1a	22.9	117	
	1b	24.2	123	
	2a	41.9	213	shear line through ice, not at inter-face
	2b	36.9	188	coating removed from plate with ice
	3a	9.2	46.9	coating removed from plate with ice
	3b	10.6	54.0	primarily bare steel tested
A 24	1a	16.6	84.3	
	1b	12.8	65.2	
	2a	26.8	136	
	2b	28.2	144	
	3a	25.2	128	
	3b	16.6	84.5	
A 25	1a	26.0	132	
	1b	17.3	88.1	
	2a	22.6	115	
	2b	21.9	112	
	3a	32.5	166	
	3b	33.6	171	

<u>Coating No.</u>	<u>Sequence</u>	<u>Force (lbs.)</u>	<u>Shear Strength (psi)</u>
A 26	1a	2.40	12.2
	1b	0.50	2.55
	2a	2.30	11.7
	2b	2.55	13.0
	3a	2.50	12.7
	3b	2.05	10.4
A 28	1a	19.1	97.0
	1b	25 +	127 +
	2a	31.2	159
	2b	28.3	144
	3a	30.5	155
	3b	36.4	185
A 29	1a	20.1	102
	1b	16.2	82.5
	2a	27.4	139
	2b	21.6	110
	3a	26.0	132
	3b	25.4	129

Tests Supervised and Certified By:


 Dr. Ray L. Hauser, Research Director

(Reference 57)

September 27, 1974
Test Report No. 74-343

CLIENT: Ball Brothers Research Corporation
P.O. Box 1062
Boulder, Colorado

Attention: Mr. George Ahlborn

P.O. 48046

MATERIALS: Two concrete and two asphalt pavement cores cut from U.S. 36
supplied by client.

TESTS: Ice Adhesion. Shear tests were devised to duplicate as nearly as
possible the test conditions in previous tests of adhesion to coatings
on steel plates (Test Reports No. 74-225 and 74-319).

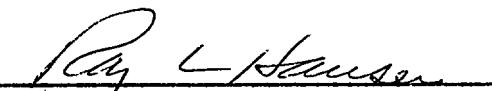
The cores were soaked in water for two hours then cooled to approximately 10°F. Two teflon rings 0.50 inch I.D. x 0.25 inch high were located on each core, filled with water, then frozen. The cores were allowed approximately 16 hours temperature soak at 10°F. Specimens were tested by attaching a 1/16 inch diameter steel cable to the upper or fixed crosshead member of a tensile test machine. The cable was looped around the teflon ring whereupon the core, attached to the movable crosshead member, was pulled away at crosshead rate 0.50 cm/sec. (11.8 inches/minute). Load was measured by a 500 lb. Bytrex load cell with electronic readout to an X-Y Recorder. Tests were conducted at $-12^{\circ} \pm 1^{\circ}\text{C}$ with triplicate tests at each location.

RESULTS:

<u>Core</u>	<u>Test #</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>
#1 concrete with greater amount of asphalt on surface	1a	34.5	176
	1b	32.0	163
	2a	40.7	207
	2b	33.4	170
	3a	41.6	212
	3b	34.4	175

<u>Core</u>	<u>Test #</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>
#2 concrete with lesser asphalt	1a	38.2	195
	1b	37.5	191
	2a	30.9	157
	2b	40.7	207
	3a	35.7	182
	3b	35.4	180
#3 pavement	1a	33.7	172
	1b	35.6	181
	2a	38.9	198
	2b	41.4	211
	3a	32.6	166
	3b	34.7	177
#4 pavement	1a	34.2	174
	1b	45.2	230
	2a	49.9	254
	2b	30.3	154
	3a	36.5	186
	3b	38.4	196

Tests Supervised & Certified By:



Dr. Ray L. Hauser, Research Director

(Reference 58)

October 15, 1974
Test Report No. 74-381

CLIENT: Ball Brothers Research Corporation
P.O. Box 1062
Boulder, Colorado 80302

Attention: Mr. George Ahlborn P.O. N6229

MATERIAL: Concrete and asphalt cores supplied by client (reference Test Report No. 74-343, September 27, 1974). The asphalt cores were cut in half, then each of the six specimens were coated by Bill Deshler of Ball Brothers.

TESTS: Ice Adhesion. The same test procedure was used as was detailed in Test Report No. 74-343.

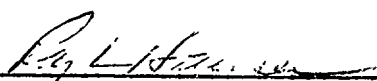
RESULTS:

<u>Core Type</u>	<u>Coating</u>	<u>Test No.</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>	
Concrete	Mod	1a	22.5	114	partial coating removal
		1b	17.5	89.1	"
		2a	14.7	74.6	"
		2b	16.4	83.5	"
		3a	36.3	185	"
		3b	20.8	106	"
Concrete	Pet	1a	26.8	136	
		1b	28.3	144	
		2a	25.4	129	
		2b	20.6	105	
		3a	41.9	213	
		3b	27.9	142	

RESULTS:

<u>Core Type</u>	<u>Coating</u>	<u>Test No.</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>	
Asphalt	Mod	1a	20.4	104	partial coating removal
		1b	26.1	133	"
		2a	26.6	135	"
		2b	29.9	152	"
		3a	30.6	156	"
		3b	26.7	136	"
Asphalt	Pet	1a	37.3	190	
		1b	24.8	126	
		2a	25.3	129	
		2b	31.4	160	
		3a	29.0	148	
		3b	29.9	152	
Asphalt	DR1	1a	13.6	69.0	
		1b	12.7	64.4	
		2a	13.2	67.2	
		2b	9.4	47.9	
		3a	17.5	89.1	
		3b	20.7	105	
Asphalt	G31	1a	28.8	146	
		1b	23.9	122	
		2a	15.7	79.7	
		2b	28.7	146	
		3a	24.0	122	
		3b	26.4	134	

Tests Supervised and Certified By:


 Dr. Ray L. Hauser, Research Director

(Reference 59)

April 8, 1975
Test Report No. 75-168

CLIENT: Ball Brothers Research Corporation
P.O. Box 1062
Boulder, Colorado 80302

Attention: Mr. George Ahlborn

P.O. No. 20282

MATERIALS: Eleven pavement cores supplied and identified by client.

TESTS: Ice Adhesion. The test procedure was identical to that described in Test Report No. 74-343, dated September 27, 1974, except that:

1. The test temperature was -5°C .
2. The cores, as received, had varying amounts of road dirt and dust on them. To equalize the tests as much as possible, the cores were given a warm water rinse of approximately two minutes per core with a kitchen sink type spray attachment.

RESULTS:

<u>Core Designation</u>	<u>Test No.</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>
H 7 Control	1a	30.3	154
	1b	32.6	166
	2a	37.8	193
	2b	32.4	165
	3a	41.4	211
	3b	32.4	165
H 7 A (Inside)	1a	39.2	200
	1b	28.8	147
	2a	37.2	189
	2b	30.4	155
	3a	47.0	239
	3b	30.1	153
H 7 B (inside)	1a	37.5	191
	1b	24.8	126
	2a	37.3	190
	2b	26.3	134
	3a	43.8	223
	3b	28.6	146

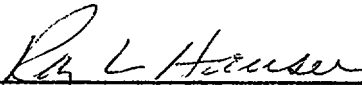
<u>Core Designation</u>	<u>Test No.</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>
H 7 C (inside)	1a	24.3	124
	1b	25.8	131
	2a	31.2	159
	2b	29.6	151
	3a	39.9	203
	3b	42.7	217
H 7 A (outside)	1a	18.0	91.7
	1b	11.0	56.0
	2a	19.8	101
	2b	16.2	82.5
	3a	37.0	188
	3b	--	--

Partial coating removal occurred during each test. When 3b test was run, we didn't get a proper force/distance recording. A repeat test was thought to be more misleading than useful.

H 7 B (outside)	1a	39.4	201
	1b	31.6	161
	2a	32.8	167
	2b	37.9	193
	3a	32.9	168
	3b	26.4	134
H 7 C (outside)	1a	20.0	102
	1b	23.1	118
	2a	24.4	124
	2b	25.5	130
	3a	34.0	173
	3b	32.5	166
BB Control	1a	43.0	219
	1b	27.6	141
	2a	35.3	180
	2b	34.4	175
	3a	35.7	182
	3b	39.7	202

<u>Core Designation</u>	<u>Test No.</u>	<u>Max Load (lbs.)</u>	<u>Shear Strength (psi)</u>
BB A	1a	23.9	122
	1b	20.2	103
	2a	19.7	100
	2b	22.9	117
	3a	32.4	165
	3b	31.7	161
BB B	1a	31.5	160
	1b	26.8	136
	2a	39.5	201
	2b	38.2	195
	3a	32.9	168
	3b	28.9	147
BB C	1a	33.2	169
	1b	27.8	142
	2a	38.1	194
	2b	30.2	154
	3a	33.4	170
	3b	42.1	214

Tests Supervised & Certified By:


 Dr. Ray L. Hauser, Research Director

(Reference 60)

September 5, 1974
Test Report No. 74-314

CLIENT: Ball Brothers Research Corporation
Aerospace Division
P.O. Box 1062
Boulder Industrial Park
Boulder, Colorado 80302
Attention: Mr. George Ahlborn P.O. #41142

MATERIAL: Eleven 3 inch x 6 inch coated coupons, E prefix #1 - #11.

TESTS: pH, solids, BOD and COD.

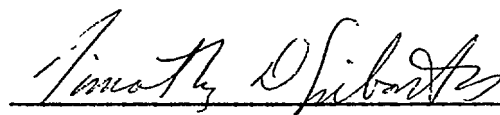
METHOD: Per US EPA.

RESULTS: Samples were soaked 48 hours in one liter distilled water per sample. Tests were performed on portions of the water solutions. Blanks were treated in a manner identical to the samples.

Sample	pH	Total Solids (grams/sample)	Residue Color	mg / liter	
				BOD 5 day @ 20°	COD
Blank	6.37	-0.0006	None	--	--
E 1	6.10	+0.0063	White	3	11
E 2	5.95	+0.0040	White	11	42
E 3	6.60	+0.0137	White	4	15
E 4	6.40	0.0103	White	8	32
E 5	6.73	0.0023	White	9	34
E 6	6.10	0.0020	White	4	16
E 7	5.90	0.0007	White	2	5
E 8	5.90	0.0023	White	3	16
E 9	6.98	0.0140	Yellow	5	15

E 10	6.90	0.0013	White	14	55
E 11	6.10	0.0183	Orange	22	84

Tests Supervised By:

A handwritten signature in cursive script, reading "Timothy D. Ziebarth", is written over a horizontal line.

Dr. Timothy D. Ziebarth, Chief Chemist

September 25, 1974

Addendum to Test Report No. 74-314 (9/5/74)

CLIENT: Ball Brothers Research Corporation
Aerospace Division
P.O. Box 1062
Boulder, Colorado 80302 P.O. # 48046

DISCUSSION OF BOD/COD VALUES

In raw sewage wastes and polluted natural waters, Biochemical Oxygen Demand (BOD) indicates the concentration of oxygen-demanding organic materials. A high BOD discharge into a natural water course can seriously result in oxygen depletion within the water course or stream--a serious threat to aquatic vegetation and fish life. Contamination of down stream water supply sources is also possible.

BOD test results are affected by a number of factors--air temperature, biological population, water movement, sun light, oxygen concentration, etc. Because of these factors, acceptable fluctuations of BOD test results may vary ± 20 percent for samples taken at the same time and same location. The BOD test is most effective when used as a long term measure of the efficiency of a sewage treatment plant by giving a daily measure of the removal of waste loading of the plant by tests on both influent and effluent. As an example, the tables on the following page lists BOD values for two sewage treatment stations and the water treatment plant within the city of Boulder for random days during January, 1974.

Simply described, the BOD test involves incubating a sealed waste water sample (or a prepared dilution) for a period of five days, and then determining the change in dissolved oxygen content. The BOD value is calculated from the results of the dissolved oxygen tests before and after the incubation period. The loss of dissolved oxygen is due to the ingestion of the oxygen by the microbic life in the sample.

TABLE 1. TYPICAL BOD VALUES, 5 day, 20° mg/liter

		Day, 1974				
		<u>Jan. 2</u>	<u>Jan. 8</u>	<u>Jan. 13</u>	<u>Jan. 19</u>	<u>Jan. 25</u>
75th Street Sewage Treatment Plant	Influent	170	195	132	339	390
	Effluent	10	35	9	22	124
Pearl Street Sewage Treatment Plant	Influent	246	168	345	210	176
	Effluent	113	79	120	112	110
Water Treatment Plant	Influent	0.5	0.3	0.5	0.4	0.4
	Effluent	0.4	0.4	0.4	0.3	0.3

TABLE 2. TYPICAL COD VALUES mg/liter

75th Street Sewage Treatment Plant	Influent	378	367	321	414	566
	Effluent	49	118		84	215
Pearl Street Sewage Treatment Plant	Influent	451	362	480	335	467
	Effluent	175	241	185	120	219
Water Treatment Plant	Influent	1.0	0.6	1.0	0.7	0.6
	Effluent	0.9	0.6	0.8	0.5	0.4

In like manner, the chemical oxygen demand (COD) test is another indicator of the oxygen demanding characteristics of waste water. This test measures the oxygen equivalent of materials present in waste water that are subject to oxidation by dichromate. Acceptable variation in laboratory results may run \pm 10 percent. Typical COD values for the city of Boulder are shown in the previous table.

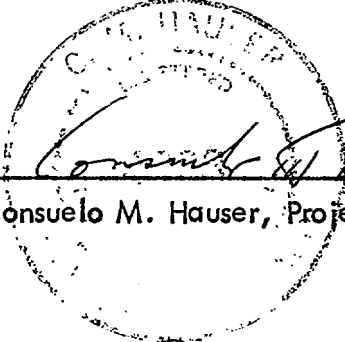
Both BOD and COD tests together are used as industry standards (and waste water treatment can certainly now be respectably described as an "industry") for evaluating the efficiency of treatment methods. While the BOD is a bacterial measure of oxygen demand and the COD is a chemical measure, no valid correlation or definitive relationship exists between them and EPA warns against making such an erroneous assumption.

Staff of the Region VIII Environmental Protection Agency in Denver were contacted to learn BOD/COD information in regard to possible contaminants in rain and melted snow run off from highway surfaces. Discussion with Mr. Dale J. Vodehnal and Mr. Robert J. Burmdefined such a possibility of pollution as a non-point source of contamination (as contrasted with a point source of contamination such as an industrial discharge of wastes into a natural water course). No BOD/COD criteria exist at present for non-point sources. For point sources, the maximum BOD loading permissible is 30 milligrams per liter and no value given for COD. Since new EPA standards and criteria are being finalized on a day-to-day basis in the Federal Register, this information is valid only for the date on this report.

In light of the above discussion and a review of the BOD/COD test values in Hauser Laboratories Report No. 74-314, the following comments can be made:

- (1) Under current EPA regulations the BOD/COD values as reported are allowable in snow-melt/rainwater run off discharges into natural water courses.

- (2) The BOD/COD values as reported indicate very little contamination compared to average sewage effluent flows (Table 1 and 2). These BOD/COD values are not exactly in line with water for domestic use processed by the Boulder water treatment plant, but they certainly are tolerable.



Mrs. Consuelo M. Hauser, Project Engineer

CLIENT

Ball Brothers Research Corporation
Aerospace Division
P.O. Box 1062
Boulder Industrial Park
Boulder, Colorado 80302

Attention: Mr. George Ahlborn

P.O. No. 20180

DESCRIPTION OF WORK

Hauser Laboratories was retained to collect and analyze soil samples for content of anti-ice surface treatment chemicals. The soil samples were collected at a BBRC test location on East Pearl in Boulder, Colorado. Analysis was a priori to be performed by pyrolysis gas chromatography, but as this method proved unsatisfactory for quantifying amount of surface treatment material present in the soil, other experiments were subsequently conducted.

Samples of three surface treatment formulations were supplied and labeled as follows:

- Sample A - Traffic Paint/Silicone Rubber
- Sample B - Phillips "Petroset AT"
- Sample C - Siliconate/Silicone Rubber

FIELD SAMPLING

Four samples of soil were taken from each of three test areas, identified as areas A, B, and C to correspond to the type of surface treatment given the particular stretch of road. Two samples, labeled one and four, were taken at a distance of about six inches from the asphalt surface, and at a depth not exceeding one inch. Two samples, labeled two and three, were taken at distances of from 18 to 24 inches from the asphalt surface, and at a depth of up to three inches. Samples one and four in each set were very dry, while samples two and three were quite wet as this area was below road level. The ground was frozen at the time of sampling at about the three inch level.

SAMPLE TREATMENT

Formulation samples A through C were dried at 105°C for twenty four hours before use as gas chromatographic reference materials. Samples A and C were converted to solids but Sample B remained a sticky oil as a result of this treatment.

The soil samples were dried twenty four hours at 105°C, then sieved to give 80 plus and 80 minus mesh fractions. The 80 plus powders were used for the pyrolysis gas chromatographic experiments.

Doped soil samples were prepared of each of the twelve field samples by adding known amounts of the appropriate coating formulation (diluted) to tared soil samples. The doped samples were then hand mixed, and subsequently tumbled for twenty four hours to provide homogeneity.

PYROLYSIS GAS CHROMATOGRAPHIC ANALYSIS

The gas chromatograph used was a Varian 920 equipped with thermal conductivity detector. Samples were pyrolyzed in the GC injection port using a CDS Pyroprobe 100 and a coil probe. Samples were generally pyrolyzed at 1000°C for five seconds in quartz tubes.

The pyrograms of the three dried formulation samples were recorded on a number of columns, including the following:

- 3' x 1/8" 10% SE30 on 60/80 Chromasorb W
- 10' x 1/8" 10% SE30 on 60/80 Chromasorb W
- 5' x 1/4" 1.5% OV101 on 100/120 Chromasorb G H/P
- 5' x 1/8" 10% Carbowax 20M on 60/80 Chromasorb W
- 5' x 1/4" Porapak Q

This assortment of conditions allowed perusal of components ranging in volatility from gases (CO₂ , CH₄ , etc.) to those of low volatility (estimated molecular weights up to 400).

There were significant differences observed in the pyrograms. The characteristic peaks were ostensibly of use in assessing levels of contamination in the soil samples.

Pyrolysis of the soil samples, including the doped references, failed to reveal any contamination by the surface treatment formulations in all samples. The pyrograms were generally not quantitatively reproducible, apparently due to the large amount of sample required and the resulting non-uniform heating in the pyroprobe itself. The large amount of pyrolyzable organic matter in the soil, as well as the variation in its composition, added to the problem.

The lower limit of detection is estimated to be a minimum of 500 ppm. Inability to consistently observe pyrolysis peaks characteristic of the polymer formulations in both soil and doped soil samples did not allow a firm lower contamination level to be set.

Experiments were also performed where the pyrolysis product compositions were recorded as a function of pyrolysis temperature. These efforts were generally unsuccessful in demonstrating a viable analytical technique for the soil.

PYROLYSIS INFRARED SPECTROSCOPY

An attempt was made to use infrared spectroscopy as a tool to evaluate contamination levels in soil samples. This method seemed a viable alternative to gas chromatography because much larger samples could be pyrolyzed. Raw formulation samples and doped soil samples were pyrolyzed in an evacuated system connected to an infrared gas cell. However, no absorptions were obtained which were characteristic of the formulation type, and which could be observed in the pyrolysis gases from the soil samples.

WATER SOLUBILITY-EXPOSURE TESTS

An alternative to the analysis of soil for contamination was the determination of the susceptibility of the coating formulations to water dissolution. The following experiments were therefore constructed.

Approximately 20 mil films (wet) of each formulation were made on teflon sheets. These films were dried at 100°F for one week. One film of each formulation was then subjected to UV irradiation for a one year outdoor exposure equivalent, and a remaining film of each was heated an additional week at 100°F. UV exposure was for one week in an ASTM D795/1148 test unit.

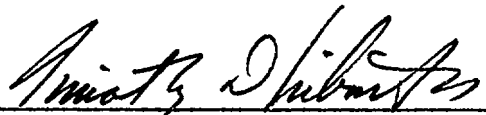
The exposed and unexposed films were then removed and subjected to soxhlet extraction for twenty four hours with distilled water. The water which was in contact with the films was 70 to 80°C. These conditions are probably more severe than those encountered by the road films in a natural environment, but should give good upper limit solubility characteristics.

Data were collected by quantifying the weight loss from each film sample, and the weight extracted by evaporating the water extracts and quantifying the residue. Results were as follows:

<u>Sample</u>	<u>As Percent of Dry Film Weight</u>	
	<u>Weight Lost</u>	<u>Residue Weight</u>
A Unexposed	1.3	0.6
A Exposed	9.0	6.6
B Unexposed	4.6	0.5
B Exposed	16.7	7.8
C Unexposed	3.8	6.0
C Exposed	5.4	1.7

The results show that there is some water solubility to all three polymer films. Second, the solubility increases markedly with UV exposure for formulations A and B, but may in fact decrease for formula C.

If further investigation of the environmental contaminating potential of these three road treatments is required, we can suggest the following program. First, water soluble components of the three films can be isolated and identified. Second, water solubility as a function of film age can be studied. Third, other conditions of exposure more closely approximating those actually encountered by the road surface can be used, such as setting up exposure-wash basins and asphalt coated samples on the roof at Hauser Laboratories where we have other equipment designed for outdoor exposure testing. With the water solubility and extracted moieties identified, a program of water (soxhlet) extractions on large soil samples could be made to assess soil contamination levels.

A handwritten signature in dark ink, appearing to read "Timothy D. Ziebarth", written over a horizontal line.

Dr. Timothy D. Ziebarth, Chief Chemist

APPENDIX B.
CONTACT REPORT
(Reference 62)
M & P

DEPT. B5501

ORGANIZATION CONTACTED: Colorado Department of Highways (C.D.H.)

ADDRESS: 4201 East Arkansas Avenue

BY: G. H. Ahlborn

Denver, Colorado

ZIP CODE 80202

REF. NO.: 680-3-0359

TYPE OF CONTACT ☒ FIELD ☐ AT BBRC ☐ PHONE TIME OF CONTACT A.M. DATE OF CONTACT 2/21/74 DATE OF REPORT 2/21/74

PHONE No.: 759-9266

CONFEREES	TITLE	PHONE EXT	MAILING LIST		
			YES	NO	ADD
B. B. Gerhardt	Research Engineer	757-9267			
Bud A. Brakey	Head Materials' Engineer	757-9011			
Betty Davey	Clerk(?) - Accident Report Summaries	757-9345			

SUMMARY OF DISCUSSION:

Bob Jackman and writer visited the Colorado Department of Highways for preliminary information on road testing and material application to roads for the EPA Contract.

A. SPECIFIC DATA/SPECIFICATIONS

- Copy of State of Colorado version of EPA Regulation No. 7. This governs allowable quantities of solvents discharged into atmosphere. Needed in planning application techniques.
- Copy of letter to C.D.H. on powdered glass source and photomicrographs of same on road surface. May be needed to reduce slickness of otherwise good coatings.
- Copy of latest traffic density map for Denver Metro area. Covers sufficient area to be used for primary site selection (for road tests).
- ASTM E303-69 governs portable road skid tester; ASTM E274-70 governs trailer skid road tester; C.D.H. has both items and we may be able to borrow the portable unit.
- C.D.H. maintains accident records and road/weather conditions for most areas in the state. Betty Davey can supply such information for selected sites as background data and during road test phase.
- We must contact the local C.D.H. maintenance foreman for special road treatment at selected site(s) during the application phase.

CONTACT REPORT (Continued)

<p>ACTION REQUIRED:</p> <ul style="list-style-type: none"> • Check weather instrument at Boulder airport and NCAR. • Check composition of Highways 119, 7 and 93 near Boulder. • Library - Get (borrow) copy of ASTM 1973 Parts 10 and 11. • Bob, please write letter of thanks to C.D.H. • C.D.H. regards 0.02-0.04 gallons per (yd)² as "normal" for coating application. (0.00009 meters to 0.00018 meters in SI units.) Our original estimates of 250-500 ft²/gallon look close to actual practice. • "Average" road macrostructure is about 0.04 to 0.06 inches (AMS) and controls high speed skid resistance. Studs as used in Colorado tend to <u>reduce</u> this value. • <u>Microstructure</u> controls low speed skid resistance. Studs increase this roughness value (no numbers cited) and would also remove coatings. Note, coatings would thus also reduce this and may create low speed skid problems (see also reference on "How Tires Wear" in writer's EPA file). • Buck Scott (825-2307) can make cast replicas of highways. We'll need this during application study. 	<p>DISTRIBUTION:</p> <p>DEPT. MGR. <u>X</u></p> <p>FILE <u> </u></p> <p>DIRECTOR <u> </u></p> <p>ADDITIONAL</p> <p><u>Jackman</u></p> <p><u>Noble</u></p> <p><u> </u></p> <p><u> </u></p>
---	---

B. GENERAL COMMENTS

C.D.H. very cooperative and helpful. Feeling seemed to be "select a site and we'll help as much as possible". Bridge icing seems to be their biggest worry. This is rather out of the spirit of our contract (opinion by writer based on EPA report R2-72-125) and I emphasized long stretches of road. General agreement that Boulder vicinity would be a good test region if sufficient past history and types of roads can be found in close proximity.

Adopted: September 13, 1973
Effective Date: November 28, 1973

REGULATION NO. 7

Regulation to Control the Emissions of
Hydrocarbon Vapors

A.

1. Sections F and G shall apply Statewide.
2. Sections B, E, H, I, and J, shall apply only to designated air pollution control areas.
3. Sections C and D shall apply only to the designated Denver-Metro air pollution control area.
4. All references to designated air pollution control areas, throughout this regulation, shall be as shown on page 1.13 of the Commission Regulation No. 1.

B. PETROLEUM PRODUCT STORAGE:

1. The storage of any type of petroleum distillate in any stationary tank, reservoir, or other container of more than 40,000 gallons (152,000 liters) shall be in a pressure tank capable of maintaining working pressures sufficient at all times to prevent vapor loss to the atmosphere. Said tank, reservoir, or other container shall be equipped with one or more of the following, properly installed, in good working order, and properly maintained:
 - (a) A pontoon-type or double deck-type floating roof, or internal floating cover, which shall rest on the surface of the liquid contents and shall be equipped with a closure seal or seals to close the space between the roof edge and tank wall. This control equipment shall be acceptable for said tanks, reservoirs, or other containers only if any type of petroleum distillate has a vapor pressure not exceeding 11 pounds per square inch absolute (568mm.Hg) under actual storage conditions. All gauging or sampling devices shall be vapor-tight, except when tank gauging or sampling is taking place; or
 - (b) A vapor recovery system, consisting of a vapor gathering system capable of collecting the hydrocarbon vapors discharged, together with a vapor disposal system capable of processing such vapors so as to prevent their emission to the atmosphere. All gauging and sampling devices shall be vapor-tight except when gauging or sampling is taking place.

(c) Other equipment of equal control efficiency, provided the design and effectiveness of such equipment as documented is submitted to and approved by the Division.

2. This Section B.1 shall also apply to the storage of crude oil within the designated Denver-Metro air pollution control area.
3. Propane or butane and similar products shall be stored in pressure tanks maintaining working pressures sufficient at all times to prevent hydrocarbon vapor loss to the atmosphere, or at refrigerated low temperature, or in low pressure storage equipped with vapor collection and compression equipment designed to prevent the loss of hydrocarbon vapor to the atmosphere.
4. The storage of any petroleum distillate in any stationary storage vessel of more than 3,500 gallons (13,300 liters) capacity shall be in a vessel equipped with a permanent submerged fill pipe or with a vapor recovery system.

C. PETROLEUM DISTILLATE LOADING INTO TANK TRUCKS, TRAILERS, AND OTHER TRANSPORT VEHICLES:

1. The loading of any type of petroleum distillate into any tank truck, trailer, or other transport vehicle shall be from a loading facility equipped with a vapor collection and disposal system or its equivalent, properly installed, in good working order, and properly maintained. Also, the loading facility shall be equipped with a loading arm with a vapor collection adaptor. Said system must also have pneumatic, hydraulic, or other equivalent mechanical means to force a vapor-tight seal between the adaptor and the hatch. A means shall be provided to prevent drainage of petroleum distillate from the loading device when it is removed from the hatch of any tank truck, trailer, or other transport vehicle, or to accomplish complete draining before the removal. When loading is effected through means other than hatches, all loading and vapor lines shall be equipped with fittings which make vapor-tight connections and which close automatically when disconnected.
2. Vapor recovery may be accomplished by one or more of the following:
 - (a) A vapor-liquid absorber system where vapor emissions do not exceed 1.5 pounds (1,000 gallons loaded at 70°F, 1 Atmosphere)
 - (b) Bottom loading (closed hatches) at terminal racks where vapor emissions do not exceed 1.5 pounds (1,000 gallons loaded at 70°F, 1 Atmosphere).
 - (c) Other equipment where vapor emissions do not exceed 1.5 pounds (1,000 gallons loaded at 70°F, 1 Atmosphere) and as approved by the Division.

3. This section C shall apply only to petroleum distillate loading facilities where 40,000 gallons or more, averaged over the work days of any month, are loaded in any one day. Facilities loading under 40,000 gallons per day, such as bulk plants, shall install telescoping top-loading equipment, or a demonstrated equivalent, to provide 97% submerged fill.
4. For the purpose of this regulation, "loading facility" means any aggregation or combination of petroleum distillate loading equipment which is (1) owned or operated by one person, and (2) located so that all the petroleum distillate loading outlets for such aggregation or combination of loading equipment as encompassed within a circle of 300 feet in diameter.

D. WATER SEPARATION FROM PETROLEUM PRODUCTS:

1. Single or multiple compartment oil and effluent water separation equipment which receives effluent water containing 200 gallons (760 liters) or more a day or more of any petroleum product or mixture of petroleum products from any equipment used for processing, refining, treating, storing, or handling of petroleum products having a Reid vapor pressure of 0.5 pound or greater, shall be equipped with one or more of the following vapor loss control devices, properly installed, in good working order, and properly maintained:
 - (a) A solid cover with all openings sealed and the liquid contents totally enclosed. All gauging and sampling devices shall be vapor-tight except when gauging or sampling is taking place.
 - (b) A pontoon-type or double deck-type floating roof, or internal floating cover, resting on the surface of the contents and equipped with a closure seal or seals to close the space between the roof edge and container wall. All gauging and sampling devices shall be vapor-tight except when gauging or sampling is taking place.
 - (c) A vapor recovery system consisting of a vapor gathering system capable of collecting the hydrocarbon vapors discharged and a vapor disposal system capable of processing such hydrocarbon vapors so as to prevent their emission to the atmosphere. All container gauging and sampling devices shall be vapor-tight, except when gauging or sampling is taking place.
 - (d) Other equipment of equal or greater efficiency, provided the design and effectiveness of such equipment as documented is submitted to and approved by the Division.
2. This section D shall also apply to oil and effluent water separators used in conjunction with the production of crude oil.

E. PUMPS AND COMPRESSORS:

1. No person may build, install, or permit the building or installation of any rotating pump or compressor handling any type of petroleum distillate unless said pump or compressor is equipped with mechanical seals or other equipment of equal efficiency. If reciprocating-type pumps and compressors are used, they shall be equipped with packing glands properly installed, in good working order, and properly maintained so no emissions occur from the drain recovery systems.
2. This section E shall also apply to pumps and compressors handling crude oil within the designated Denver-Metro air pollution control area.

F. WASTE GAS DISPOSAL:

Any waste gas stream containing hydrocarbon compounds from any polymer process emission source shall be burned at 1,300°F (704°C.) for 0.3 second or longer, in a direct-flame afterburner or an equally effective device. The emissions of hydrocarbon vapors from a vapor blowdown system or emergency relief shall be burned in smokeless flares, or equipment of equal efficiency, provided the design and effectiveness of equipment, as documented, is submitted to and approved by the Division.

G. ORGANIC SOLVENTS:

1. No person may discharge into the atmosphere more than 15 pounds of organic materials in any one day, nor more than 3 pounds thereof in any one hour, from any article, machine, equipment or other contrivances in which any organic solvent or any material containing organic solvent comes in contact with flame or is baked, heat-cured, or heat-polymerized, in the presence of oxygen, unless said discharge has been reduced by at least 85 percent. Those portions of any series of articles, machines, equipment, or other contrivances designed for processing a continuous web, strip, or wire which emit organic materials and use operations described in this subsection 1 shall be collectively subject to compliance with this subsection.
2. No person may discharge into the atmosphere more than 40 pounds of organic materials in any one day, nor more than 8 pounds in any one hour, from any article, machine, equipment, or other contrivance used under conditions other than described in section 1, for employing, or applying, any photochemically reactive solvent, as defined in subsection 10 of this section, or material containing such photochemically reactive solvent, unless said discharge has been reduced by at least 85 percent. Emissions of organic materials into the atmosphere resulting from air or heated drying of products for the first 12 hours after their removal from any article, machine, equipment, or other contrivance described in this section G shall be included in determining compliance with this section. Emissions resulting from baking, heat-curing, or heat-polymerizing as described in subsection 1 of this section shall be excluded from determination of compliance with this section. Those portions of any series of articles, machines, equipment, or other contrivances designed for processing a continuous web, strip, or wire which emit organic materials and use operations described in this subsection 2 shall be collectively subject to compliance with this subsection 2.

3. No person may, after December 31, 1974, discharge into the atmosphere more than 3,000 pounds of organic materials in any one day, nor more than 450 pounds in any one hour, from any article, machine, equipment, or other contrivance in which any non-photochemically reactive organic solvent or any material containing such solvent is employed or applied, unless said discharge has been reduced by at least 85 percent. Emissions of organic materials into the atmosphere resulting from air or heated drying of products for the first 12 hours after their removal from any article, machine, equipment, or other contrivance described in this section G shall be included in determining compliance with this subsection 3. Emissions resulting from baking, heat-curing, or heat-polymerizing as described in subsection 1 of this section shall be excluded from determination of compliance with this subsection. Those portions of any series of articles, machines, equipment, or other contrivances designed for processing a continuous web, strip, or wire which emit organic materials and use operations described in this subsection 3 shall be collectively subject to compliance with this section.
4. Emissions of organic materials to the atmosphere from the clean-up, with photochemically reactive solvent as defined in subsection 10 of this section, of any article, machine, equipment, or other contrivance described in subsections 1, 2, or 3, of this section G shall be included with the other emissions of organic materials from that article, machine, equipment, or other contrivance for determining compliance with this section G.
5. Emissions of organic materials into the atmosphere required to be controlled by subsections 1, 2, and 3 of this section G shall be reduced by:
 - (a) Incineration, provided that 90 percent or more of the carbon in the organic material being incinerated is oxidized to carbon dioxide,
 - (b) Adsorption, or
 - (c) Processing in a manner to be not less efficient than (a) or (b) above, provided said processing and equipment, as documented, is submitted to and approved by the Division.
6. A person processing organic materials pursuant to this section G shall provide, properly installed, in good working order, and properly maintained devices as specified in the authority to construct and the permit to operate, or as otherwise specified by the Division, for indicating temperatures, pressures, rates of flow, or other operating conditions necessary to determine the degree and effectiveness of air pollution control.
7. Any person using organic solvents or any materials containing organic solvents shall supply the Division, upon request and in the manner and form prescribed by it, written evidence of the chemical composition, physical properties, and amount consumed for each organic solvent used.

8. The provisions of this section G shall not apply to:

- (a) The manufacture of organic solvents, or the transport or storage of organic solvents or materials containing organic solvents.
- (b) The use of equipment for which other requirements are specified by subsections 1, 2, and 3, of this section G this regulation, or which are exempt from air pollution control requirements.
- (c) The spraying or other employment of insecticides, pesticides, or herbicides.
- (d) The employment, application, evaporation, or drying of saturated halogenated hydrocarbons, perchloroethylene, or trichloroethylene, provided the emission of organic materials is controlled to less than 40 pounds per day or 8 pounds per hour.
- (e) The use of any material, in any existing article, machine, equipment or other contrivance described in subsections 1, 2, 3, or 4, of this section G or the use of any material in any new or substantially modified article, machine, equipment, or other contrivance described in these sections, if the organic solvent or any material containing organic solvent does not come into direct contact with flame, and if the total volatile content of the material is not photochemically reactive as defined in section 10 of this regulation, ~~and it meets any one of the following conditions:~~
 - (i) The total volatile content contains not more than 20% by weight organic solvent, and the remainder consists only of water, or
 - (ii) ~~the total volatile content does not exceed 20% by weight, and a substantial portion of which~~ evaporates before reaching the first heated zone, or
 - (iii) the total volatile content does not exceed 5% by weight.

9. For the purposes of this section G, organic solvents include diluents and thinners and are defined as organic materials which are liquids at standard conditions and which are used as dissolvers, viscosity reducers or cleaning agents, except that such materials which exhibit a boiling point higher than 220°F at 0.5 millimeter mercury absolute pressure or having an equivalent vapor pressure shall not be considered to be solvents unless exposed to temperatures exceeding 220°F.

10. For the purposes of this Regulation No. 7, a photochemically reactive solvent is any solvent with an aggregate of more than 20 percent of its total weight composed of the chemical compounds classified below or which exceeds any of the following individual percentage composition limitations, referred to the total weight of solvent.

- (a) A combination of hydrocarbons, alcohols, aldehydes, esters, ethers, or ketones having an olefinic or cyclo-olefinic type of unsaturation: 5 percent;
- (b) A combination of aromatic compounds with eight or more carbon atoms to the molecule, except ethylbenzene: 8 percent;
- (c) A combination of ethylbenzene, ketones having branched hydrocarbon structures, trichloroethylene or toluene: 20 percent.

Whenever any organic solvent or any constituent of an organic solvent may be classified from its chemical structure into more than one of the above groups of organic compounds, it shall be considered as a member of the most reactive chemical group, that is, that group having the least allowable percent of the total volume of solvents.

11. For the purposes of this section G, organic materials are defined as chemical compounds of carbon excluding carbon monoxide, carbon dioxide, carbonic acid, metallic carbides, metallic carbonates, and ammonium carbonate.

For the purpose of this section G the terms "baked, heat cured, or heat polymerized" refer to coatings and other organic, solvent containing materials which:

- (a) have been heated in devices in which the air temperature exceeds 175°F (80°C), and
- (b) which have become insoluble in solvents in which they were soluble before being subjected to heat.

H. ARCHITECTURAL COATINGS:

- 1. No person may sell or offer for sale for use in containers of one quart capacity or larger, any architectural coating containing photochemically reactive solvent, as defined in subsection 10 of section G of this regulation.
- 2. No person may employ, apply, evaporate or dry any architectural coating, purchased in containers of one quart capacity or larger, containing photochemically reactive solvent, as defined in subsection 10 or section G of this regulation.

3. No person may thin or dilute any architectural coating with a photochemically reactive solvent, as defined in subsection 10 of section G of this regulation.
4. For the purposes of this section H, an architectural coating is defined as coating used for residential or commercial buildings and their appurtenances, or industrial buildings.

I. DISPOSAL AND EVAPORATION OF SOLVENTS:

No person may, during any one day, dispose of a total of 1 quart capacity or larger, any photochemically reactive solvent as defined in subsection 10 of section G of this regulation, or of any material containing 1 quart or more of any such photochemically reactive solvent by any means which will permit the evaporation of such solvent into the atmosphere.

J. DRY CLEANING SOLVENTS:

1. No person may operate a drycleaning operation unless the uncontrolled organic vapor emissions from such operation have been reduced by at least 85 percent. Drycleaning operations emitting less than 3 pounds per hour and less than 15 pounds per day of uncontrolled organic vapors are exempt from this section J.
2. Any owner or operator of a source subject to this section J shall achieve compliance with the requirements of subsection 1 of this section J by discontinuing the use of photochemically reactive solvents as defined in subsection 10 of section G of this regulation.
3. If incineration is used as a control technique, 90 percent or more of the carbon in the organic compounds being incinerated must be oxidized to carbon dioxide.

K. DEGREASING OPERATIONS:

No person may use for a degreasing operation any photochemically reactive solvent as defined in subsection 10 of section G of this regulation unless the emission of organic materials is controlled to less than 40 pounds per day or 8 pounds per hour.

L. EFFECTIVE DATE:

Except as otherwise stated in this regulation, said regulation shall become effective November 28, 1973, as to new sources of hydrocarbon vapor emissions and effective December 31, 1974 as to existing sources, except that acceptable compliance schedules and permit applications for all existing sources affected by this regulation must be received by the Division by no later than March 1, 1974.

(General References)

February 1, 1974

Mr. Bob Jackman
Ball Brothers Research Corp.
Box 1026
Boulder, Colorado 80302

Dear Mr. Jackman:

Confirming your telephone inquiry yesterday, we are pleased to send you, under separate cover, samples of the following cationic materials which may meet your requirements for application on forming hydrophobic road surfacing:

Chemical 39 Base
Chemical 39 High Conc
Chemical 39S
Ceranine HCA Granules
Ceranine PNS Granules
Cartaretin F-4
Cartaretin F-8
Cartarex FL
Viscospin B

We have learned that Viscospin B is being used at a concentration of 30% in kerosene as an additive to asphalt for the purpose in which you are interested. Unfortunately we have no data on this application.

If in your evaluations we can be of any technical assistance, please do not hesitate to contact us.

Thank you for your interest in our products.

Very truly yours,



Herman Brown
SANDOZ Colors & Chemicals

HB/db

Enclosures (8)

CONTACT REPORT

DEPT. B5501 M & P

ORGANIZATION CONTACTED: Goodyear Chemical

ADDRESS: _____ BY: G. H. Ahlborn

_____ ZIP CODE _____ REF. NO.: 680-3-0359

TYPE OF CONTACT ☐ FIELD ☐ AT BBRC ☒ PHONE TIME OF CONTACT 1140 DATE OF CONTACT 5/21/74 DATE OF REPORT 5/21/74

PHONE No.: <u>(714) 523-9770</u>					
CONFEREES	TITLE	PHONE EXT	MAILING LIST		
			YES	NO	ADD
Jack Ellis	Senior Sales Representative				

SUMMARY OF DISCUSSION:

Jack called to check on the potential of the FED. SPEC. TT-D-115D traffic paint sample I requested. I explained the EPA program and our thought of using proven traffic paints as binders for hydrophobic materials. He seems quite interested and offered any help possible.

General Impression from Goodyear and Ashland Contacts: The paint people seem quite enthusiastic about this.

ACTION REQUIRED:

None - Possible visit in July.

DISTRIBUTION:

DEPT. MGR. _____
FILE _____
DIRECTOR _____

ADDITIONAL
HCP

CONTACT REPORT

DEPT. B5501 M & P

ORGANIZATION CONTACTED: Phillips Petroleum Company, Chemical Division

ADDRESS: 1503 Phillips Building BY: G. H. Ahlhorn

Bart., Oklahoma ZIP CODE 74004 REF. NO.: 680-3-0359

TYPE OF CONTACT ☐ FIELD ☐ AT BBRC ☒ PHONE TIME OF CONTACT 1400 DATE OF CONTACT 6/17/74 DATE OF REPORT 6/17/74

PHONE No.: <u>(918) 661-5538</u>					
CONFEREES	TITLE	PHONE EXT	MAILING LIST		
			YES	NO	ADD
Jim Dykes					

SUMMARY OF DISCUSSION:

Called Phillips to check on their bridge surface coating.

The coating used in their TV AD is "Petromat"® and consists of a polyethylene film impregnated with binders and laid down when the roadway (bridge) surface is first applied. Does not appear applicable to our EPA Contract which stresses the treatment of existing road surfaces.

However, Mr. Dykes mentioned a rubber emulsion that Phillips has used on their own runway. Said emulsion -- when used with a normal asphalt seal coat -- reportedly shows great differences in ice accumulation when compared to untreated runway sections. Material is designated "Petroset AT"®.

Mr. Dykes is sending a one-gallon sample and literature.

ACTION REQUIRED:

Test material - If time permits.

DISTRIBUTION:

DEPT. MGR. _____
 FILE _____
 DIRECTOR _____
 ADDITIONAL
CEN
Poehlmann
Roller

CONTACT REPORT

DEPT. B5204 M & P

ORGANIZATION CONTACTED: Phillips Petroleum Co., Chemical Division

ADDRESS: 1503 Phillips Building BY: G. H. Ahlborn

Bart, Oklahoma ZIP CODE 74004 REF. NO.: 680-3-0359

TYPE OF CONTACT ☐ FIELD ☐ AT BBRC ☒ PHONE TIME OF CONTACT 1100 DATE OF CONTACT 10/29 DATE OF REPORT 10/29

PHONE No.: <u>(918) 661-5538</u>					
CONFEREES	TITLE	PHONE EXT	MAILING LIST		
			YES	NO	ADD
Lou Grey	Tech. Representative				

SUMMARY OF DISCUSSION:

Mr. Grey called due to our recent purchase of a drum of Petroset AT. Explained application.

Mr. Grey reported that a test at Wolfcreek, Montana (Contact: Lehman Fox (406) 442-2092) in winter of 1971-72, Petroset AT greatly reduced the adhesion of snow and prevented the formation of frost on treated asphalt roads.

Rate of application was 0.3 gal/yd^2 for this test. This is 1.35 l/m^2 or about 3/1 our planned rate of 0.4 l/m^2 .

Mr. Grey also recommended application within 8-10 hours of dilution with water. This, to avoid the "precipitation" we have observed for stored Petroset AT after dilution. Also, keep water slightly acid.

ACTION REQUIRED:

- Call Mr. Fox, maybe, for report.
- Consider uping Petroset AT application rate.

DISTRIBUTION:

DEPT. MGR. _____

FILE _____

DIRECTOR _____

ADDITIONAL _____

HCP _____



Figure C-1 Location of Boulder Test Area in Relation to Denver Metropolitan Area

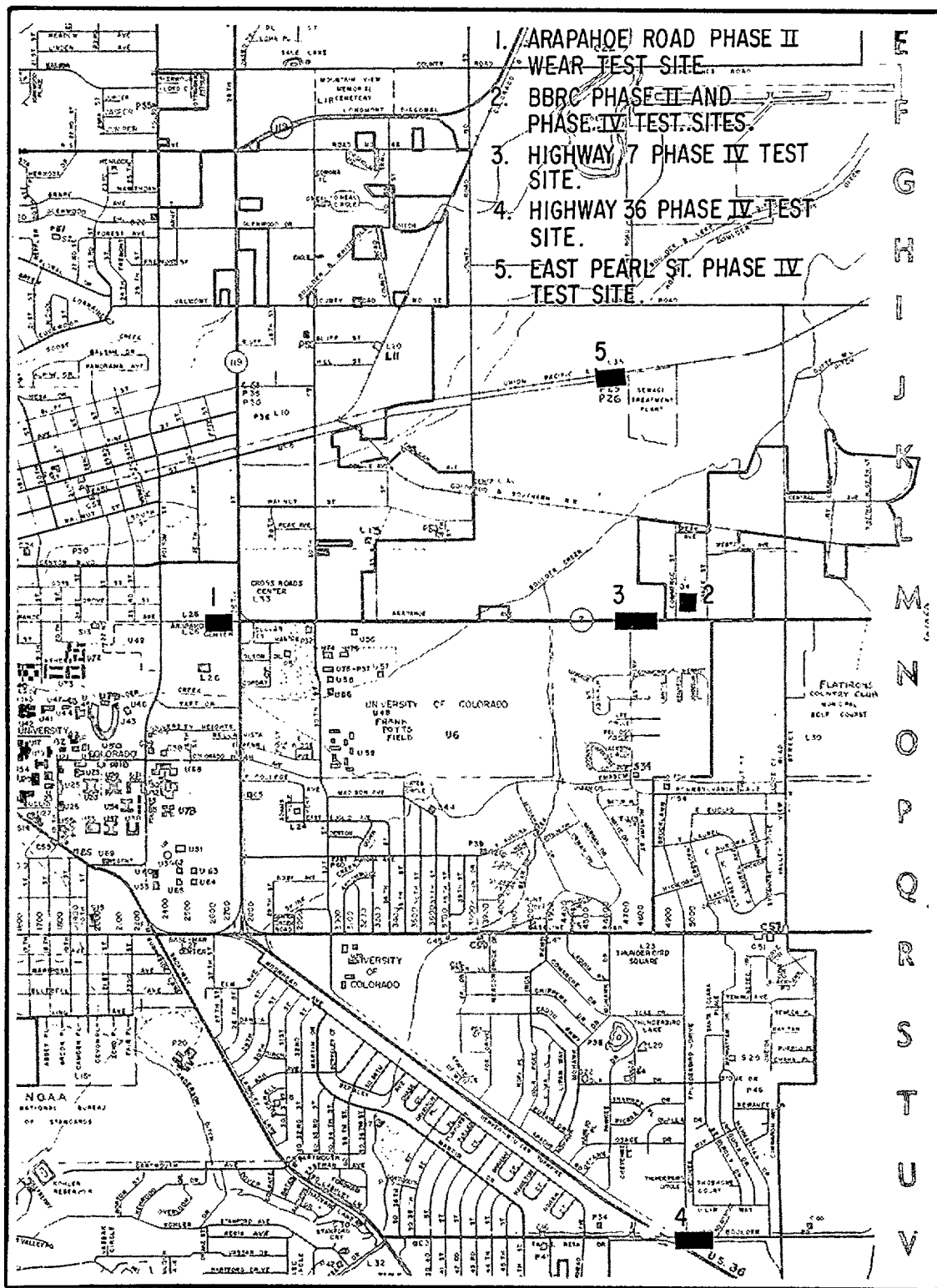


Figure C-2 Location of Boulder Area Test Sites

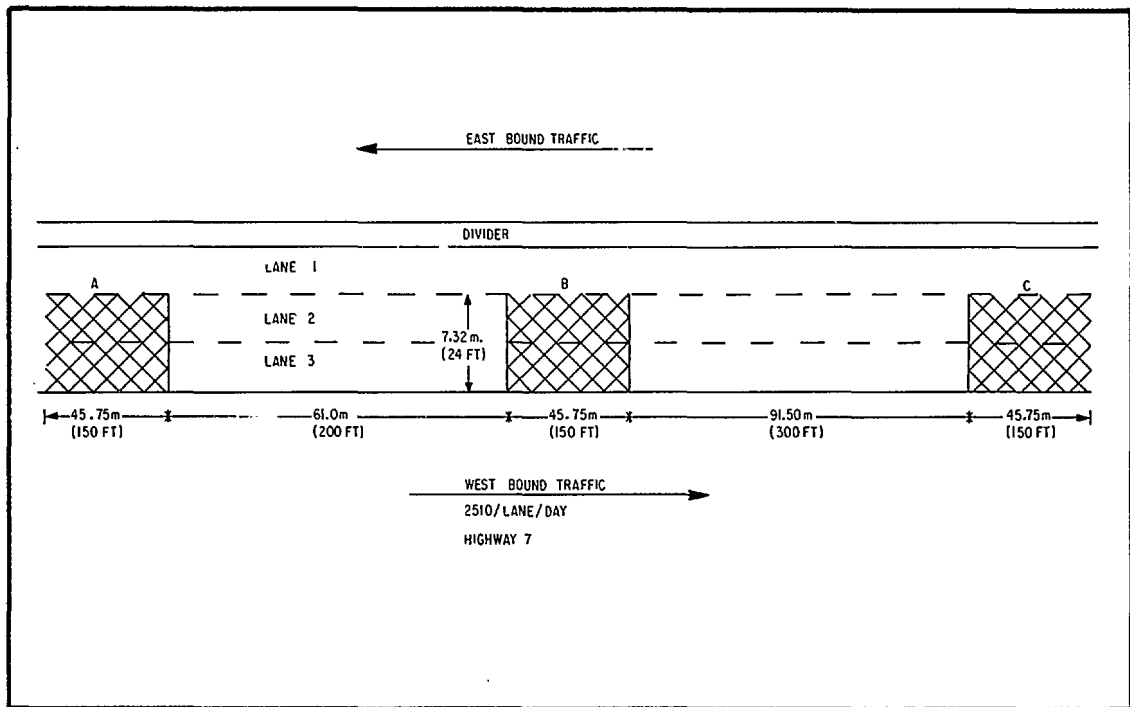


Figure C-5 Configuration of Highway 7 Test Site

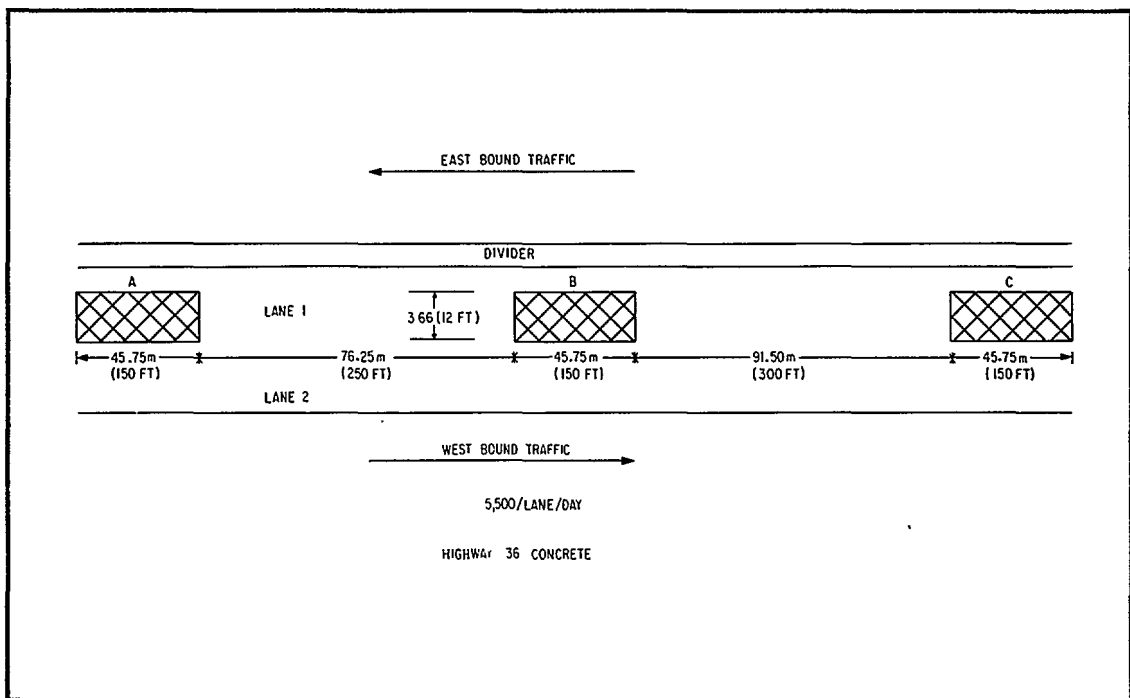


Figure C-6 Configuration of Highway 36 Concrete Test Site

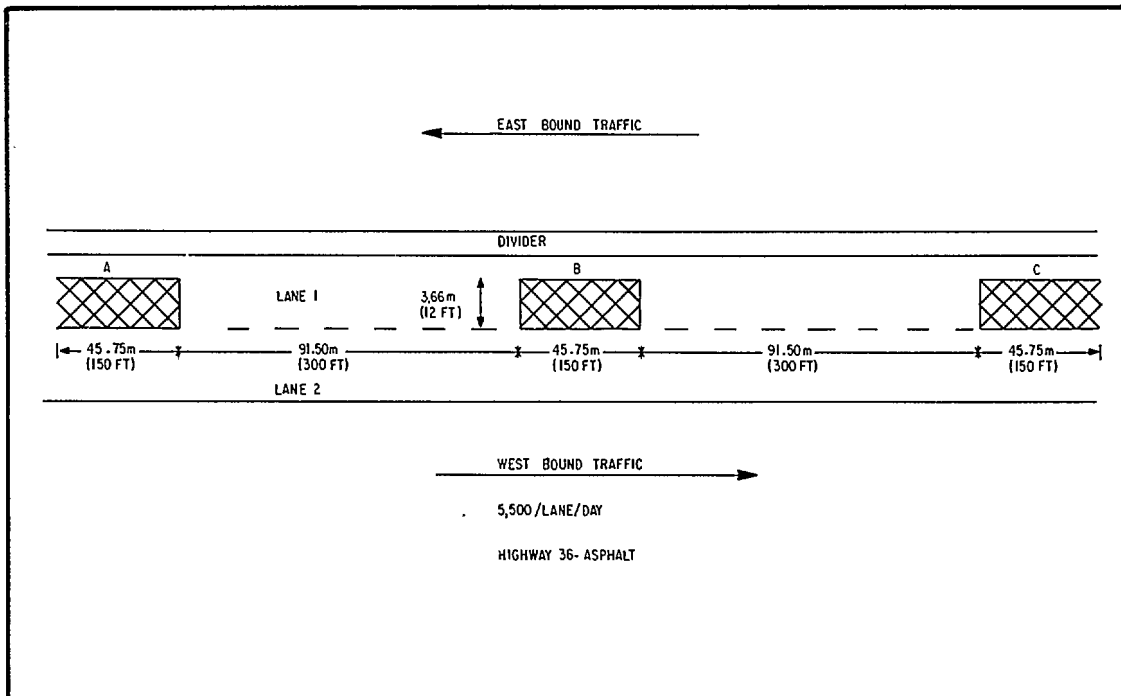


Figure C-7 Configuration of Highway 36 Asphalt Test Site

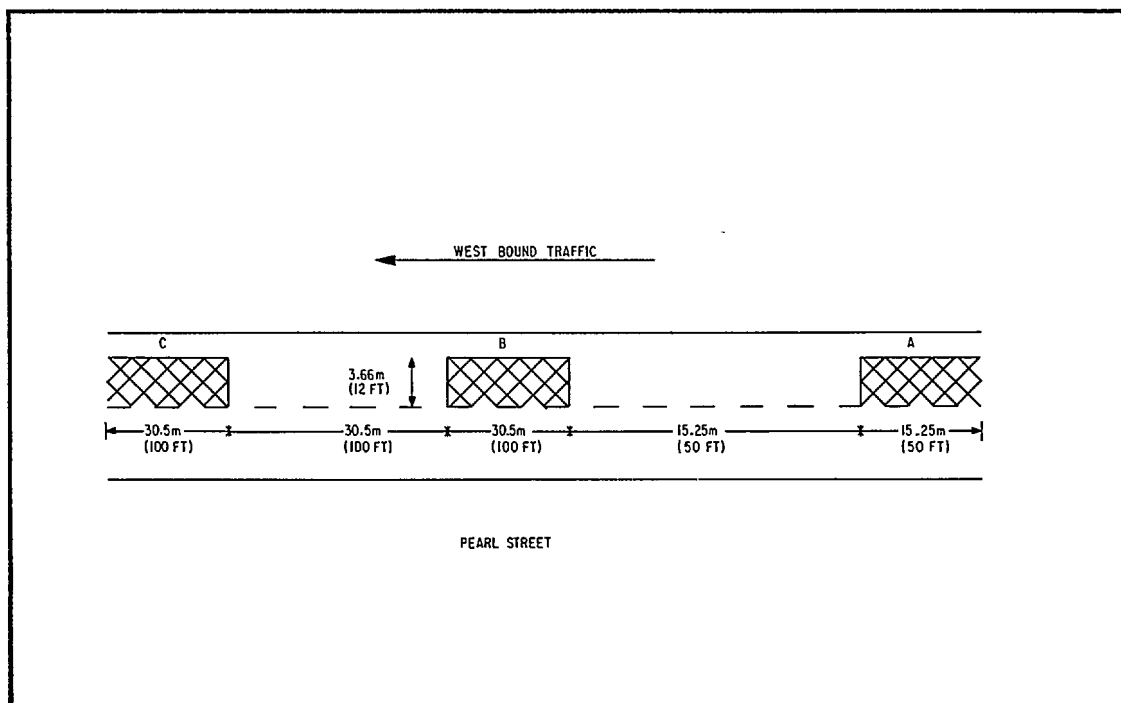


Figure C-8 Configuration of East Pearl Street Test Site

APPENDIX D (Reference 63)
COLORADO HIGHWAY DEPARTMENT ACCIDENT REPORTS

DIVISION OF HIGHWAYS
E. N. HAASE
CHIEF ENGINEER

STATE OF COLORADO



COLORADO STATE PATROL
COL. C. WAYNE KEITH,
CHIEF

4201 EAST ARKANSAS AVENUE • DENVER, COLORADO 80222 • (303) 757-9011

File Nos.
880.007.02
880.036.02
813.31.1

TRAFFIC
(Accidents)

October 4, 1974

Mr. G. H. Ahlborn
Member Technical Staff
Ball Brothers Research Corporation
P. O. Box 1062
Boulder, Colorado 80302

DOH File 16-01

Dear Mr. Ahlborn:

In response to your letter of September 19, 1974, we have checked accident experience on SH 36 from 1.3 mile west of Jct. SH 157 to the Cherryvale Road underpass and on SH 7, from 1.0 mile west to 1.0 mile east of the intersection of Commerce Street for the periods November 1, 1972 to April 1, 1973 and November 1, 1973 to April 1, 1974. Enclosed for your information and use are traffic accident summary sheets for both locations. Also enclosed is a memorandum that explains the Relative Accident Severity Index.

If we can be of additional assistance, please contact us.

Yours very truly,

E. N. HAASE
Chief Engineer

By

WM. E. TUCKER
Staff Traffic Engineer

WET:bn
Encl.

cc: D. M. Bower w/encl.
File

SUMMARY OF TRAFFIC ACCIDENT EXPERIENCE

LOCATION: SH 7, from 1.0 mi. W. to 1.0 mi. E. of the Int. of Commerce Street (2.0 miles)

PERIOD: From November 1, 1972		To April 1, 1973	
November 1, 1973		April 1, 1974	
I. <u>NUMBER OF ACCIDENTS REPORTED</u>		III. (Continued)	
One-car accidents	6	Noncollision	
Two-car accidents	21	Overtaken on road	
Three or more cars	3	Ran off road	3
Total	30	Total	30
II. <u>SEVERITY</u>		IV. <u>ESTIMATED ECONOMIC LOSS</u>	
Persons killed	0	Deaths @ \$52,000	\$ 0
Persons injured	7	Injuries @ \$ 3,100	\$ 21,700
Fatal accidents	0	Vehicle property damage	\$ 9,415
Injury accidents	6	Other property damage	\$ 260
Property damage only	24	Total	\$ 31,375
Total	30	V. <u>LIGHT</u>	
III. <u>TYPES OF ACCIDENTS</u>		Daylight	18
Collision		Dark, highway not lighted	2
Pedestrian		Dark, highway lighted	10
Head-on		VI. <u>ADVERSE CONDITIONS</u>	
Rear-end	10	Weather -- raining	
Broadside	5	-- snowing	1
Sideswipe S.D.	3	--	
Sideswipe O.D.		Road -- wet	3
Approach turn	2	-- snowy	2
Overtaking turn	4	-- icy	3
Fixed object (curb)	3	Unknown	10
Parked car		VII. <u>DRIVER</u>	
Animal		"Apparently Asleep"	
Train		"Drinking - Under the Influence"	2
		"Driving over safe speed	
		for existing road, weather,	
		and light conditions"	1

COMMENTS: During the study period, 30 accidents were reported with no fatalities and seven persons nonfatally injured. Total estimated economic loss was \$31,375.

The average Relative Accident Severity Index for these accidents is 0.21 the same as the Statewide Average. This value of RASI indicates an average accident severity potential for this location.

SUMMARY OF TRAFFIC ACCIDENT EXPERIENCE

LOCATION: US 36, from 1.3 mi. W. of Jct. SH 157, to Str. E-16-FE (Cherryvale Rd. Underpass)
(2.7 miles)

PERIOD: From November 1, 1972 To April 1, 1973
November 1, 1973 April 1, 1974

I. <u>NUMBER OF ACCIDENTS REPORTED</u>		III. (Continued)	
One-car accidents	28	Noncollision	
Two-car accidents	13	Overtaken on road	1
Three or more cars	2	Ran off road	15
		Other noncollision	2
Total	43	Total	43
II. <u>SEVERITY</u>		IV. <u>ESTIMATED ECONOMIC LOSS</u>	
Persons killed	0	Deaths @ \$52,000	\$ 0
Persons injured	18	Injuries @ \$ 3,100	\$ 55,800
Fatal accidents	0	Vehicle property damage	\$ 23,220
Injury accidents	13	Other property damage	\$ 390
Property damage only	30	Total	\$ 79,410
Total	43	V. <u>LIGHT</u>	
III. <u>TYPES OF ACCIDENTS</u>		Daylight	16
Collision		Dark, highway not lighted	27
Pedestrian		Dark, highway lighted	
Head-on		VI. <u>ADVERSE CONDITIONS</u>	
Rear-end	10	Weather -- raining	
Broadside		-- snowing	8
Sideswipe S.D.	3	--	
Sideswipe O.D.	1	Road -- wet	2
Approach turn		-- snowy	3
Overtaking turn		-- icy	*22
Fixed object (Median Barrier)	11	VII. <u>DRIVER</u>	
Parked car		"Apparently Asleep"	
Animal		"Drinking - Under the Influence"	4
Train		"Driving over safe speed for existing road, weather, and light conditions"	4

COMMENTS: During the study period, 43 accidents were reported with no fatalities and 18 persons nonfatally injured. Total estimated economic loss was \$79,410.

The average Relative Accident Severity Index for these accidents is 0.25 compared to the Statewide average of 0.22. This value of RASI indicates an above average accident severity potential for this location.

* Nine accidents were reported on 2-6-73.

File No. '813.51

DIVISION OF HIGHWAYS
STATE OF COLORADO
4201 E. Arkansas Ave.
DENVER, COLORADO 80222

TRAFFIC
(Accidents)

March 14, 1974

TO: District Engineers

FROM: M. A. Kahm DOH File 14-09

SUBJECT: Relative Accident Severity Index

Enclosed for your information is a listing by accident type of the estimated economic loss (EEL) per accident and the Relative Accident Severity Index (RASI). RASI is the quotient of the EEL for the specific accident type and the largest value for EEL determined by this study -- for TRAIN accidents, EEL = \$8391.98/accident.

RASI provides a measure of the relative severity potential of the various accident types; for example, TRAIN accidents are approximately six times as severe as collisions with a MEDIAN BARRIER, and HEAD-ON accidents are 4.5 times as severe as REAR-END collisions. In the future, RASI will be included in certain accident studies and Safety Improvement Project justifications to describe the severity potential of the various accidents which occurred. This information will supplement the usual accident data and hopefully result in an even more effective distribution of corrective action efforts.

M. A. Kahm by net
M. A. KAHM

Planning and Research Engineer

MAK:bn
Encl.

cc: Shumate-Haase-Capron-Cox	w/encl.
All District Traffic and Safety Engineers	w/encl.
M. A. Kahm	w/encl.
Angelo J. Siccardi	w/encl. (3)
Cordell Smith	w/encl.
R. F.	
File	

STATE DEPARTMENT OF HIGHWAYS
DIVISION OF HIGHWAYS - STATE OF COLORADO
PLANNING AND RESEARCH DIVISION - TRAFFIC ENGINEERING SECTION

Relative Accident Severity Index (RASI)

(based on 1972 accident data)

<u>Accident Type</u>	<u>% of All Accidents</u>	<u>*EEL/Accident</u>	<u>RASI</u>
TRAIN	0.1	\$8391.98	1.00
PEDESTRIAN	1.2	6763.06	0.81
HEAD-ON	1.8	6053.09	0.72
BRIDGE ABUTMENT	0.2	4791.87	0.57
BICYCLE	1.1	4120.01	0.49
OVERTURNED ON ROAD	0.9	3777.50	0.45
RAN OFF ROAD	13.2	3340.65	0.40
BRIDGE RAIL	0.4	2990.53	0.36
GUARDRAIL	0.6	2888.34	0.34
CURB	1.2	2627.17	0.31
OTHER	0.1	2445.96	0.29
OTHER NON-COLLISION	0.5	2368.40	0.28
APPROACH TURN	4.8	2282.49	0.27
UNDERCROSSING COLUMN	0.1	2118.61	0.25
LIGHT POLE	0.1	1897.52	0.23
BROADSIDE	17.7	1834.64	0.22
GUARD POST	0.7	1779.98	0.21
MACHINERY	0.1	1764.59	0.21
TRAFFIC SIGNAL POLE	0.2	1469.09	0.18
MEDIAN BARRIER	0.4	1410.28	0.17
SIDESWIPE-OPPOSITE DIR.	2.8	1401.22	0.17
UTILITY POLE	0.1	1393.68	0.17
REAR END	24.0	1381.93	0.16
BARRICADE	0.2	1289.47	0.15
ROCKS IN ROADWAY	0.2	1163.37	0.14
SIGN	0.2	1160.82	0.14
OVERTAKING TURN	3.1	1053.89	0.13
ANIMAL	1.9	1007.59	0.12
OTHER OBJECT	0.6	933.05	0.12
PARKED MOTOR VEHICLE	13.0	873.58	0.10
SIDESWIPE-SAME DIR.	8.4	800.92	0.10
FENCE	0.1	727.22	0.09
<hr/>			
TOTAL, STATEWIDE	100.0	1885.96	0.22

* Estimated Economic Loss per Accident

(Reference 64)

STATE DEPARTMENT OF HIGHWAYS

CHAS. E. SHUMATE

EXECUTIVE DIRECTOR

STATE OF COLORADO

DIVISION OF HIGHWAYS
E. N. HAASE
CHIEF ENGINEER



COLORADO STATE PATROL
COL. C. WAYNE KEITH.
CHIEF

4201 EAST ARKANSAS AVENUE • DENVER, COLORADO 80222 • (303) 757-9011

File Nos. 813.31.1
880.007.02
880.036.02

TRAFFIC
(Accidents)

June 6, 1975

Mr. G. H. Ahlborn
Member, Technical Staff
Ball Brothers Research Corporation
P. O. Box 1062
Boulder, Colorado 80302

DOH File 16-01

Dear Mr. Ahlborn:

In response to your telephone request June 5, 1975, we have checked accident experience on SH 36, from 1.3 miles west of Jct. SH 157 to the Cherryvale Rd. underpass and on SH 7, from 1.0 mile west to 1.0 mile east of the intersection with Commerce St. for the period November 1, 1974 to April 1, 1975. Enclosed for your information and use are accident summary sheets. This information is supplemental to that provided in our letter dated October 4, 1974.

Yours very truly,

E. N. HAASE
Chief Engineer

By *Wm E. Tucker*
WM. E. TUCKER
Staff Traffic Engineer

WET:jmw
Encls.

cc: D. M. Bower w/encls.
File

SUMMARY OF TRAFFIC ACCIDENT EXPERIENCE

LOCATION: SH 7, from 1.0 mi. W. to 1.0 mi. E. of the intersection of Commerce Street
(2.0 miles)

PERIOD: From November 1, 1974 To April 1, 1975

I. NUMBER OF ACCIDENTS REPORTED

One-car accidents	<u>1</u>
Two-car accidents	<u>8</u>
Three or more cars	<u>1</u>
Total	<u>10</u>

III. (Continued)

Noncollision	
Overtaken on road	<u>1</u>
Ran off road	<u>1</u>
Total	<u>10</u>

II. SEVERITY

Persons killed	<u>1</u>
Persons injured	<u>0</u>
Fatal accidents	<u>1</u>
Injury accidents	<u>0</u>
Property damage only	<u>9</u>
Total	<u>10</u>

IV. ESTIMATED ECONOMIC LOSS

Deaths @ \$90,000	\$ <u>90,000</u>
Injuries @ \$ 3,700	\$ <u>0</u>
Vehicle property damage *	\$ <u>5,255</u>
Other property damage	\$ <u>0</u>
Total	\$ <u>95,255</u>

III. TYPES OF ACCIDENTS

Collision	
Pedestrian	<u> </u>
Head-on	<u> </u>
Rear-end	<u> </u>
Broadside	<u>4</u>
Sideswipe S.D.	<u>3</u>
Sideswipe O.D.	<u> </u>
Approach turn	<u>2</u>
Overtaking turn	<u> </u>
Fixed object	<u> </u>
Parked car	<u> </u>
Animal	<u> </u>
Train	<u> </u>

V. LIGHT

Daylight	<u>9</u>
Dark, highway not lighted	<u>1</u>
Dark, highway lighted	<u> </u>

VI. ADVERSE CONDITIONS

Weather -- raining	<u> </u>
-- snowing	<u> </u>
--	<u> </u>
Road -- wet	<u> </u>
-- snowy	<u> </u>
-- icy	<u> </u>
unknown	<u>9</u>

VII. DRIVER

"Apparently Asleep"	<u> </u>
"Drinking - Under the Influence"	<u> </u>
"Driving over safe speed for existing road, weather, and light conditions"	<u> </u>

COMMENTS:

During the study period there were 10 reported accidents resulting in one fatality. The total estimated economic loss was \$95,255.

* Estimated at \$500 per accident if not stated on the report.

STATE DEPARTMENT OF HIGHWAYS
DIVISION OF HIGHWAYS
STATE OF COLORADO
Staff Traffic and Traffic Safety Division

File No. 880.036.02

Sheet 1 of 1
Date June 6, 1975

SUMMARY OF TRAFFIC ACCIDENT EXPERIENCE

LOCATION: US 36, from 1.3 mi. W. of Jct. SH 157 to Str. E-16-FE (Cherryvale Rd. Underpass)
(2.7 miles)

PERIOD: From November 1, 1974 To April 1, 1975

I. NUMBER OF ACCIDENTS REPORTED		III. (Continued)	
One-car accidents	10	Noncollision	
Two-car accidents	4	Overtaken on road	
Three or more cars	2	Ran off road	2
Total	16	Total	16
II. SEVERITY		IV. ESTIMATED ECONOMIC LOSS	
Persons killed	0	Deaths @ \$90,000	\$ 0
Persons injured	4	Injuries @ \$ 3,700	\$ 14,800
Fatal accidents	0	Vehicle property damage *	\$ 11,525
Injury accidents	3	Other property damage	\$ 350
Property damage only	13	Total	\$ 26,675
Total	16	V. LIGHT	
III. TYPES OF ACCIDENTS		Daylight	11
Collision		Dark, highway not lighted	2
Pedestrian		Dark, highway lighted	3
Head-on		VI. ADVERSE CONDITIONS	
Rear-end	3	Weather -- raining	
Broadside	1	-- snowing	4
Sideswipe S.D.		--	
Sideswipe O.D.		Road -- wet	
Approach turn		-- snowy	1
Overtaking turn		-- icy	6
Fixed object	9	VII. DRIVER	
Parked car	1	"Apparently Asleep"	
Animal		"Drinking - Under the Influence"	1
Train		"Driving over safe speed for existing road, weather, and light conditions"	1

COMMENTS: During the study period there were 16 reported accidents resulting in four injuries. The total estimated economic loss was \$26,675. The fixed objects struck included eight median barriers and one sign.

* Estimated at \$500 per accident if not stated on the report.

GLOSSARY

All technical concepts employed in this research report are defined in conceptual or mathematical terms where they are first used. The intent of this glossary is to introduce necessary concepts and terms in, so far as possible, non-technical language.

Adhesion: The sticking tendency of unlike substances, such as ice to coatings or coatings to surfaces.

Cohesion: The tendency of a single material to hold together, such as the cohesive strength of ice or the cohesiveness of a coating.

Biological (Biochemical) Oxygen Demand (BOD): The degree to which organic contaminants, when introduced into water, deplete the dissolved oxygen thus depriving living vegetation and animal life of this substance.

Chemical Oxygen Demand (COD): The total tendency of all materials in a water sample to react with dissolved oxygen thus reducing its availability.

Contact Angle: A measure of the "steepness" of the edge of a fluid drop with the surface on which it is resting. For example, water has a high contact angle on a good car polish.

Deicing Chemicals: As employed in this report, these are chemicals which "dissolve" ice by being very highly soluble in water and thus greatly lowering its freezing point.

Dispersion (London) Dipole Forces: Attractive (adhesive) forces acting over fairly long distances caused by molecules having magnetic fields. The effect is similar to the way a bar magnet and a piece of iron are attracted to each other.

Hydrophobic: Literally, a surface or material property meaning "hates water". As here employed, it means the rejection of water by all means including insolubility, high contact angles, resistance to water vapor and non-reactiveness with water.

Hydrophilic: A surface or material property meaning "likes water". As here used, it means having the reverse of any of the hydrophobic characteristics listed above.

Icephobic: A recently coined word describing the ability of a surface or material to (mechanically) reject ice as a solid. Thus deicing chemicals, for example, are not icephobic.

Oleophilic: Literally, likes (attracts) oil. Practically, oleophilic materials are mutually soluble in oil-like (in the present case limited to hydrocarbons) substances. Oleophilic materials are generally hydrophobic except where, as in surface active agents with both hydrocarbon (oleophilic) and water-soluble (hydrophilic) "ends", molecular orientation is controlling.

Surface Active Agents: Materials which, due to the structure of the molecule, have "ends" attracted to chemically different surfaces. They are defined by the charge of the hydrophobic (hydrocarbon or other type) portion of the molecule. Thus, soaps like sodium (+) stearate (-) are called "anionic" since the stearate is negatively charged and is attracted to a positive anode. Of specific interest here is the practical ability of surface-active agents to orient themselves on a surface leaving a single type of end protruding. This changes the wetting characteristics of the surface.

Surface Energy: As here used, a property of a surface controlled by the molecular types outermost on that surface and affecting the wetting by water and the contact angle of water on said surface. For example, tetrafluoroethylene (TFE) has CF_3 -and- CF_2 -groups outermost, has low surface energy and is not wet by water (which also exhibits a very high contact angle on TFE).

Surface Tension: The film "strength" of a fluid causing it to form small droplets. Water is high while gasoline is low. Low surface tension materials wet better and have lower contact angles than do high surface tension materials. This property is controlled by molecular type and structure.

Wetting: A complex phenomenon involving energy/tension balances, solubility effects and surface/test conditions. Good wetting is characterized by low contact angles and (usually) high adhesion.

TECHNICAL REPORT DATA <i>(Please read Instructions on the reverse before completing)</i>		
1. REPORT NO. EPA-600/2-76-242	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE DEVELOPMENT OF A HYDROPHOBIC SUBSTANCE TO MITIGATE PAVEMENT ICE ADHESION		5. REPORT DATE December 1976 (Issuing Date)
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) G.H. Ahlborn H.C. Poehlmann, Jr.		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Ball Brothers Research Corporation P.O. Box 1062 Boulder, Colorado 80302		10. PROGRAM ELEMENT NO. 1BC611
		11. CONTRACT/GRANT NO. 68-03-0359
12. SPONSORING AGENCY NAME AND ADDRESS Municipal Environmental Research Laboratory Office of Research and Development U. S. Environmental Protection Agency Cincinnati, Ohio 45268		13. TYPE OF REPORT AND PERIOD COVERED Final 1/74 to 10/75
		14. SPONSORING AGENCY CODE EPA-ORD
15. SUPPLEMENTARY NOTES PO: Hugh Masters		
16. ABSTRACT <p>This research was undertaken to investigate the feasibility of the use of hydrophobic substances on highway and bridge deck surfaces to reduce ice adhesion. Such a coating could reduce or eliminate the possibility of pollution of ground water by currently-used deicing chemicals and the multi-billion dollar yearly cost of automotive frame, bridge deck and highway surface deterioration caused by such chemicals.</p> <p>The research program herein described was conducted in four phases and, in addition to the basic technical evaluation, included consideration of all aspects of prospective coatings such as cost effectiveness, pollution potential, application techniques, effective life and detailed characterization of the formulations and chemicals employed.</p> <p>The feasibility of this approach is demonstrated with three coatings showing practical promise. Specific recommendations are presented to optimize the concepts developed in this program.</p>		
17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Deicers Ice Control Ice Breakup Water Pollution Pavements Economic Analysis	Environmental Impact Hydrophobic Materials Pavement Deicers Water/Ice Phobicity	13B
18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC	19. SECURITY CLASS (This Report) UNCLASSIFIED	21. NO. OF PAGES 218
	20. SECURITY CLASS (This page) UNCLASSIFIED	22. PRICE