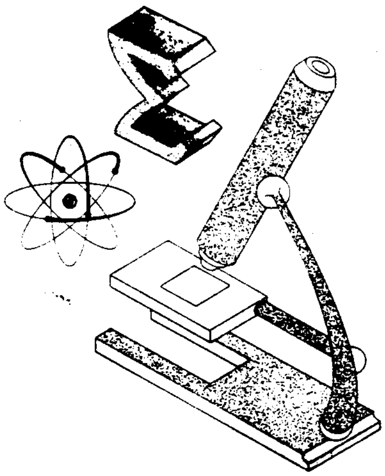
Report No. FHWA-RD-78-35

PB 283 94

STYRENE-BUTADIENE LATEX MODIFIERS FOR BRIDGE DECK OVERLAY CONCRETE



DEPARTMENT OF TRANSPORTATION • UNITED STATES OF AMERICA

April 1978

Interim Report

Prepared for

FEDERAL HIGHWAY ADMINISTRATION

Offices of Research & Development

Washington, D. C. 20590

FOREWORD

This report presents the findings of an FHWA staff research study in which five commercially-available styrene butadiene (S/B) latex emulsions for portland cement concrete were evaluated. The findings will be of interest to engineers and administrators responsible for the construction or reconstruction of bridge decks located in areas where deicing salts are used.

Each latex emulsion was identified (fingerprinted) and its chemical and physical properties were documented. The physical properties of simulated bridge deck overlay concretes, made using the latices, were also determined. A qualification program for use in defining the properties of other S/B latices and determining their effects in concrete, and a certification program to insure that each production batch of emulsion is similar to the emulsion which was prequalified, are also presented.

This report is being distributed in sufficient numbers to provide a minimum of two copies to each regional and division office and four copies to each State highway agency. Additional copies of the report for the public are available from the National Technical Information Service (NTIS), Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Charles F. Scheffey
Director, Office of Research

NOTICE

Technical Report Documentation Page

1. Report No.
FHWA-RD-78-35

2. Government Accession No.

3. Recipient's Catalog No.
PB283945

4. Title and Subtitle
Styrene-Butadiene Latex Modifiers for Bridge
Deck Overlay Concrete

5. Report Date
April 1978

6. Performing Organization Code

7. Author(s)
Kenneth C. Clear and Brian H. Chollar

8. Performing Organization Report No.

9. Performing Organization Name and Address
Federal Highway Administration
Offices of Research and Development
Materials Division, HRS-20
Washington, D.C. 20590

10. Work Unit No. (TRAIS)
FCP 24B1-562

11. Contract or Grant No.

12. Sponsoring Agency Name and Address
Department of Transportation
Federal Highway Administration
Washington, D.C. 20590

13. Type of Report and Period Covered
Interim Report
October 1976 through
March 1978

14. Sponsoring Agency Code
M/0443

15. Supplementary Notes
This staff study is a work unit in Federally Coordinated Program Project 4B, "Eliminate Premature Deterioration of Portland Cement Concrete."

16. Abstract

Styrene-butadiene (S/B) latex modified concrete overlays are being used to protect new bridge decks from rapid deicer-borne chloride intrusion and also in bridge deck rehabilitation efforts. The purposes of this research were to evaluate several commercially-available S/B latex modifiers for bridge deck overlay concrete; and to develop chemical specifications for the material, a prequalification program to permit evaluation of other S/B latex modifiers which become available, and a certification program to insure the user receives a prequalified product.

The chemical and physical properties of the latex emulsions were studied (percent solids, percent butadiene, particle size, surface tension, viscosity, etc.) and the material from each manufacturer was fingerprinted using infrared spectroscopy. The physical properties of concrete made with each material were also determined (workability, strength-compressive, flexual and bond, freeze-thaw and scaling resistance, and chloride permeability).

In general, the studies showed that the following materials exhibit similar properties and thus would be expected to perform similarly as bridge deck overlays: Modifier A - Dow Chemical Company; Deco-Rez 4776 - General Polymers Corporation; Thermoflex 8002 - Reichhold Chemicals, Inc.; and Arco Dylex 1186 - Arco Polymers, Inc.

The prequalification procedures given in the report are suggested for use in evaluating other S/B latex emulsions offered by industry. The certification procedures are suggested for use to insure that each production batch of emulsion is similar to an emulsion which was prequalified.

17. Key Words
Concrete, Corrosion, Permeability, Latex, Polymer, Emulsion, Bridge Deck, Overlay

18. Distribution Statement
No restrictions. This document is available through National Technical Information Service, Springfield, Virginia 22161.

19. Security Classif. (of this report)
Unclassified

20. Security Classif. (of this page)
Unclassified

22. Price
A07- A01

Form DOT F 1700.7 (8-72) **Reproduction of completed page authorized**

TABLE OF CONTENTS

LIST OF FIGURES

(continued)
LIST OF FIGURES

LIST OF TABLES

ACKNOWLEDGEMENTS

The authors wish to acknowledge the valuable technical assistance provided by the following latex emulsion manufacturers: Dow Chemical Company; Reichhold Chemicals, Inc.; General Polymers Corporation; and Arco Polymers, Inc. The latex manufacturers provided educational seminars and quality control test methods and procedures, and formulated special latices for testing in this research.

Additionally, the authors would like to thank the following FHWA Materials Division personnel for their aid in sample preparation and testing, data analysis, and report preparation and review: Richard E. Hay, Barry Nunemaker, Thomas J. Pasko, W. Clayton Ormsby, W. R. Jones, D. Simmons, D. Jones, W. Williams, R. W. Appleton, (retired), E. Dwyer, and H. Jackson.

STYRENE-BUTADIENE LATEX MODIFIERS FOR BRIDGE DECK OVERLAY CONCRETE

I. Introduction

One of the most severe problems facing the highway industry is chloride (deicer) induced corrosion of reinforcing steel and the subsequent deterioration of concrete bridge decks. The Federal Highway Administration's Offices of Research and Development consider elimination of bridge deck deterioration as one of their highest priority efforts. The approaches to solving the problems are defined in the Federally Coordinated Program of Research and Development in Highway Transportation (FCP) under the task title "Elimination of Spalling and Delamination of Structural Concrete." One work unit within that task is the staff research study "Alternate Styrene Butadiene Latex Modifiers for Bridge Deck Overlay Concrete."

Styrene butadiene (S/B) latex-modified mortar was first used as a thin bonded bridge deck overlay by the Dow Chemical Company in 1957. Only limited usage, mostly in rehabilitation, took place until the late 1960's. Usage of latex-modified concrete, which is similar to the mortar except that it contains coarse aggregate and less cement, increased in the early 1970's when deck evaluations and laboratory studies (2) indicated that the chloride permeability of the Dow S/B latex-modified concrete was significantly less than that of conventional bridge deck concrete. In early 1976, the Federal Highway Administration authorized the use of a 1.25-inch (32 mm) thick layer of Dow Modifier A (SM-100 was the old trade name) S/B latex-modified concrete as one of several approved alternative protective systems for new bridge decks constructed in deicing salt environments. Also, more widespread experimental use of the material as a rehabilitative overlay without removal of chloride-contaminated concrete was permitted. To date, more than 80,000 yd^3 (61,000 m^3) of latex-modified concrete have been used on over 1,000 bridge decks.

Recently, several other companies have offered S/B latex modifiers for use in portland cement concrete. This study was performed to test several of the products and to develop specifications and test methods for identifying other satisfactory modifiers.

Purpose of the Investigation

The purposes of this investigation were to:

a. Evaluate several alternative styrene butadiene (S/B) latex modifiers for bridge deck overlay concrete,

b. Develop, based upon the findings of that investigation and additional testing,

(1) A chemical specification (with test methods) for S/B latex emulsions for bridge deck concrete,

(2) A prequalification program for other S/B latex modifiers,

(3) A certification test program which, if performed on each production batch of latex emulsion, will insure the user receives a prequalified product.

Latices

Five commercially available S/B latex emulsions for concrete were studied:

1. Dow Modifier A
Dow Chemical Company
Midland, Michigan

2. Thermoflex 8002 (also called Reichhold 8002)
Reichhold Chemicals, Inc.
Dover, Delaware

3. Deco-Rez 4776
General Polymers Corp.
Cincinnati, Ohio

4. Arco Dylex 1184
Arco Polymers, Inc.
Monaca, Pennsylvania

5. Arco Dylex 1186
Arco Polymers, Inc.
Monaca, Pennsylvania

Also, in addition to the above, several specially formulated latex emulsions, with an additive missing or present in unusual amounts, were studied to evaluate the critical levels of essential ingredients needed for adequate performance and to insure that the control tests were effective.

II. Latex-Modified Concretes - Physical Properties

Concrete Mixes

All concrete mixes were prepared in the laboratory using a Lancaster pan type mixer. Total mixing time was 3.5 minutes and the ingredients were combined as follows:

1. The coarse aggregate and latex were combined and mixed 1/2 minute;
2. The sand and cement were added and mixed for 1 additional minute; and
3. The water was added and mixed for 2 minutes.

Slump was measured immediately after completion of mixing and 4 minutes after completion of mixing.

Type I portland cement was used in all mixes at a rate of 7 bags/yd^3 (658 lb/yd^3 = 390 kg/m^3). All of the latex emulsions were adjusted with distilled water to a solids content of 46 percent. The design emulsion content of all mixes was 24.5 gal/yd^3 (121 l/m^3) of concrete, which translates to approximately 15 percent latex solids by weight of cement. The water/cement (w/c) ratios of the various latex concretes are given in the section entitled Fresh Concrete Tests. Conventional concretes used for comparison purposes had water/cement ratios of 0.40 and 0.50 by weight, air contents of 5 to 7 percent, and cement factors of 7 bags/yd^3 (658 lb/yd^3 = 390 kg/m^3).

The coarse aggregate was a very angular dolomitic limestone (S.G. = 2.77), 1/2 inch (13 mm) maximum size and graded to follow the approximate midpoint of AASHTO M43 size number 7. An angular aggregate was used to insure that the water/cement ratio required for a 4- to 6-inch (102- to 152-mm) slump would be near the maximum allowed by most specifications (0.40 by weight, including the water in the latex emulsion). The fine aggregate was a natural sand from White Marsh, Maryland, with a fineness modulus of 2.71. The fine to coarse aggregate ratio was 60:40 by absolute volume of total aggregate (59:41 by weight) in all instances except for the initial permeability and flexual strength studies for which the ratio was 55:45 by volume.

Curing of Concrete

Styrene butadiene latex-modified concrete is normally cured quite differently than conventional portland cement concrete. The latex concrete requires an air drying period after the initial 24-hour moist cure to allow the polymer particles to unite and bond, whereas, a continuous moist cure is beneficial to a conventional concrete. The lack of the need for the extended moist cure is normally advantageous in field use of latex-modified concrete because it allows a bridge deck to be opened to traffic at an earlier date. However, different curing procedures do present problems when two materials such as latex concrete and conventional concrete are compared. For example, if the latex concrete is air dried prior to freeze-thaw testing while a conventional concrete is moist cured, the moisture contents of the two materials will be different when testing is initiated. Because of this, various curing procedures were used throughout this investigation, and the curing procedure for each test is listed in the section describing that test and findings. The phases used to describe the curing conditions are:

1. One day wet burlap - a 24-hour cure in specimen molds covered with wet burlap,
2. Lab air - storage at 23° ± 1.1°C and 50 ± 4 percent Rh in a drying room of the type specified in ASTM C157-75, "Length Change of Hardened Cement, Mortar, and Concrete,"
3. Moist cure - storage at 23° ± 1.7°C and 95 to 100 percent Rh as specified in ASTM C511-75, "Moisture Cabinets and Rooms Used in the Testing of Hydraulic Cements and Concretes," and
4. Saturated limewater - storage completely submerged in 73° ± 3°F (23° ± 2°C) water which contains 2 g/l or more lime ($Ca(OH)_2$) in solution.

A typical curing procedure is listed as 1 day wet burlap, 27 days lab air, and 14 days saturated limewater which means the concrete was cured 24 hours in the molds, then in lab air from 24 hours through 28 days, followed by submersion in saturated limewater from the 29th day through the 42nd day of age. The saturated limewater storage was used to allow determination of the water susceptibility of the latex-modified concrete, or to increase the free water content of the latex-modified concrete, prior to freeze-thaw testing.

Fresh Concrete Tests

The initial testing of each concrete involved the determination of the slump (4 minutes after mixing) at a total water/cement ratio of 0.40 (see Table 1). This was followed by a series of tests in which the water/cement ratio required for a 4- to 6-inch (102- to 152-mm) slump, which is the normal placement range, was determined and used in all subsequent mixes made with each emulsion. The results of these tests and the ranges of slumps encountered at each set water/cement ratio are also summarized in Table 1. It should be noted that the water/cement ratios of the Arco materials had to be adjusted to meet the slump requirement. The relatively high air content in the Arco Dylex 1186 concrete is probably the cause of the lower water content needed for a slump over 4 inches.

Table 1. Water/cement ratios and slumps of various concretes.

Concrete	Slump at w/c = 0.40 (inches)	Water/Cement Ratio Used in Tests	Slump Range Encountered (inches)
Conventional	0.1	--	--
Dow Modifier A	4.2	0.40	4.0 to 4.2
Deco Rez 4776	4.0	0.40	4.0 to 4.7
Thermoflex 8002	5.5	0.40	5.5 to 6.0
Arco Dylex 1184	0.5	0.47	4.0 to 5.5
Arco Dylex 1186	7.0	0.36	5.5 to 6.0

To convert inches to mm, multiply by 25.4

As a measure of field placeability of each latex concrete, rate of slump loss versus time was determined. Each 1.5-ft^3 (0.0425-m^3) batch of concrete was mixed in the manner described in the section entitled Concrete Mixes and then discharged into a 30-inch (760-mm) diameter container. Slump was then determined, without remixing, at various intervals up to 2 hours or until the slump was 1 inch (25 mm). The slump 4 minutes after completion of mixing was assigned a value of 100 percent and the concrete remained in the container exposed to laboratory air, 73° ± 3°F (23° ± 2°C) and 50 ± 4 percent Rh, throughout the testing.

Figure 1 presents the slump versus time curves for each of the five latex-modified concretes, a 0.50 water/cement ratio conventional concrete, and, for comparison, a concrete containing a specially formulated latex emulsion which coagulates very rapidly (due to the lack of certain additives). Table 2 lists the time required for a 50-percent slump loss for each concrete. The data show that at 73°F (23°C), 50-percent Rh, the slump loss versus time characteristics of the following concretes are similar: conventional concrete, Dow Modifier A, Thermoflex 8002, Deco-Rez 4776 and Arco Dylex 1186. The Arco Dylex 1184 latex concrete, on the other hand, required only about half as much time for 50-percent slump loss as the other concretes, except the special latex which was formulated to exhibit almost instantaneous slump loss.

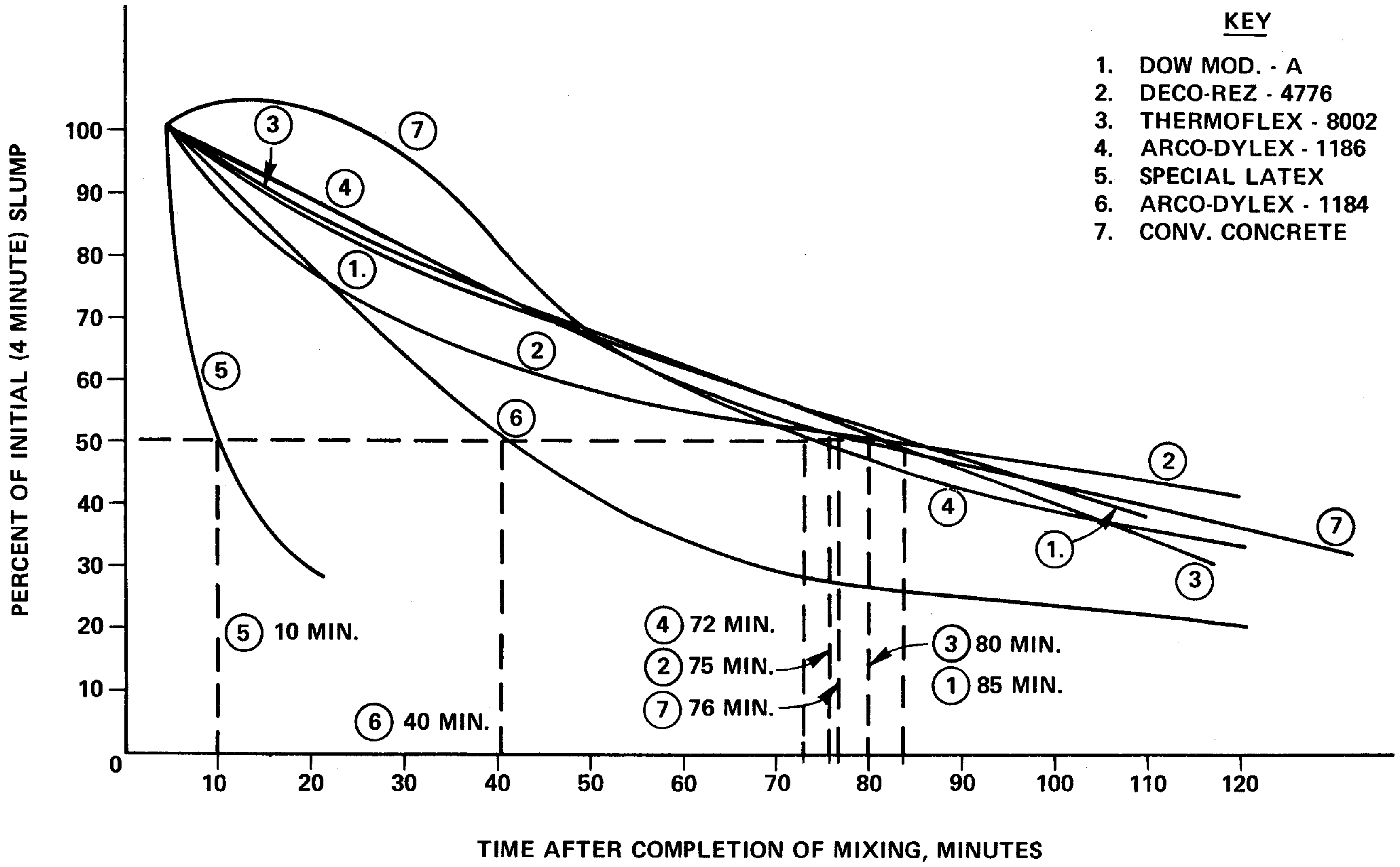


FIGURE 1. SLUMP LOSS VERSUS TIME

Table 2. Time to 50 percent slump loss for various concretes.

Concrete	Time from Completion of Mixing to 50% Slump Loss (minutes)
Conventional Concrete, w/c = 0.50	76
Dow Modifier A	83
Thermoflex 8002	80
Deco-Rez 4776	75
Arco Dylex 1184	40
Arco Dylex 1186	72
Special Latex	10

Because of the high water demand of the Arco Dylex 1184 concrete and its high rate of slump loss, it was deleted from the physical test program.

The air contents of the remaining four latex concretes measured during the test program (no air-entraining agents used) are summarized below:

1. Dow Modifier A - 4.0 to 4.5 percent
2. Deco-Rez 4776 - 4.3 to 4.4 percent
3. Thermoflex 8002 - 8.2 to 9.0 percent
4. Arco Dylex 1186 - 7.0 to 8.0 percent

A large difference was found in air contents. The Thermoflex 8002 and Arco Dylex 1186 latex-modified concretes had approximately double the air content of the concrete made with the Dow Modifier A or Deco-Rez 4776 latex emulsions. Even though present latex-modified concrete specifications normally require air contents of 6 percent or less, high air alone was not believed to be sufficient cause for rejection of an emulsion unless it caused a degradation of the physical properties of the hardened concrete.

Compressive Strength

The compressive strengths of the various concretes were determined using 3- by 6-inch (76- by 152-mm) cylinders in accordance with ASTM C39-72. The latex-modified concretes were cured 1 day in the molds under wet burlap, 27 days in lab air, and 14 days in saturated limewater. The conventional concretes were cured 1 day wet burlap, 27 days in the moist room, and 14 days in saturated limewater. Testing was performed at 1, 7, 28, and 42 days of age.

Figure 2 and Table 3 present the compressive strength test results. In general, the S/B latex-modified concretes exhibited a lower compressive strength than the conventional concrete of equal water/cement ratio and cement content. However, this may be misleading for actual construction since a continuous moist cure rarely, if ever, occurs on bridge decks and the equal water/cement ratio conventional concrete had a lower than usual water/cement ratio and virtually no slump.

All the concretes exhibited average 28-day compressive strengths greater than 4,000 psi (27.6 MPa). At 42 days of age, all the concretes except the Thermoflex 8002 modified concrete had average compressive strengths greater than 4,000 psi (27.6 MPa). The average 42-day compressive strengths for the four latex-modified concretes ranged from 3,780 to 4,560 psi (26.1 to 31.4 MPa) and were lower than the average 28-day compressive strengths in all instances. The variation in 42-day compressive strength between the various S/B concretes can probably be attributed to the 4- to 5-percent difference in air contents between the various materials.

The major difference between the latex-modified and conventional concretes with respect to compressive strength occurred during limewater soaking from 28 to 42 days of age. The strength of the conventional concrete increased slightly (+8.8 percent) while the strengths of all the latex concretes decreased. The decrease averaged approximately 9 percent for the Dow Modifier A, Deco-Rez 4776, and Thermoflex 8002 concretes and 16 percent for the Arco Dylex 1186 concrete. This finding indicates that all the latex concretes are slightly water susceptible. The Arco Dylex 1186 concrete appears to exhibit the highest strength loss upon soaking. However, it also exhibited the highest strength prior to soaking; an unexpected finding since the air content of the Arco Dylex 1186 concrete was about twice (8 versus 4 percent) that of the Dow Modifier A and Deco-Rez 4776 concretes. The lower water/cement ratio used in the Arco 1186 concrete (0.36 versus 0.40 for the other concretes) was probably a contributing factor. Also, as discussed in the section on chemical testing, the butadiene content of the Arco Dylex 1186 latex is slightly less than that of the other laticies. This is probably the cause of the higher dry strength and perhaps the greater decrease upon soaking, although the latter may also be due to the stabilization system in the emulsion or the degree of polymer cross linking (7).

Another item of interest, because of the possible water susceptibility of latex, is the effect of specimen size on

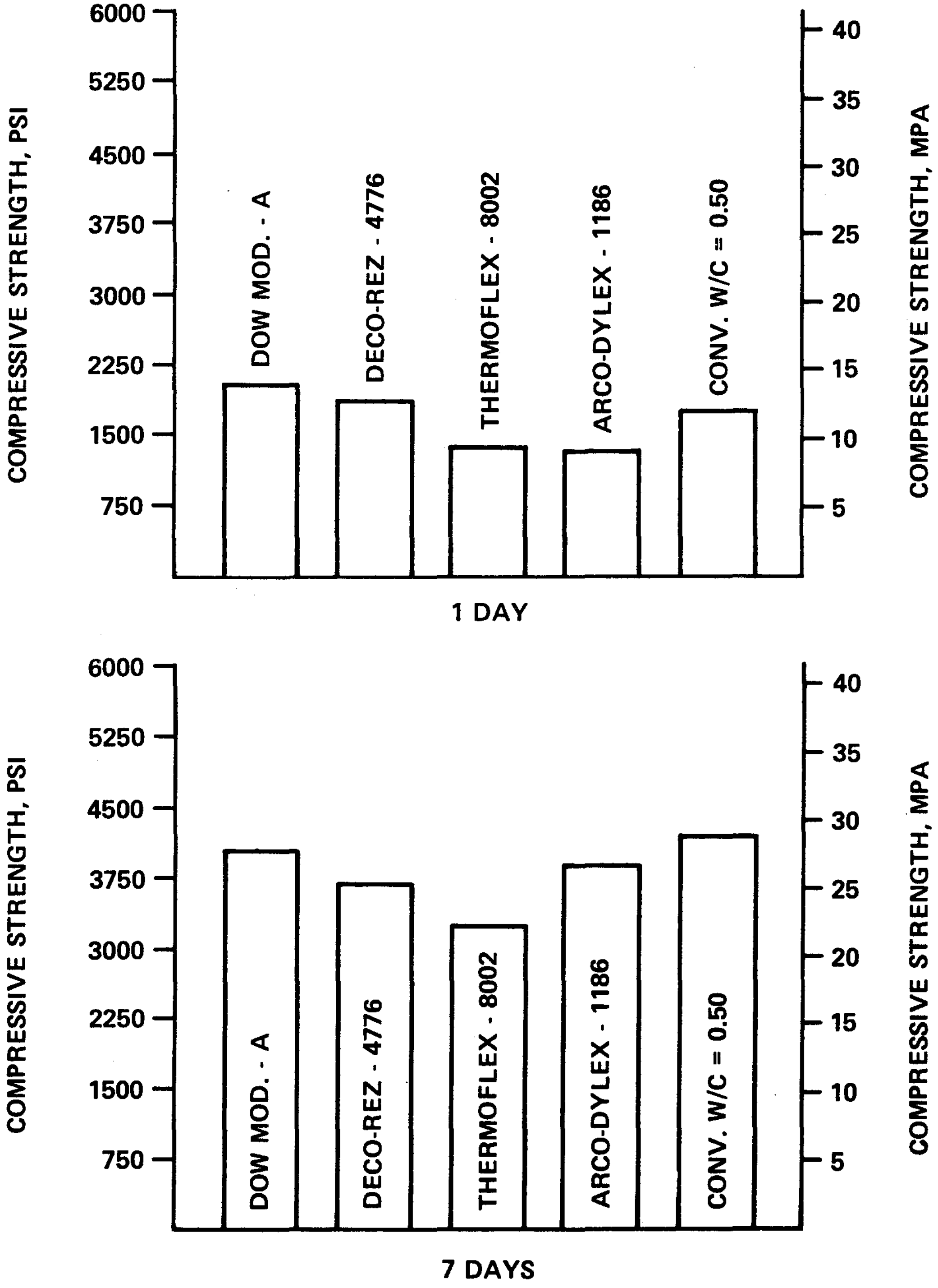


FIGURE 2A. AVERAGE COMPRESSIVE STRENGTHS.

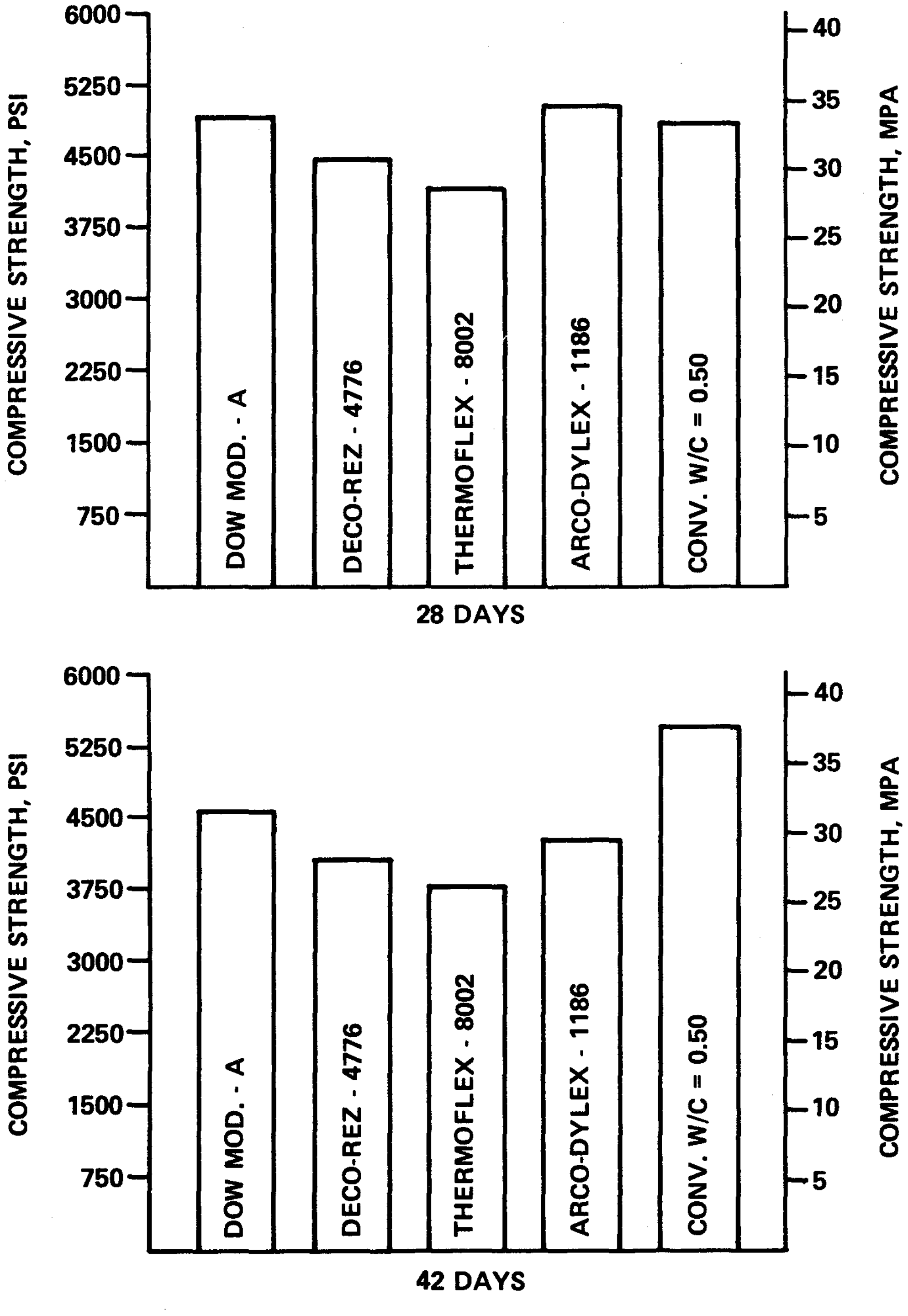


FIGURE 2B. AVERAGE COMPRESSIVE STRENGTHS.

Table 3. Compressive strength.

Compressive Strength[1], psi at

	1 day		7 days		28 days		42 days		Average % change
	Average	SD	Average	SD	Average	SD	Average	SD	28 to 42 days[2]
Dow Modifier A	2050	99	4030	150	5010	118	4560	71	-9.0
Deco-Rez 4776	1890	192	3675	208	4460	387	4060	62	-9.0
Thermoflex 8002	1370	66	3215	96	4170	345	3780	254	-9.4
Arco Dylex 1186	1360	38	3845	353	5095	86	4255	125	-16.5
Conventional Concrete w/c = 0.5	1735	173	4180	398	4960	219	5395	243	+8.8
Conventional Concrete w/c = 0.4	2370	72	4160	146	6015	422	—	—	—

[1] Average compressive strength of six 3- by 6-inch (0.076- by 0.152-m) cylindrical specimens at each age (three specimens at each age from each of two separate batches made on different days). SD = standard deviation. Sand content of concrete = 60 percent of volume of total aggregate.

[2] $$\frac{\text{(Average 42-day strength - Average 28-day strength) x 100}}{\text{Average 28-day strength}}$$

[3] To convert psi to MPa, multiply by 6.894×10^{-3}.

compressive strength test results. Specimens of three sizes (2- by 2-inch (51- by 51-mm) cubes, 3- by 6-inch (76- by 152-mm) cylinders, and 6- by 12-inch (152- by 305-mm) cylinders) were fabricated from each of two batches and cured in the manner described above for latex-modified concrete. The average results of the tests are shown in Figure 3 for test ages of 1, 7, 28, and 42 days. At 1 day, all three specimen sizes yielded similar average compressive strengths. However, at the other test ages, the 2- by 2-inch (51- by 51-mm) cubes indicated higher strengths (by about 1,000 to 1,500 psi (6.9 to 10.3 MPa)) than either of the two sizes of cylinders. The reduction in strength upon limewater soaking (from 29 to 42 days of age) was, however, indicated by the 2- by 2-inch (51- by 51-mm) cubes. The strength results for the cylindrical specimens of both sizes were virtually identical except at 42 days of age (i.e., after the limewater soak) when the 3- by 6-inch (76- by 152-mm) cylinders indicated a strength decrease, whereas, the 6- by 12-inch (152- by 305-mm) cylinders did not, probably because of their larger mass. Thus, since it is desirable to identify any water susceptibility during the 14-day limewater soak and simultaneously obtain realistic strength measurements, it appears that the 3- by 6-inch (76- by 152-mm) cylinder is the best specimen for compressive strength testing of latex-modified concrete with 1/2 inch maximum size coarse aggregate.

Flexural Strength

The flexural strengths of the latex-modified and conventional concretes were determined at 7, 28, and 42 days of age using 3- by 4- by 16-inch (76- by 102- by 406-mm) beams in accordance with the third point loading procedure in ASTM C78-75. The latex concretes were cured for 1 day under wet burlap, 27 days in lab air, followed by 14 days in saturated limewater, whereas, the conventional concrete was cured 1 day under wet burlap, 27 days in the moist room, and 14 days in limewater.

In general, the findings presented in Figure 4 and Table 4 show that latex-modified concrete exhibits higher flexural strength than conventional concrete. However, after soaking for 14 days in limewater, the flexual strengths of the latex concretes decreased to levels only slightly greater than that of conventional concrete. The Thermoflex 8002 latex concrete is an exception in that its flexural strength was approximately equal to that of the conventional concrete at all ages and did not decrease upon limewater soaking. Similarly as in the compressive strength tests, the concrete made using the Arco Dylex 1186 latex emulsion exhibited the highest flexural strength after air drying (i.e., at 28 days) and also the greatest decrease after the limewater soak.

Although the range of flexural strengths of the four air-dried, latex-modified concretes at 28 days of age was quite large, from 725 psi (5.00 MPa) for Thermoflex 8002 to 1020 psi (7.03 MPa) for Arco Dylex 1186, the range at 42 days (after limewater soaking) was small, 768 psi (5.30 MPa) for Deco-Rez 4776 to 845 psi (5.83 MPa) for Dow Modifier A.

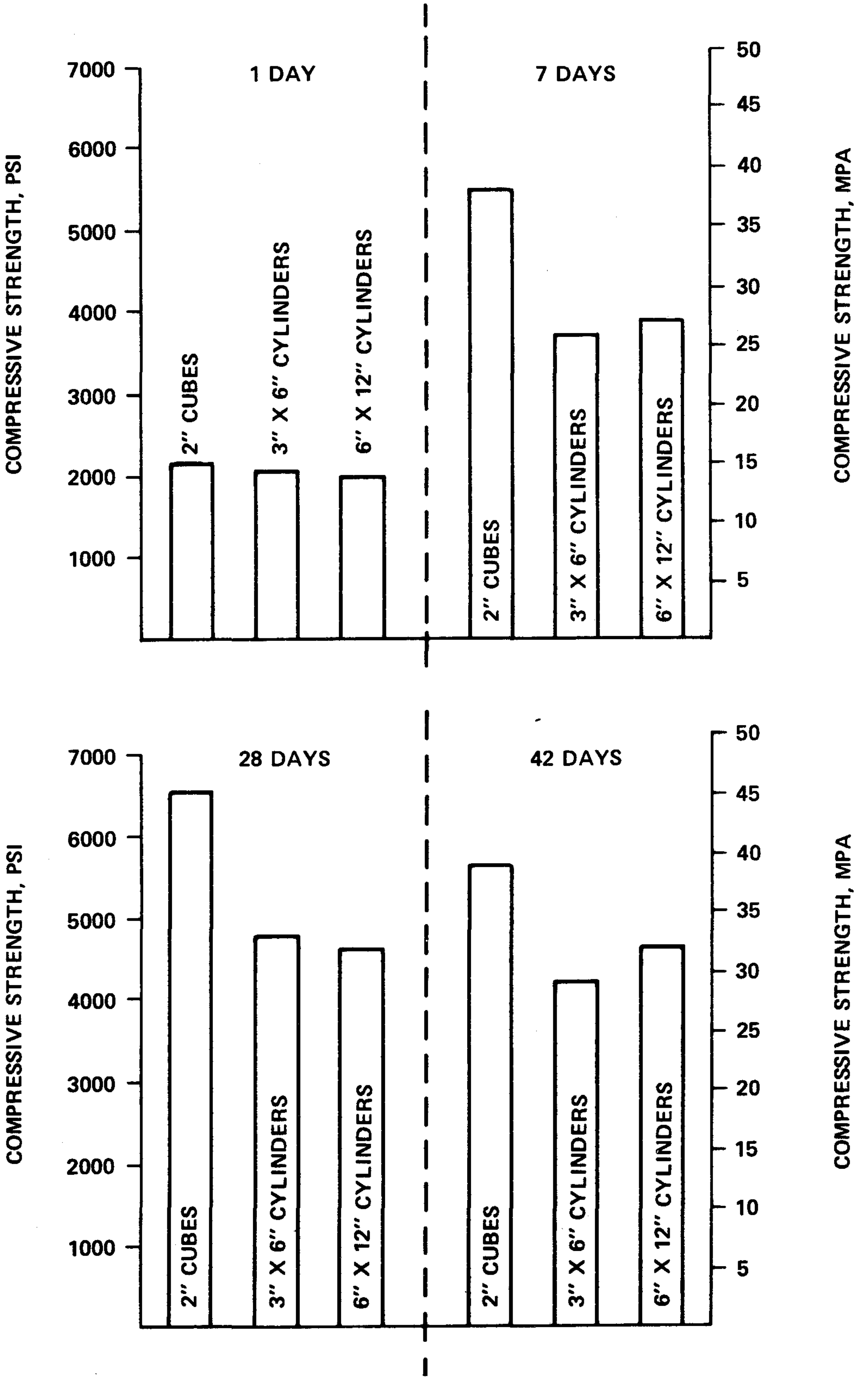


FIGURE 3. AVERAGE COMPRESSIVE STRENGTHS INDICATED BY VARIOUS SIZE LATEX MODIFIED CONCRETE SPECIMENS.

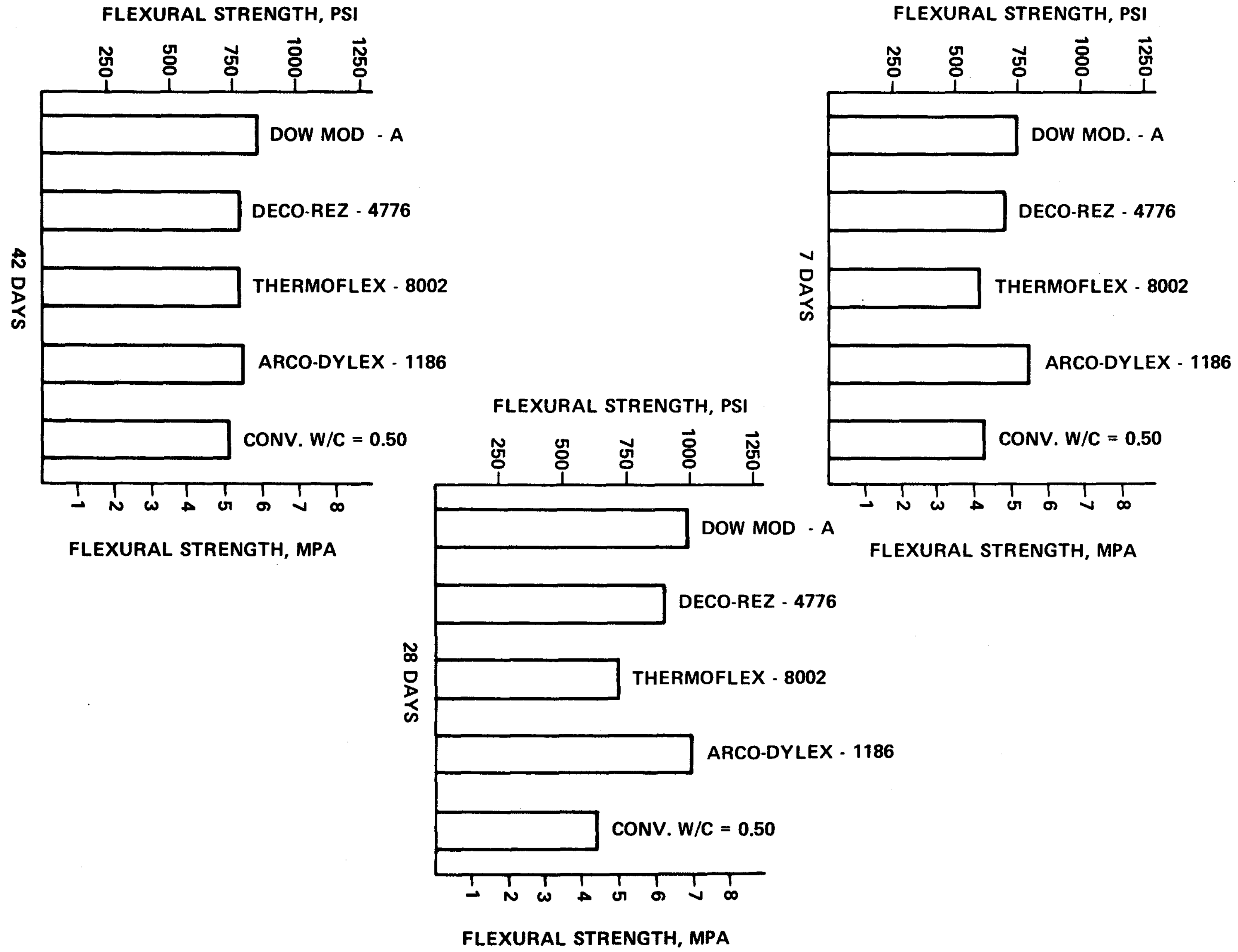


FIGURE 4. AVERAGE FLEXURAL STRENGTHS.

Table 4. Flexural strength.

		Flexural Strength[1], psi at						
		7 days		28 days		42 days		Average % Change
		Average	SD	Average	SD	Average	SD	28 to 42 days[4]
Dow Modifier A	- Batch 1[2]			955	48			
	- Batch 2[3]	750	54	990	26	845	45	-14.6
Deco-Rez 4776	- Batch 1			985	68			
	- Batch 2	705	61	905	34	768	3	-15.1
Thermoflex 8002	- Batch 1			670	20			
	- Batch 2	600	33	725	30	775	17	+6.9
Arco Dylex 1186	- Batch 1			1060	51			
	- Batch 2	790	51	1020	61	785	29	-23.0
Conventional Concrete w/c = 0.5	- Batch 1			595	43			
	- Batch 2	640	21	680	34	735	44	+8.0
Conventional Concrete w/c = 0.4	- Batch 1			625	25			

[1] Determined using 3- by 4- by 16-inch beams in accordance with AMTM C78-75. SD = standard deviation. See text for curing procedures.

[2] Three specimens per testing age. The fine to coarse aggregate ratio in the concrete was 55:45 by volume of total aggregate.

[3] Four specimens per testing age. The fine to coarse aggregate ratio in the concrete was 60:40 by volume of total aggregate.

[4] To convert psi to MPa, multiply by 6.894×10^{-3}.

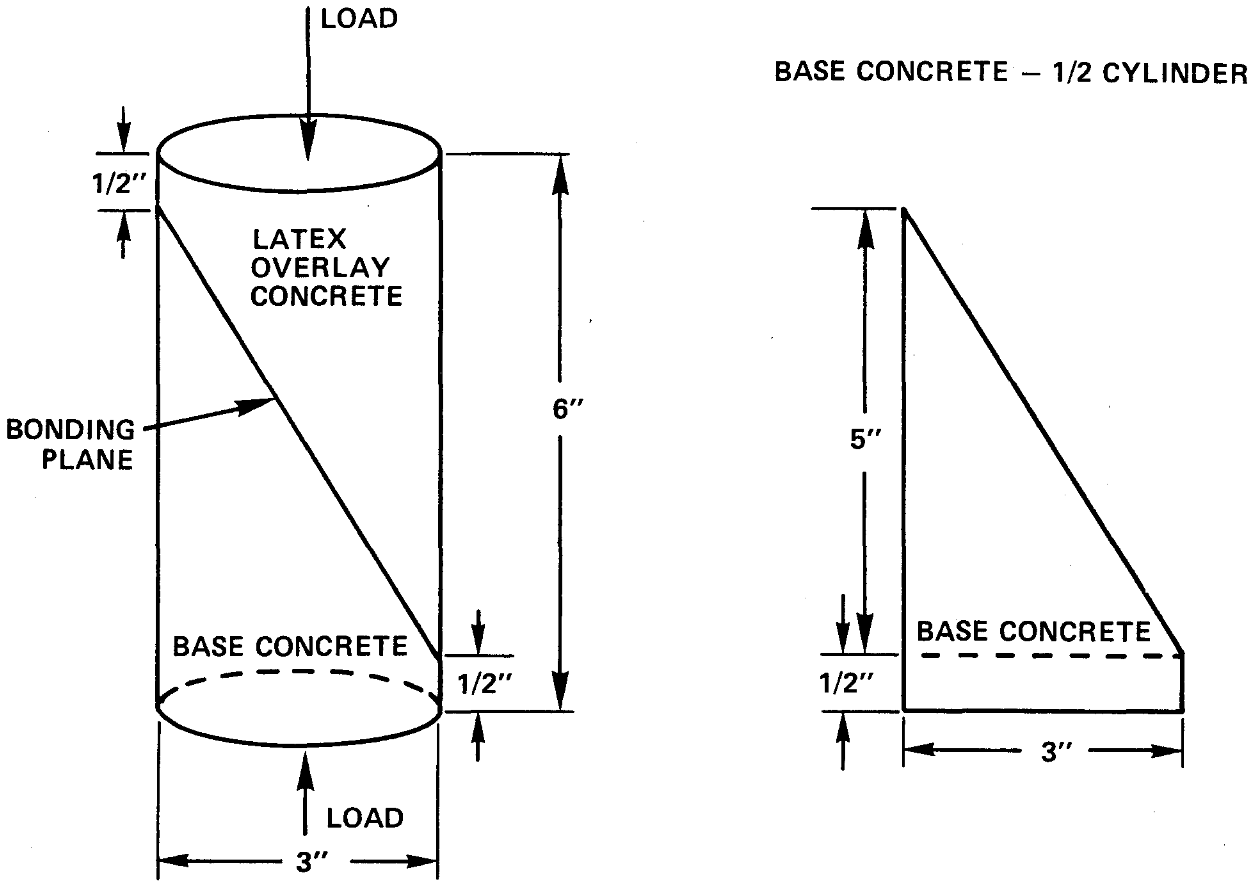
Bond to Underlying Concrete

The bond of the various latex-modified concretes to underlying portland cement concrete was studied using the Arizona Slant Shear Bond Test (1). Three- by six-inch (0.076- by 0.152-m) conventional concrete (w/c = 0.5, CF = 7) cylinders were cast and moist-cured for 28 days and then cut with a diamond saw along the diagonal (see Figure 5). One-half of each damp cylinder was then placed in a cylinder mold, and latex concrete was used to form the top of the cylinder after mortar from the latex mix was brushed onto the bonding plane. The specimens were then cured 1 day wet burlap, 27 days lab air, and 14 days in saturated limewater. The strengths of the composite cylinders were determined at 1, 7, 28, or 42 days of age by loading in compression. Companion cylinders from the same latex-modified concrete batches were also made and tested at identical ages and curing procedures. The compressive force for failure was calculated by dividing the ultimate load by the cross sectional area in all instances. Although this does not yield a bond or shear strength per se, comparison of the compressive force required for failure of the composite cylinders with that required to fail the full latex cylinders provides an indication of the strength of the bond compared to that of the overlay concrete.

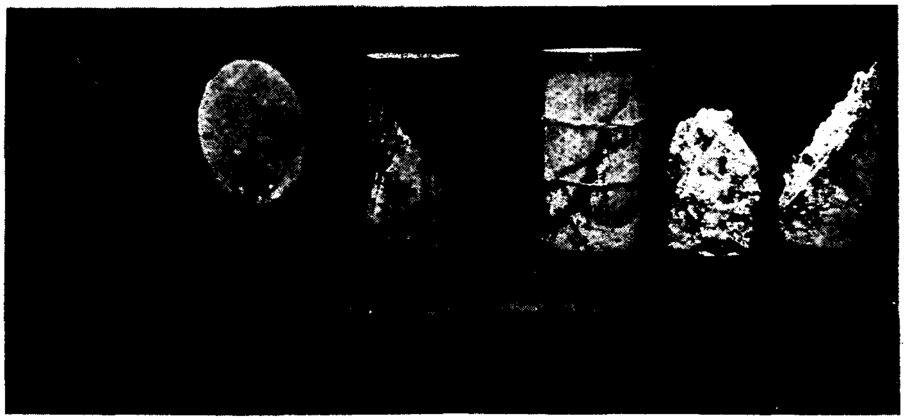
The results of this testing (Figure 6 and Table 5) show that at room temperature, the compressive forces required for failure of the composite cylinders were proportional to the compressive strengths of the full latex cylinders. In other words, as the compressive strength of the full latex cylinders increased during air drying from 1 to 28 days, the force to fail the composite cylinders also increased; whereas, the limewater soak from 28 to 42 days of age resulted in a decrease in the compressive force required to fail both types of cylinders. On the average, for all four latex overlays at all test ages, the compressive force required to fail a composite cylinder was 60 percent of the compressive strength of the companion monolithic latex-modified concrete cylinder. The ranges of composite cylinder strengths for the four latex-modified concretes were not large at 1 day of age (860 psi (5.93 MPa) for Thermoflex 8002 to 1050 psi (7.24 MPa) for Deco-Rez 4776), and no large differences are readily apparent at any age, although the Arco Dylex 1186 average composite cylinder strengths were higher at 7, 28, and 42 days than those of the other three latex-modified concretes.

A conventional concrete (w/c = 0.50, CF = 7, Air = 6 percent) overlay was also tested. This concrete was cured 1 day under wet burlap, then 27 days moist, plus 14 days saturated limewater, and a portland cement grout was used as a bonding agent. Surprisingly, the composite cylinder compressive strengths (Table 5) of the conventional concrete equaled or exceeded those of the latex-modified concretes at all ages.

The major difference between the conventional and latex concrete composite cylinder strengths was that the conventional concrete exhibited an increase in composite cylinder strength



1 inch = 25.4 mm



CUT LOWER HALVES

COMPOSITE CYLINDER

FAILED CYLINDERS AFTER TEST

FIGURE 5. ARIZONA SLANT SHEAR BOND SPECIMENS.

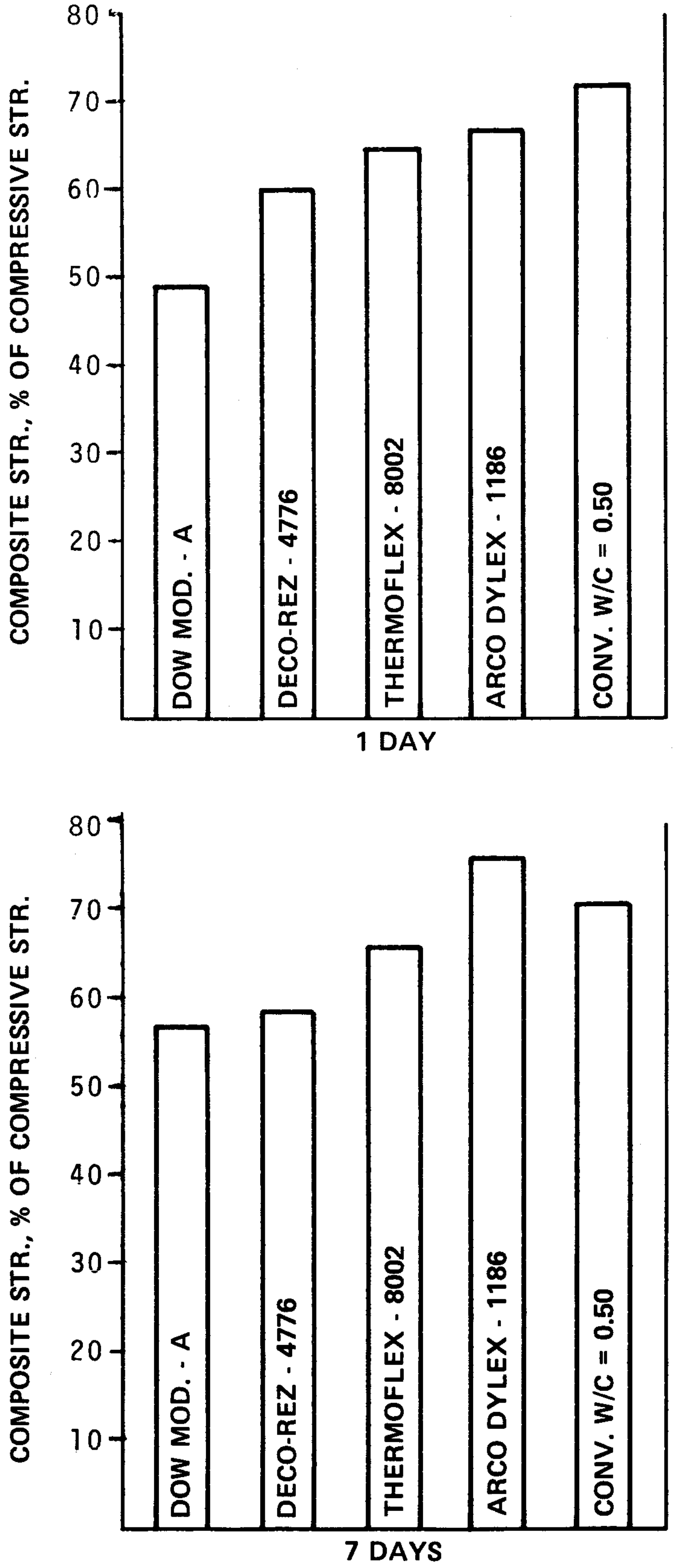


FIGURE 6A. AVERAGE SLANT SHEAR BOND STRENGTHS.

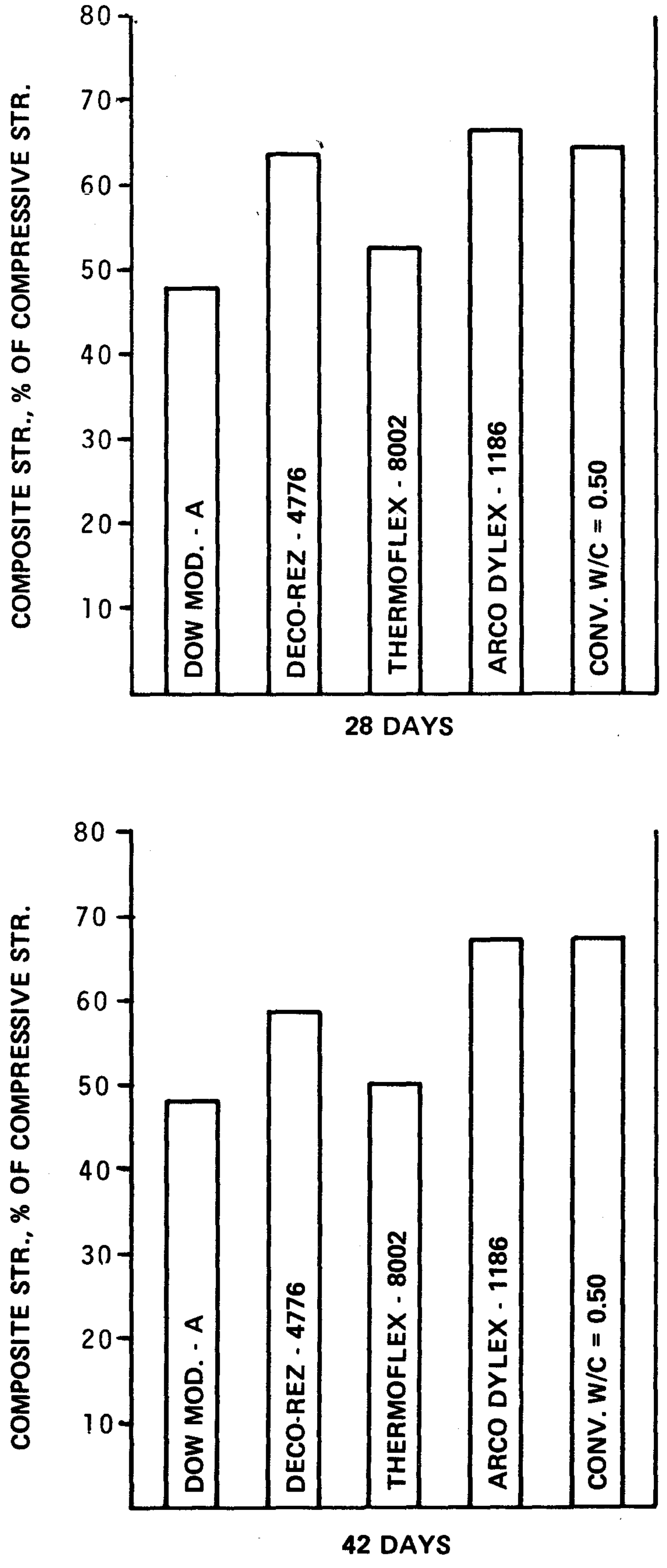


FIGURE 6B. AVERAGE SLANT SHEAR BOND STRENGTHS.

Table 5. Arizona slant shear bond test results.

Overlay Concrete	Overlay Concrete Age When Loaded in Compression								
	1 day		7 days		28 days		42 days		Average % change[1]
	Average	SD	Average	SD	Average	SD	Average	SD	28 to 42 days
Dow Modifier A[3]									
Composite psi	980	193	2270	294	2510	161	2200	315	-12.4
Monolithic psi	1970	44	3910	100	5110	70	4540	43	-11.2
Percent	49.7		58.1		49.1		48.5		
Deco-Rez 4776									
Composite psi	1050	67	2080	177	2780	458	2390	411	-14.0
Monolithic psi	1720	29	3490	23	4260	482	4020	25	-5.6
Percent	61.0		59.6		65.3		59.5		
Thermoflex 8002									
Composite psi	860	98	2220	206	2400	319	1990	273	-17.1
Monolithic psi	1310	31	3300	21	4460	87	3930	277	-11.9
Percent	65.6		67.3		53.8		50.6		
Arco Dylex 1186									
Composite psi	930	42	2800	387	3510	200	2870	496	-18.2
Monolithic psi	1380	35	3620	402	5170	21	4200	134	-18.8
Percent	67.4		77.3		67.9		68.3		
Conventional Concrete w/c = 0.5, PC grout									
Composite psi	1140		2900		3400		3850		+13.2
Monolithic psi	1560	38	4030	69	5180	44	5630	49	+8.7
Percent	73.1		72.0		65.6		68.4		

[1] $$\frac{(\text{Average 42-day strength} - \text{Average 28-day strength}) \times 100}{\text{Average 28-day strength}}$$

[2] To convert psi to MPa, multiply by 6.894 x 10^{-3}.

[3] Composite psi is the compressive force required to fail the composite cylinder. Monolithic psi is the compressive force required to fail the full cylinders fabricated entirely of the overlay material. Percent is composite psi divided by monolithic psi, times 100. Psi is calculated by dividing ultimate load by the cross sectional area of the specimen. Three monolithic and three composite specimens per variable.

after limewater soaking from 29 to 42 days of age, whereas, the latex-modified concretes lost strength. This result was expected in light of the similar performance discussed earlier in the sections on compressive and flexual strengths.

Resistance to Rapid Freezing and Thawing

The resistances of the concretes to freeze-thaw damage were studied following the procedures described in ASTM C666-77 Method A. The latex concretes were cured 1 day wet burlap, 13 days lab air, and 14 days in limewater prior to test, while the conventional concretes received a 1-day wet burlap, 13-day moist, and 14-day limewater cure. After completion of the required 300 freeze-thaw cycles, the flexual strength of each specimen was determined in accordance with ASTM C78-75.

The results of this testing (Table 6) show that for the curing procedure given above, the Thermoflex 8002 and Arco 1186 latex concretes and the air-entrained conventional concretes are resistant to damage through 300 cycles of rapid freezing and thawing. Conversely, the latex concretes made with Dow Modifier A and Deco-Rez 4776 latices were damaged significantly by the testing. For example, the average flexual strength of the Dow Modifier A concrete dropped from 845 psi (5.83 MPa) prior to testing to 200 psi (1.38 MPa) after 300 cycles, and the dynamic modulus after 300 cycles averaged only 34 percent of the original value. This finding was considered unusual because of the excellent field service history of Dow latex-modified concrete. Conversations with latex chemists led to the hypothesis that the Dow latex-modified concrete may require more air drying time for the polymer particles to unite and bond throughout the specimens. To check this hypothesis, additional tests were run after the following cures:

1. 1 day wet burlap, 27 days lab air, and 14 days limewater;
2. 1 day wet burlap, 41 days lab air, and 28 days limewater; and
3. 1 day wet burlap and 27 days lab air.

The results of these tests, also presented in Table 6, show that the Dow Modifier A concrete was not significantly damaged by the 300 freeze-thaw cycles for either extended curing procedure (i.e., durability factors of 87 and 84 and flexural strengths of 585 psi (4.03 MPa) and 620 psi (4.27 MPa) after 300 freeze-thaw cycles for Cures 1 and 2 above, respectively). Concrete tested using the curing procedure described in item 3 above, on the other hand, showed some damage (durability factor of 63 and flexural strength of 340 psi (2.34 MPa) after 300 freeze-thaw cycles), although the damage in this instance (27 days air drying and

Table 6. Results of fast freeze-thaw tests.

Cure Prior to Testing and Concrete Type	Results after 300 Freeze-Thaw Cycles	
	Average % of Original Dynamic Modulus[1]	Average Flexural Strength[2], psi[3]
1. Cured 1 day wet burlap, 13 days fog room, 14 days limewater		
Conventional, w/c = 0.50 Air content = 6 percent	90	619
2. Cured 1 day wet burlap, 13 days lab air, 14 days limewater		
Dow Modifier A	34	201
Deco-Rez 4776	31	197
Thermoflex 8002	93	633
Arco Dylex 1186	87	625
Dow Modifier A - with air-entraining agent (6 percent air)	92	780
3. Cured 1 day wet burlap, 27 days lab air		
Dow Modifier A	63	340
4. Cured 1 day wet burlap, 27 days lab air, 14 days limewater		
Dow Modifier A	87	585
5. Cured 1 day wet burlap, 41 days lab air, 28 days limewater		
Dow Modifier A	84	620

[1] Three specimens per variable tested using ASTM C666-77 Method A.

[2] Three specimens per variable tested using ASTM C78-75.

[3] To convert psi to MPa, multiply by 6.894×10^{-3}.

testing initiated at 28 days) was less than that which occurred within the earlier specimens cured 1 day wet burlap, 13 days lab air, and 14 days limewater prior to testing.

Although the above testing tends to confirm the hypothesis that greater air drying and specimen age are needed for adequate ASTM C666 performance of the Dow material, it does not explain the early age differences in performance between the various latex concretes. The only obvious difference in the concretes is that those which performed well in the fast freeze-thaw test (Arco and Thermoflex) had high air contents (8 and 9 percent, respectively), whereas, the concretes which performed poorly (Dow and Deco-Rez) had only about 4 percent air. Examinations of the air void systems within the various concretes (in accordance with the linear traverse procedures in ASTM C457-71) were performed to further study this difference. The findings, presented in Table 7, indicate that the void spacing factors for the Thermoflex and Arco concretes were much less than those for the Dow and Deco-Rez materials. Thus, the high air contents and the resulting superior air void systems are probably one cause of the superior early age freeze-thaw durability of the Thermoflex and Arco concretes. It is interesting to note, however, that the spacing factors for the Thermoflex and the Arco concretes were larger than the 0.008-inch (0.20-mm) spacing factor normally considered necessary for freeze-thaw resistant conventional concrete.

Even greater evidence that the early age freeze-thaw durability relates to the air void system within the latex-modified concrete was obtained by performing freeze-thaw tests on high air content Dow Modifier A concrete. A small amount of vinsol resin air-entraining agent was added to a Dow latex-modified concrete mix to increase the air content to about 6 percent. Beams made from the concrete were subjected to 300 freeze-thaw cycles after curing 1 day wet burlap, 13 days lab air, and 14 days saturated limewater. In these tests, the Dow Modifier A concrete performed very well (average durability factor = 92 and flexural strength after 300 cycles = 780 psi (5.38 MPa)). As expected, the air void spacing factor within the 6 percent air Dow Modifier A concrete was lower than that within the Dow concrete with only 4 percent air. However, although the spacing factor was reduced, it still was about double the 0.008-inch (0.20-mm) generally accepted limit for conventional concrete; thus, it does not appear that the 0.008-inch (0.20-mm) criterion is applicable to the latex-modified concretes studied herein.

Although the air void testing did result in an understanding of the fast freeze-thaw test findings, the reader is cautioned from concluding that Dow Modifier A and Deco-Rez latex-modified concretes will exhibit poor early age freeze-thaw durability on bridge decks. In general, the field service history of the Dow material has been excellent and it may well be that the ASTM C666

Method A test procedure is much more severe than actual field exposure on bridge decks. Additional study is needed in this area to fully understand the differences in performance and the significance of the test results. Additionally, another possible cause of differences in early age, fast freeze-thaw results which deserves attention in future studies is the effect of reactive groups in the polymer particles. Some of the S/B emulsions contain such groups designed to react with the calcium in cement, whereas others do not.

Table 7. Studies of the air void[1] systems in various concretes.

Concrete	Air Content (percent)		Specific Surface, inches^{-1}	Void Spacing Factor $\bar{L}$, inches[3]
	Fresh Concrete	Hardened Concrete		
Dow Modifier A - no AEA[2]	4.5	3.84	294	0.0210
	4.5	2.89	301	0.0231
Dow Modifier A - with AEA	5.9	5.17	289	0.0167
	5.9	5.16	341	0.0153
Conventional Concrete - with AEA	7.0	6.70	513	0.0078
Conventional Concrete - no AEA	2.1	1.65	272	0.0293
Deco-Rez 4776	4.4	4.40	235	0.0253
Thermoflex 8002	9.0	8.97	377	0.0104
		8.59	394	0.0102
Arco Dylex 1186	8.0	6.88	377	0.0120
		7.16	415	0.0106

[1] Linear traverse air void studies of hardened concrete performed in accordance with ASTM C457-71. Paste contents were determined and used in calculation of spacing factor.

[2] AEA = air-entraining agent.

[3] To convert inches to mm, multiply by 25.4.

Deicer Scaling

The resistance of the various concretes to deicer scaling was determined in accordance with ASTM 672-76 except that a 3-percent NaCl solution was used rather than a calcium chloride solution. The latex-modified concretes were cured 1 day wet burlap, 13 days lab air, plus 14 days limewater prior to test, while the conventional concretes were cured 1 day wet burlap, 13 days moist room, plus 14 days limewater.

The findings, presented in Table 8, show that all the latex concretes are resistant to deicer scaling if the often suggested criterion of a maximum allowable rating of 3 at 50 cycles is used. However, there were significant differences in performance through 90 cycles. The Thermoflex 8002 and Arco Dylex 1186 latex concretes were the best performers, exhibiting no scaling damage, followed by the Deco-Rez 4776 latex concrete and the air-entrained conventional concrete. The relatively poor performance of the Dow Modifier A latex concrete was surprising in light of the excellent field history available on the material and the excellent scaling resistance of large Dow Modifier A concrete slabs through 5 years of outdoor exposure with daily salting (2, 3). As discussed in the section on freeze-thaw testing, the cause was possibly the short air drying period (13 days) prior to limewater soak, and the early age of the concrete when tested (28 days); although the point that the air content of the Dow Modifier A concrete was only half that of the Thermoflex 8002 and Arco Dylex 1186 concretes may also have had an effect. Additional study is needed in this area to fully understand the differences in performance and the significance of the test results.

After the 90 scaling cycles were completed, samples from each concrete were taken for chloride analyses. The results of these analyses are presented in the section entitled Chloride Permeability.

Chloride Permeability

The chloride permeabilities of the various concretes were evaluated using a 90-day ponding test. After small concrete slabs were made and cured, the surfaces were sandblasted to remove any latex film which, in the field, would be similarly removed by traffic. The concrete was then conditioned in a drying room at 73° ± 2°F (23° ± 1°C), 50 ± 4 percent Rh for 7 days. It was then continuously ponded with a 3-percent NaCl solution for 90 days, and chloride ingress after ponding was determined using the FHWA wet chemical analysis procedure for total chloride (4). The fine/coarse aggregate ratio in all these concrete mixes was 55:45 by volume and the other mix properties are presented in the section entitled Concrete Mixes. Specifically, the curing procedures were:

Latex concrete - 1 day wet burlap, 20 days lab air, sandblast surface, 7 days lab air, start ponding.

Conventional concrete - 1 day wet burlap, 13 days moist room, 6 days lab air, sandblast surface, 7 days lab air, start ponding.

The findings of the chloride testing are given in Figure 7 and Table 9 for two sampling depths (1/16 to 1/2 inch (1.6 to 13 mm) and 1/2 to 1 inch (13 to 25 mm)). In addition to the four

Table 8. Resistance to deicer scaling.

	Scaling Rating[1] at Cycles Indicated							
	25		50		75		90	
	Median	Range[2]	Median	Range	Median	Range	Median	Range
Dow Modifier A	1	0 - 1	3	3	4	4	5	5
Deco-Rez 4776	0	0	0	0	2	1 - 2	3	3
Thermoflex 8002	0	0	0	0	0	0	0	0
Arco Dylex 1186	0	0	0	0	0	0	0	0
Conventional Concrete, w/c = 0.5, Air = 7.0%	1	1	1	1	2	1 - 2	2	1 - 2
Conventional Concrete, w/c = 0.5, Air = 2.0%	5	5	5	5	5	5	5	5

[1] ASTM C672-76 rating scale of 0 (no scaling) to 5 (severe scaling).

[2] Four slabs for each concrete. The range of scaling ratings are listed. If only a single number is listed, all four slabs exhibited that rating.

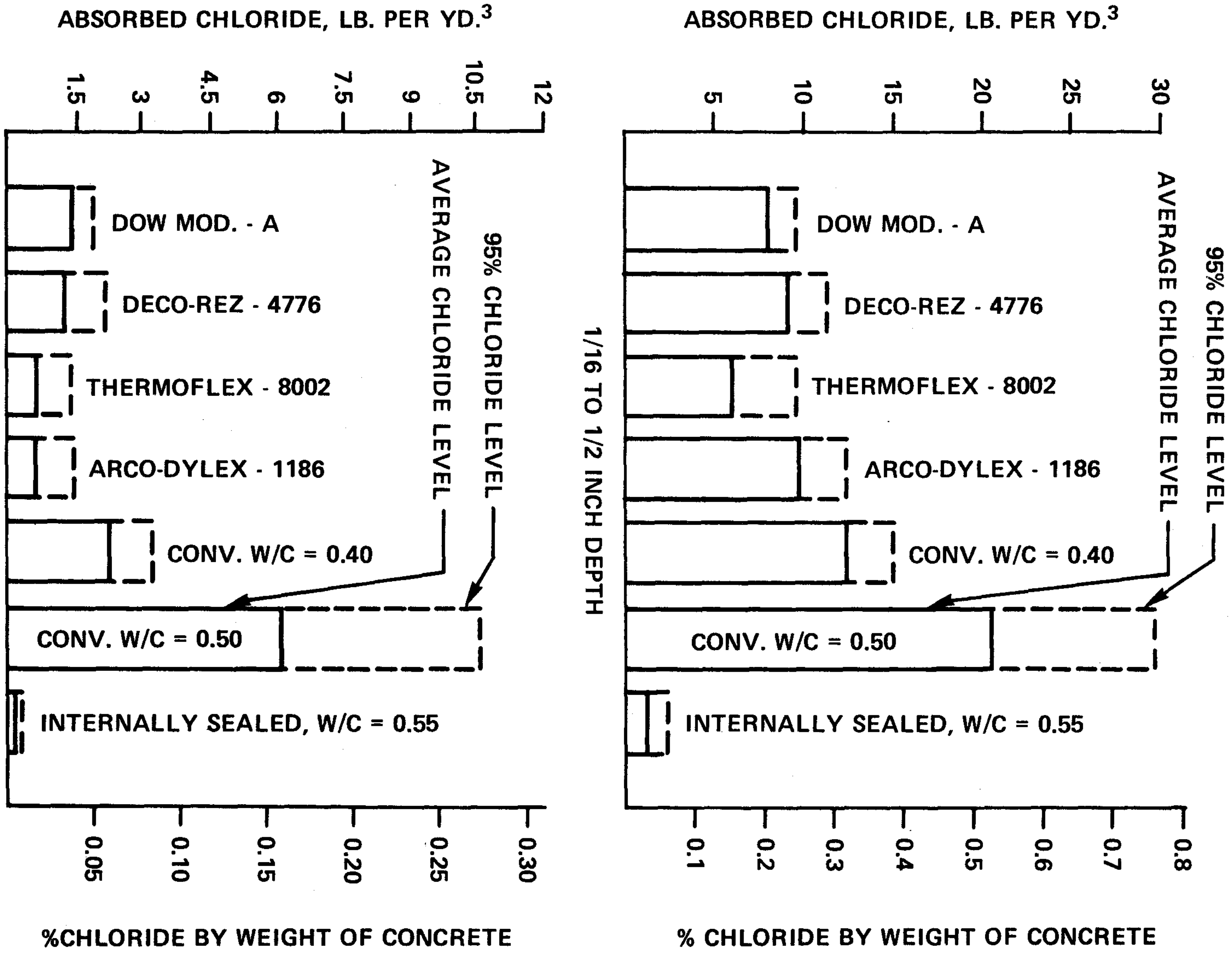


FIGURE 7. CHLORIDE PERMEABILITIES

Table 9. Chloride permeability findings.

Concrete	Mean[2] Sampling Depth, (inches)	Number of Samples	Clorides Absorbed[1], lb $\mathrm{Cl}^-/\mathrm{yd}^3$ Average	Standard Deviation	Median	Min.	Max.	95% Cl^- level[3]
1. Series 1 - 55:45 sand/stone ratio by volume; surface sandblast; ponded at 28 days.								
Dow Modifier A	0.28	8	8.07	0.91	8.00	6.37	9.14	9.57
	0.75	9	1.49	0.30	1.42	1.08	1.97	1.98
Deco-Rez 4776	0.28	9	8.99	1.40	8.89	6.46	10.93	11.29
	0.75	9	1.29	0.54	1.34	0.50	2.19	2.18
Thermoflex 8002	0.28	10	5.89	2.30	5.21	2.91	9.43	9.67
	0.75	10	0.58	0.48	0.45	0.06	1.57	1.37
Arco Dylex 1186	0.28	9	9.87	1.50	9.86	7.95	12.37	12.34
	0.75	9	0.63	0.51	0.43	0.17	1.64	1.47
Conventional Concrete, w/c = 0.40	0.28	9	12.41	1.56	12.15	10.78	15.74	14.98
	0.75	8	2.24	0.62	2.34	1.43	3.27	3.26
Conventional Concrete, w/c = 0.43	0.28	9	13.73	1.37	13.58	12.22	15.77	15.98
	0.75	9	3.85	0.55	4.17	2.80	4.44	4.75
Conventional Concrete,[5] w/c = 0.50	0.28	16	20.64	5.53	21.36	13.06	29.61	29.74
	0.75	16	6.09	2.70	5.58	2.81	12.42	10.53
Internally Sealed Concrete, w/c = 0.55	0.28	9	1.20	0.72	1.30	0.08	2.31	2.38
	0.75	9	0.10	0.08	0.12	0.00	0.20	0.23
2. Series 2 - 60:40 sand/stone ratio by volume; ponded at 28 days.								
Dow Modifier A - No sandblast	0.28	9	6.99	1.99	6.33	5.15	10.32	10.26
	0.75	9	0.86	0.51	0.61	0.42	1.94	1.70
Dow Modifier A - Sandblast	0.28	9	6.87	1.53	6.79	5.03	9.41	9.39
	0.75	9	0.56	0.42	0.52	0.05	1.50	1.25

Table 9. Chloride permeability findings. (continued)

Concrete	Mean[2] Sampling Depth (inches)	Number of Samples	Clorides Absorbed[1], lb Cl$^-$/yd^3					
			Average	Standard Deviation	Median	Min.	Max.	95% Cl$^-$ level[3]
3. Series 3 - 60:40 sand/stone ratio by volume; surface sandblast.								
Dow Modifier A - ponded at 28 days	0.28	6	5.68	1.92	5.68	3.18	7.91	8.84
	0.75	6	0.55	0.39	0.34	0.24	1.14	1.19
Dow Modifier A - ponded at 42 days	0.28	6	9.23	0.60	9.09	8.66	10.38	10.22
	0.75	6	0.90	0.43	0.82	0.44	1.65	1.61
Dow Modifier A - ponded at 90 days	0.28	6	11.29	2.36	12.91	8.27	13.47	15.17
	0.75	8	1.17	0.56	1.22	0.48	2.20	2.09
4. Kentucky DOT 90-day ponding tests.								
Dow Modifier A	0.28	8	6.49	0.53	6.39	5.63	7.52	7.36
	0.75	9	1.04	0.63	0.81	0.37	2.46*	2.08
*This value (2.46 lb/yd^3 may be an outlying data point. If it is discarded, the following values result:								
Dow Modifier A	0.75	8	0.87	0.35	0.81	0.37	1.52	1.45
Deco-Rez 4776	0.28	9	6.86	0.78	6.47	6.07	8.44	8.14
	0.75	7	0.87	0.42	0.93	0.23	1.45	1.56
Thermoflex 8002	0.28	8	4.84	0.72	4.80	3.89	5.80	6.02
	0.75	9	0.70	0.37	0.66	0.28	1.58	1.31

Table 9. Chloride permeability findings. (continued)

			Clorides Absorbed[1], lb Cl^-/yd^3					
Concrete	Mean[2] Samplng Depth (inches)	Number of Samples	Average	Standard Deviation	Median	Min.	Max.	95% Cl^- level[3]
5. Deicer scaling slabs - after 90 daily cycles.								
Dow Modifier A	0.28	6	3.05	0.78	2.94	1.96	4.19	4.33
Deco-Rez 4776	0.28	6	4.82	0.73	4.86	3.84	6.00	6.02
Thermoflex 8002	0.28	6	3.61	1.03	3.92	1.69	4.54	5.30
Arco Dylex 1186	0.28	6	2.68	0.77	2.74	1.68	3.48	3.95

Notes:

[1] Chloride absorbed = Total chloride minus average baseline (before ponding) chloride.

[2] Mean sampling depths are given. The actual depths were 1/16 to 1/2 inch and 1/2 to 1 inch.

[3] The 95-percent chloride is a statistically obtained value which indicates that 95 percent of the chloride contents in the sample will be less than or equal to that value. A normal distribution is assumed and it is calculated as follows:

95% Cl^- level = Average Cl + (Standard Deviation (X)), where X is a constant equal to 1.645.

[4] To convert inches to mm, multiply by 25.4.
To convert lb/yd^3 to kg/m^3, multiply by 0.5933.
To convert lb Cl^-/yd^3 to percent chloride by weight of concrete, multiply by 0.0255.

[5] The 0.50 water/cement ratio conventional concrete "sample" includes data from three concrete batches made several months apart.

latex concretes, the permeability test was also performed on conventional concretes with water/cement ratios of 0.40 and 0.50, and internally sealed (wax bead) concrete (5) with a 0.55 water/cement ratio. All data were analyzed statistically and materials were compared using the Student T Test and Duncans Multiple Range Test considering unequal replications and standard deviations (15).

The internally sealed concrete exhibited the lowest chloride permeability of all concretes studied. Statistical analysis of the data indicated that, at the 95-percent confidence level, internally sealed concrete (water/cement ratio of 0.55) is significantly less permeable than any of the latex-modified concretes (water/cement ratios of 0.36 to 0.40).

The Thermoflex 8002 latex concrete exhibited the lowest average chloride ingress of the four latex-modified concretes; although, the Thermoflex data exhibited relatively high variability. The 95-percent chloride level given in Table 9 for each material is generally considered a better measure of the overall permeability since it is a single, statistically obtained chloride level which indicates that 95 percent of the chloride contents at a particular depth encountered in the sample will be less than or equal to that value. It is based on the sample mean, standard deviation, and the assumption that the data are normally distributed. The 95-percent Cl^- level can exceed the maximum chloride content measured on the sample if the standard deviation of the data is high.

When this information is taken into account, it is obvious that all four latex-modified concretes have similar permeabilities. The range of 95-percent chloride levels for the four latex concretes was only:

1/16- to 1/2-inch depth - 9.57 lb Cl^-/yd^3 (Dow Modifier A) to 12.3 lb Cl^-/yd^3 (Arco Dylex 1186)

1/2- to 1-inch depth - 1.37 lb Cl^-/yd^3 (Thermoflex 8002) to 2.18 lb Cl^-/yd^3 (Deco-Rez 4776)

Several other chloride studies were also performed during development of the test method to study its applicability. The

---

Note: Statistically, the 95-percent chloride level for the population will only be equal to that of the sample if the mean and standard deviation of the population are equal to those of the sample. Such equality in reality is rare for the small number of samples normally used for chloride analyses; thus, other variability calculations would, in theory, be needed. However, for the purpose used herein (i.e., to describe the effect of chloride ponding on a sample), the 95-percent chloride value, as calculated, appears to be an acceptable indicator.

mix properties and curing and testing procedures were equivalent to those given above except that, at the recommendation of Dow Chemical Company, the fine/coarse aggregate ratio was changed to 60:40 by volume (i.e., more sand than in the above tests) for all concretes.

The effect of the sandblasting was studied by performing the ponding test on Dow Modifier A latex concrete slabs with and without the 1/8-inch (3-mm) surface sandblast prior to ponding. Statistical analysis of the results of this testing (Table 9) showed that, at the 95-percent confidence level, the sandblast had no effect on the chloride permeability of the latex concrete. It is, however, still considered an essential part of the test method. A comparison of the data for the sandblasted Dow Modifier A latex concrete with a 60:40 sand/stone ratio to that with a 55:45 sand/stone ratio showed that the higher sand content resulted in a slightly lower permeability.

To study the laboratory-to-laboratory reproducibility of the chloride test method, concretes were made and tested by the Kentucky Department of Transportation for comparison to the FHWA data (6). Three latex concretes (Dow Modifier A, Thermoflex 8002, and Deco-Rez 4776) were studied. Because of the use of local aggregates, the Kentucky latex concretes had water/cement ratios of 0.30 to 0.33, whereas, the FHWA latex concretes had water/cement ratios of 0.40. Thus, ingress into the Kentucky latex-modified concretes would be expected to be slightly less than the ingress found by FHWA. However, based on the FHWA data discussed above, the permeabilities of all three latex concretes tested by Kentucky should be similar. The Kentucky data in Table 9 show that, as expected: (1) the permeabilities of the three latex concretes are similar; and (2) the Kentucky latex concretes exhibited slightly lower permeabilities than those tested at a higher water/cement ratio by FHWA.

Dow Modifier A latex concrete was also studied to determine the effect of concrete age at the time of salting on chloride ingress. Sandblasting was done at the normal 21-day age, while ponding was initiated at three different concrete ages: 28 days, 42 days, and 90 days. After sandblasting, all concrete was stored in laboratory air (72 ± 2°F (23 ± 1°C), 50 percent Rh) and, thus, was subject to drying until ponding was initiated. All slabs in this series were fabricated from a single concrete batch to prevent batch-to-batch variation from distorting the findings.

The results, in Table 9, indicate that chloride ingress into the Dow Modifier A concrete increased as the age at initiation of salting increased. In fact, the 95-percent Cl^- levels were about twice as high for the specimens ponded at 90 days of age as for those ponded at 28 days of age. The reason for this is probably that the increased air drying period allowed more water to evaporate from the capillaries within the latex-modified concrete. As a result, chloride was able to penetrate more rapidly by capillary migration of the ponding solution than was possible for the early age concrete when migration of chloride through water-filled capillaries (in an attempt to achieve chemical equilibrium) was a more prominent migration mode. This study shows that conditioning of specimens prior to ponding is an important variable which should be specified in any chloride permeability test. The data also provide evidence against the hypothesis that the permeability of S/B modified concrete will decrease significantly when the material is conditioned in a low (50 percent) relative humidity environment between 28 and 90 days of age.

To study the chloride ingress during freeze-thaw cycling of the various latex concretes, chloride samples (1/16- to 1/2-inch depth (1.6- to 13-mm)) were taken after 90 scaling cycles (ASTM C672). The details of curing and testing are presented in the section of this report entitled Deicer Scaling and the chloride results are presented in Table 9. These specimens were not sandblasted prior to testing, and the fine/coarse aggregate ratio in the concretes was 60:40 by volume in all instances. The data show that in all instances chloride ingress during the 90 deicer scaling cycles was only about one-half that which occurred during the 90 days of continuous ponding in laboratory air; probably because the concrete was frozen at least 50 percent of the time and it had been soaked in limewater for 14 days prior to testing. Chloride ingress into the 1/16- to 1/2-inch (1.6- to 13-mm) depth of all the latex-modified concretes was of the same order of magnitude (95 percent chloride level range = 3.95 lb/yd^3 - 0.101 percent Cl$^-$ for Arco 1186 to 6.02 lb/yd^3 - 0.154 percent Cl$^-$ for Deco-Rez 4776), and thus, it appears that there were no obvious differences in chloride ingress during freeze-thaw cycling between the four latex concretes.

Ninety-Day Ponding Versus Long Term Daily Salting Test

The performance of the various concretes in this test (90-day continuous ponding) as compared to the performance of equal materials in the FHWA long term time-to-corrosion daily ponding test (2, 3, 5) is also of interest. In the latter research, large slabs (20 feet^2 (1.86 m^2)) were ponded daily to a 1/16-inch (2-mm) depth with a 3-percent NaCl solution for several years. A comparison of data shows that both studies yielded the following ranking of materials (least permeable listed first): internally sealed concrete, latex modified concrete (Dow Modifier A was the only emulsion studied in the time-to-corrosion effort), w/c 0.40 conventional concrete, and w/c 0.50 conventional concrete. Thus, the tests provide comparative data, although a direct comparison of data from the two tests is difficult because of differing sampling depths. However, based on the chloride versus depth curves in References 2 and 3 and the continuous ponding data, it appears that 90 days of continuous ponding after sandblasting is at least as severe as 300 daily time-to-corrosion salt applications (no sandblasting) for the low-permeability concretes studied herein.

III. Latex Emulsions - Chemical and Physical Properties

Introduction

Specifications for styrene-butadiene latex emulsions for bridge deck overlay concrete have, in recent years, defined the latex emulsion as having the following properties:

Solids Content	- 46.5 to 49.0 percent
Butadiene Content of Polymer	- 34 ± 1½ percent
Styrene Content of Polymer	- 66 ± 1½ percent
pH	- 9.5 to 11.0
Average Polymer Particle Size	- 1,900 to 2,500 Angstroms

The polymeric emulsion shall be stabilized with an anionic, non-ionic, and polydimethylsiloxane fluid surfactant in which the anionic surfactant is a sodium alkyl sulfate.

Color	- White
Weight per gallon	- 8.40 to 8.47 lb

The specifications did not include test methods for determining the above properties and thus, purchasers or users rarely, if ever, measured the properties of a latex emulsion to insure compliance with the specifications.

A survey of latex suppliers showed that there are several different methods in common use for measurement of almost every property of latices. A study of the various methods indicated that in most instances, the test procedures do not yield equivalent results. Thus, the meaning of each specification requirement varies with test procedure and from manufacturer to manufacturer. This is obviously an undesirable situation and was of concern in the study.

Another consideration in this study was that the emulsion specifications given above are for a single product, and the limits placed on each property are not necessarily those required for adequate performance. For example, a 50-percent solids emulsion is outside the specification limit but there is no data to indicate that such an emulsion will not perform as well as one with 47 percent solids.

Thus, the objectives of the testing described in this section were to (a) define reliable test methods for measuring the above chemical and physical properties of a styrene-butadiene latex emulsion and (b) to study the effect of emulsions which are outside the above specifications and thus permit the recommendation of limits based primarily on performance considerations.

The latex emulsion manufacturers listed in the Introduction section of this report aided in this effort by providing details on the quality control test procedures and limits used and the reasons for use of each procedure, by providing test data and general knowledge on the effect of varying emulsion properties on the properties of latex-modified concrete, and by manufacturing special emulsions for testing in our laboratory.

As this study progressed, recommendations of limits based on the performance effects of emulsions outside the above specifications became more important since it was determined that many of the emulsions did not meet all the requirements given above. This was the result of the differing production processes used by the various manufacturers and the different additives used as stabilizers, antioxidants, and antifoamers. The five emulsions studied actually represent a wide range of emulsion properties and by comparison of these properties with the concrete properties described in Section II of this report, performance-based limits are suggested. These limits can then be used in a prequalification program for new emulsions and in a certification program for prequalified emulsions to insure that each production batch is similar to the prequalified product. It is hoped that both manufacturers and users will adopt the test procedures described herein and that such standardization will aid in control and user understanding and satisfaction with the product.

Each of the tests performed on the emulsions is summarized below along with a discussion of test significance and the limits chosen. The results of the testing on each emulsion are also presented. The detailed test procedures are given in Section VI of the report. The discussion of test significance is limited in scope to include only the class of S/B emulsions for concrete studied herein.

Chemical and Physical Tests of Latices

Weight Percent Solids

The weight percent solids is equal to the quantity of polymer particles plus the solid portion of any additive contained in a latex emulsion. It is usually obtained by drying the emulsion at a given temperature and, thus, percent non-volatiles is actually being measured. The remainder of the emulsion is primarily water and since an emulsion is normally batched into concrete by total weight or volume (i.e., 24.5 gal/yd^3 - 121 l/m^3), the solids content must be controlled if the proper amount of polymer is to be added to the concrete. A slightly higher solids content than expected will provide more polymer and less water to the concrete. This will generally be slightly beneficial up to a solids content of about 53 to 55 percent. A solids content over 55 percent could adversely effect the viscosity and surface tension of the latex, causing unacceptable increases in both cases (7). However, because of the production processes used and the economics of emulsion production, a solids content above 55 percent would be very rare.

If the solids content is below an expected value, more water (and less polymer) will be inadvertently added to the concrete mix and, as shown in Table 10, the flexural strength of the concrete will be reduced and the permeability will be increased significantly; both undesirable effects. Compressive strength, on the other hand, will be relatively unaffected or might even increase slightly because the styrene-butadiene latex polymer has about the same adverse effect on compressive strength as additional water.

Because of the importance of solids content, it is recommended that it be monitored closely by the producer and the user. Job site sampling would be advisable.

Infrared Fingerprint of the Latex Solids and the Alcohol Soluble Portions

This "fingerprint" is actually two infrared spectra - one of the latex solids and the other of the alcohol soluble additives found in the latex. The additives are usually added to the latex to stabilize the emulsion, control cure rates and workability of the latex, control particle size of solids, and prevent coagulation of the latex (7). These spectra are compared with those of other batches of the same latex to be sure that the subject latex emulsion contains the same materials in about the same proportions as the original prequalified latex.

Weight Percent Butadiene in Latex Solids

The percent butadiene in the butadiene-styrene portion is a measure of the amount of solid butadiene in the latex polymer, and affects the compressive strength of the air-dried latex concrete. A latex with high butadiene content in the polymer will result in concrete of lower compressive strength than that of concrete made using a latex of low butadiene content, as the following results from Reference 9 indicate. However, very low butadiene contents will impart brittleness to the concrete (7).

Compressive strength versus butadiene level.

Charged butadiene level in percent	41	36	31
Compressive strength, psi			
after 7 days	3,320	3,925	4,370
after 28 days	4,215	4,370	5,020

Table 10. Effect of latex emulsion solids content on concrete[1] properties.

Concrete Property	Emulsion Solids Content, weight percent 20 to 23	46 to 48	55
Average 28-day Compressive Str., psi[2]			
a. Thermoflex 8002	3,240	3,325	3,080
b. Dow Modifier A	3,930	3,860	
Average 28-day Flexural Str., psi[2]			
a. Thermoflex 8002	593	738	731
b. Dow Modifier A	785	955	-
Chloride Permeability[3]			
Dow Modifier A Concrete			
Average Cl^- Ingress, lb/yd^3			
1/16- to 1/2-inch depth	13.09	8.07	-
1/2- to 1-inch depth	5.59	1.49	-

[1] All concretes contained 24.5 gallons of latex emulsion per cubic yard (121 $litres/m^3$). Because of the variable solids contents of the emulsions, the total water contents of the concretes varied significantly. The calculated total water/cement ratios were: 23 percent solids - 0.48 w/c; 46 percent solids - 0.41 w/c; 55 percent solids - .38 w/c. The air content of the Thermoflex 8002 concrete mix containing the 55 percent solids emulsion was higher than that for all other mixes (6.4 percent versus 4 to 5 percent). The coarse to fine aggregate ratio was 55:45 by volume in all mixes. This ratio and the use of a very angular coarse aggregate resulted in all strengths being lower than normal. All concrete for strength tests was cured 1 day wet burlap and 27 days lab air (72°F, 50 percent Rh). See Section II of this report for details on the chloride permeability test.

[2] To convert psi to MPa, multiply by 6.894×10^{-3}.

[3] To convert lb Cl^-/yd^3 to percent Cl^- by weight of concrete, multiply by 0.0255.

pH

This is a measure of the hydrogen ion concentration (acidity) of the latex. The pH of latex emulsions will vary from product to product because of the different additives used to stabilize the latices. Poor freeze-thaw and mechanical stability will result if the pH of a latex from particles stabilized solely by carboxyl groups falls to 9.0 or below, as the following results indicate (9, 11).

Effect of pH on freeze-thaw stability of latex stabilized with carboxyl groups.

Each freeze-thaw cycle consisted of 4 hours at 0°F followed by 4 hours at 70° ± 5°F (21 ± 3°C).

pH value	8.1	9.0	10.1	11.1	12.0
No. of cycles to cause coagulum	1	1	5+	5+	5+

However, latex particles stabilized with sulfonate groups will not display large changes in stability until a pH of 5.0 or less is obtained (11). Particles stabilized with anionic groups will show only small stability variations over pH's of 5 to 12 units (11). However, the pH of a specific latex emulsion should not vary over ± 1.0 pH unit from batch to batch.

Weight Percent Coagulum

This represents the percent of solids that has coagulated to produce particles larger than a specific size, normally 100 or 200 mesh (0.0059 or 0.0029 inches - 0.150 or 0.075 mm). A high percentage of coagulum is undesirable in that coagulated particles will not properly modify the cement phase of the concrete (7).

Viscosity

Viscosity is an indication of the resistance of a material to flow and, in the case of latex emulsions, it will vary with percent solids and particle size (11). Although a specific viscosity is not necessarily required for adequate dispersion of a well stabilized emulsion in concrete, viscosity is an excellent control to indicate whether or not two batches of the same emulsion are similar. Viscosities of a specific latex emulsion should be measured at a constant percent solids and should not vary significantly from batch to batch.

Surface Tension

In a latex emulsion, surface tension is the force opposing expansion of the surface area (breaking of the surface) (10). These forces must be overcome in concrete mixes in order that the cement and aggregate will be wetted by the latex emulsion. Water has a surface tension of 74 dynes/cm. The additives used in a latex emulsion to disperse the particles will lower the surface tension of the emulsion to values which are usually below 50 dynes/cm (7). Since surface tension is affected by the amount and type of additives in the latex, it is a good production control test. The surface tension should be measured at constant percent solids and should not vary more than ± 5 dynes/cm from batch to batch of a given latex emulsion.

Particle Size Distribution

Since latex particle size parameters influence the properties of concrete mixes containing latices, it is important to measure the mean size and the range of particle sizes. Generally, particles in the 1,900 to 2,500 Angstrom size range are more stable than larger or smaller particles. Smaller particles have more exposed particle surface area and thus larger amounts of additives are required to stabilize and disperse latices with smaller particle sizes. Also, concretes made with latices having particles of a small size may have low compressive strengths (8). Very large particles are equally undesirable in that the cement phase will not be properly modified. The particle size distribution in a latex emulsion should be unimodal to prevent heterogenous mixing with concrete and loss in concrete compressive strength, as the following data from Reference 9 illustrate.

Compressive strength versus particle size distribution mode.

Type of particle distribution and particle size, Angstroms	Unimodal 1,940	Bimodal 880 and 2,330
Compressive strength, psi		
After 7 days	3,235	2,650
After 28 days	4,140	3,445

Freeze-Thaw Stability

The freeze-thaw test is an accelerated stability test which defines the resistance of the latex to coagulation during two cycles of freezing and thawing at a rate of one-half cycle/day. Thus, the test indicates whether or not a latex emulsion can be subject to freezing temperatures prior to use in concrete. However, it should not be taken as a shelf-life stability test in that changes in additive effectiveness with time may not be accelerated and are not measured by the test.

Latex Emulsion Shelf Life

The shelf life of each latex emulsion was not measured directly in this study. However, studies performed for other purposes throughout the 1.5-year period did not identify any significant changes in chemical or physical properties of the emulsions, although fresh concrete tests did show a tendency toward higher air contents with some emulsions. Since concrete air content is controlled at the job site, it appears that a 1-year shelf life can be safely assumed although more data are needed in this area, especially with respect to the effect of non-freezing temperature cycling.

Test Results

Infrared Spectroscopy

In accordance with the latex manufacturers' suggestion, infrared spectroscopy was used in two ways during this study. The first was to fingerprint the latex emulsions and the second was to determine the butadiene/styrene ratio within the latex solids. Each of these applications is discussed in detail below. Although similarities do exist in the two uses, the differences must also be emphasized. The fingerprinting procedures involve the use of the entire infrared spectrum (4,000 to 600 cm^{-1}) to qualitatively obtain an indication of the composition of a sample. The infrared procedure is well suited to this type of analysis, provided the results are qualitatively interpreted. Use of infrared spectroscopy to determine the butadiene/styrene ratio, on the other hand, is designed to be a quantitative procedure which requires a reference curve and detailed quantitative study of a small region (1,000 to 650 cm^{-1}) of the infrared spectrum. The quantitative use of infrared spectroscopy is much more difficult and, thus, study of the accuracy of results was necessary.

All the infrared traces were obtained using the Perkin Elmer Grading Infrared Spectrophotometer shown in Figure 8. The spectrophotometer has the following capabilities: range 4,000 to 300 cm^{-1}, resolution = 0.3 cm^{-1}, frequency accuracy = 0.5 cm^{-1}, frequency repeatability = 0.25 cm^{-1}, readout - linear in wave numbers (cm^{-1}).

Many infrared spectrophotometers of differing sensitivities are in use today. Instruments with resolution and accuracy equal to or better than those given above are needed so that small peaks in latex spectra can be sharply defined. All infrared spectrophotometers sold today and most instruments now being used use linear wave numbers (frequency) as the abscissa readout. Thus, the spectra reported herein in linear wavenumbers can be readily and easily compared with spectra obtained from most other infrared instruments of equal resolution, frequency, accuracy, and repeatability.

1. Fingerprint of the Latex Emulsions

This study showed that two infrared traces, one on the latex solids and one on the alcohol solubles within the emulsion, would be required to adequately fingerprint each material.

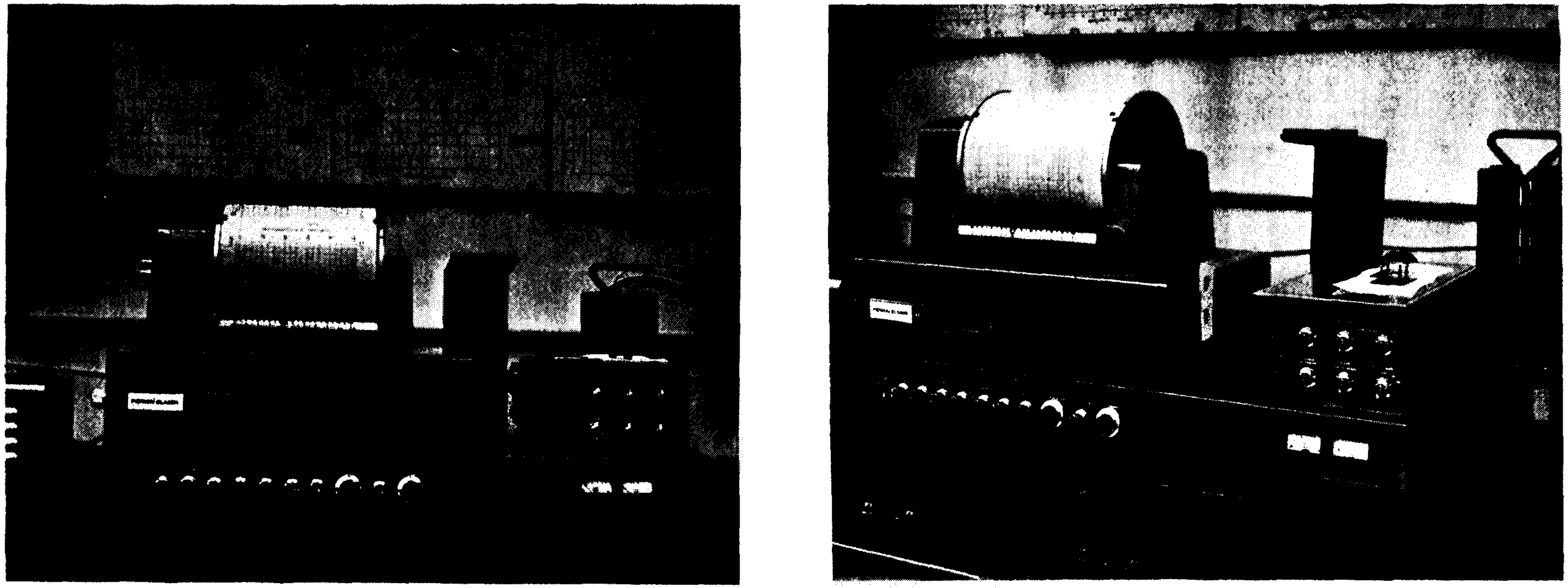


FIGURE 8. PERKIN-ELMER GRATING INFRARED SPECTROPHOTOMETER, MODEL 621.

(a) Fingerprint of the latex solids

A variety of procedures were studied for making a latex film for the solids fingerprint. The best procedure identified is given in detail in paragraph 3.2.1.1 of the Prequalification Program in Section VI of this report. In short, the latex is placed on a microslide between spacers of known thickness (see Figure 22, Section 3.2.1.1) and a glass rod is drawn over the slide and liquid latex. The coated slide is then allowed to dry and the latex film is removed by immersing the slide in hot water and carefully removing the film already loosened by the water. Films of good quality generally resulted when this technique was used. In a few instances, it was necessary to resort to techniques such as acid cleaning of the microslides prior to latex placement, the application of thicker films and subsequent use of a razor blade to aid removal, and/or heating of the microslides after coating, in order to produce acceptable films. In any event, film thickness must be controlled to meet the infrared peak criteria in Section 3.2.2 of the Prequalification Program. Films of approximately 0.08-mm thickness are recommended because they give the most detailed infrared spectra without peak distortion. Films which are too thick result in spectra containing major peaks with smaller amplitudes and areas than in actuality, relative to the other peaks in the spectra; while films that are too thin will yield spectra in which the smaller peaks are missing and thus the spectra will lack the detail necessary for a thorough and meaningful comparison between spectra. Strict adherence to the transmittance limits given in Section 3.2.2 of the Prequalification Program should minimize thickness complications.

The infrared spectra (i.e., fingerprints) of films of the latex solids for each of the five latices studied herein are shown in Figure 9. The actual infrared traces are much larger than those shown, and copies are on file at Fairbank Highway Research Station. The spectrum shown in Figure 9E is that for the Thermoflex 8002 latex emulsion originally submitted by Reichhold Chemicals, Inc. Figure 9F is the film spectrum for a sample from a batch of Thermoflex 8002 recently produced by Reichhold. Suggested procedures for comparing the spectra are given in item (c) below.

(b) Fingerprint of the alcohol solubles in the emulsion

Because additives present in any latex emulsion are primary determinants of quality and compatibility with portland cement concrete, a fingerprint of the additives is essential. This fingerprint must be determined

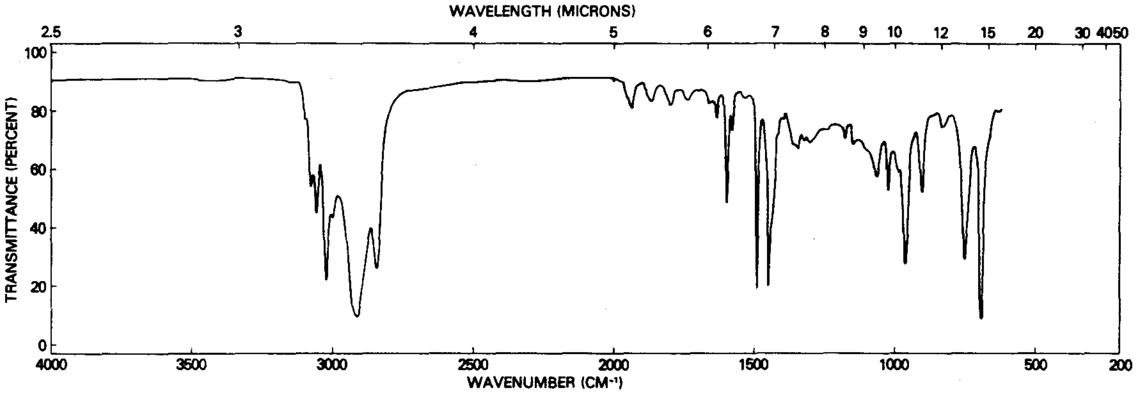


FIGURE 9A. DOW MODIFIER A

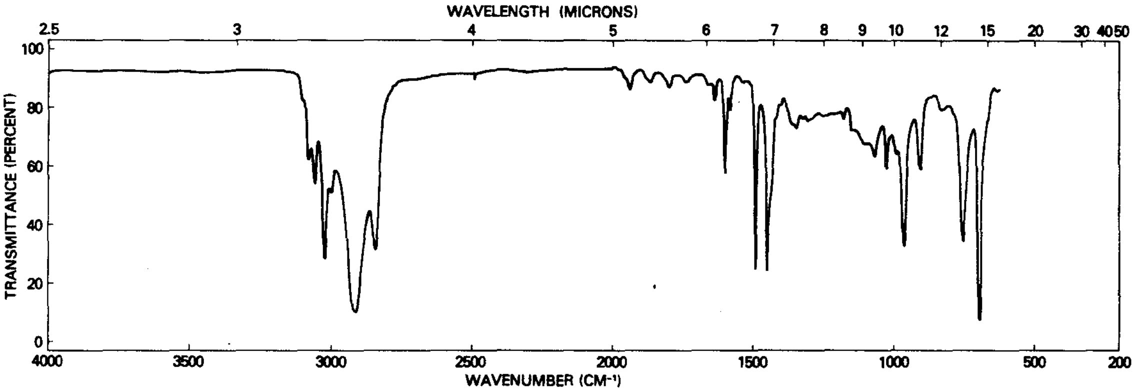


FIGURE 9B. DECO REZ 4776

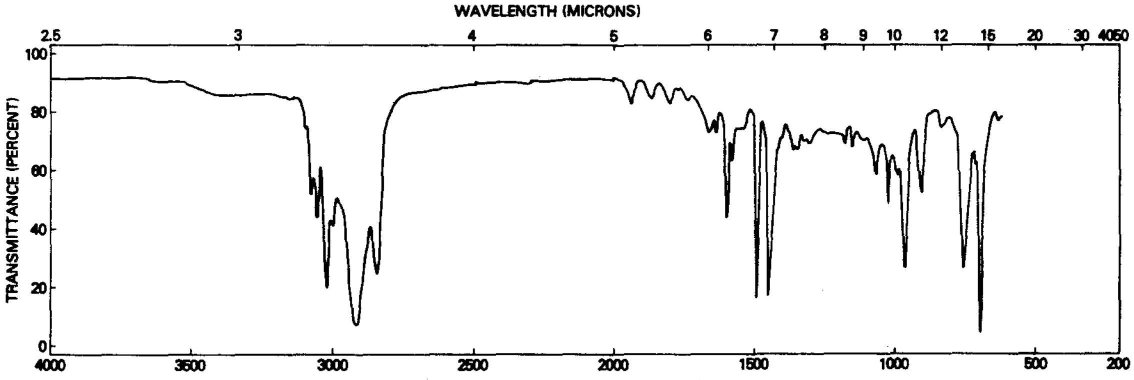


FIGURE 9C. ARCO DYLEX 1184

FIGURE 9. INFRARED SPECTRA OF LATEX FILMS.

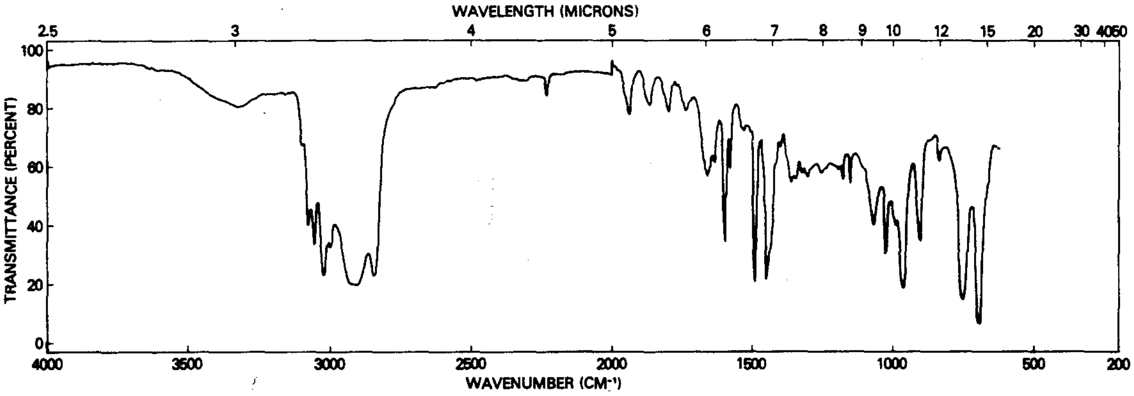


FIGURE 9D. ARCO DYLEX 1186

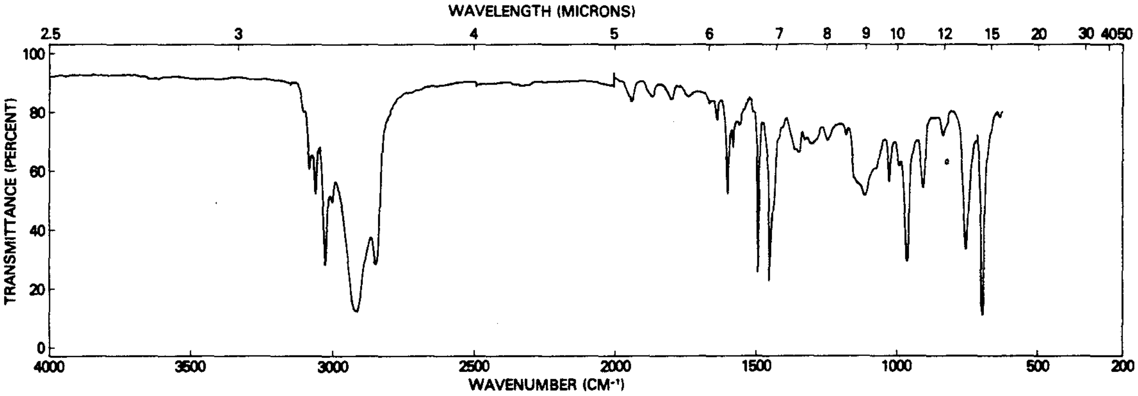


FIGURE 9E. THERMOFLEX 8002.

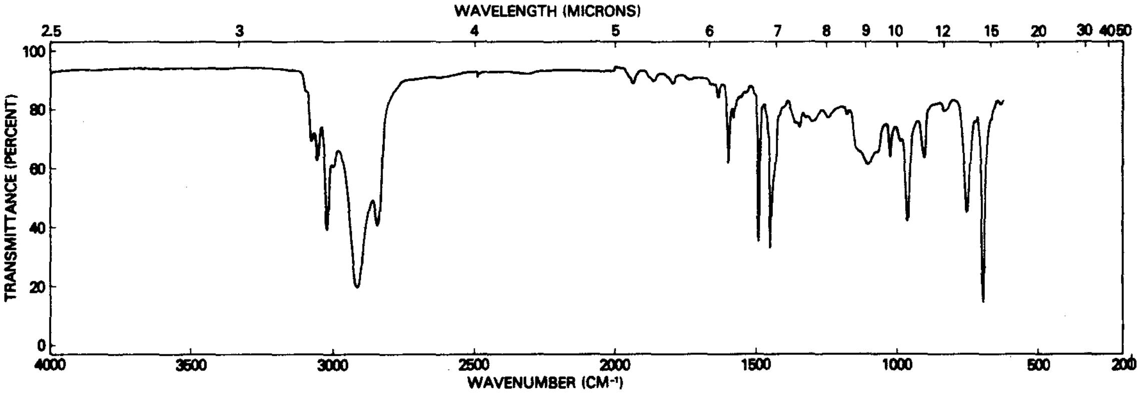


FIGURE 9F. THERMOFLEX 8002.

FIGURE 9 (Cont.). INFRARED SPECTRA OF LATEX FILMS.

separately from that of the latex film since some additives are volatile and all are present in only small quantities. The alcohol-soluble fingerprint was obtained by adding latex emulsion to isopropanol while stirring, filtering the mixture, and then recovering the alcohol solubles from the liquid by drying. The procedure is detailed in Section 3.3.1 of the Prequalification Program. The infrared spectra of the alcohol solubles were then obtained by placing the residue between sodium chloride infrared plates as described in Section 3.3.2. As in the other infrared studies, residue thickness will affect peak area and amplitude and must be carefully controlled. Figures 10A through 10E show the infrared fingerprints of the alcohol solubles for each of the five latex emulsions investigated herein. Note that there are significant differences in the various alcohol soluble spectra which indicate that the various manufacturers are using different additives. The spectra indicate that Dow-Modifier A and Deco-Rez 4776 are the two most closely related materials.

Figure 10F is the fingerprint of alcohol solubles contained in a recently produced batch of Arco Dylex 1186 emulsion. Comparison of this spectrum with that of the original Arco Dylex 1186 sample (Figure 10E) is discussed below.

(c) Comparison of fingerprints

Comparison of the infrared spectra obtained from two different batches of the same latex is a qualitative procedure, and as such, minor differences in spectra are often evident solely as a result of infrared spectroscopy procedures. However, if major differences are obvious and the spectra were obtained in strict adherence to the transmittance criteria in Section 3 of the Prequalification Program, differences in the two latices are probable. The following three steps should be followed when comparing spectra:

(1) Compare the spectra to define their general similarities. The same number of peaks of the same general shape should be present.

(2) Compare the frequencies of the tips of the peaks. Each peak frequency in the subject fingerprint should not vary by more than ± 20 cm^{-1} from the frequency of the corresponding peak of the prequalified fingerprint. This comparison is the strong point of the infrared fingerprinting process. The frequencies of all corresponding peaks should be essentially identical as sample thickness variations

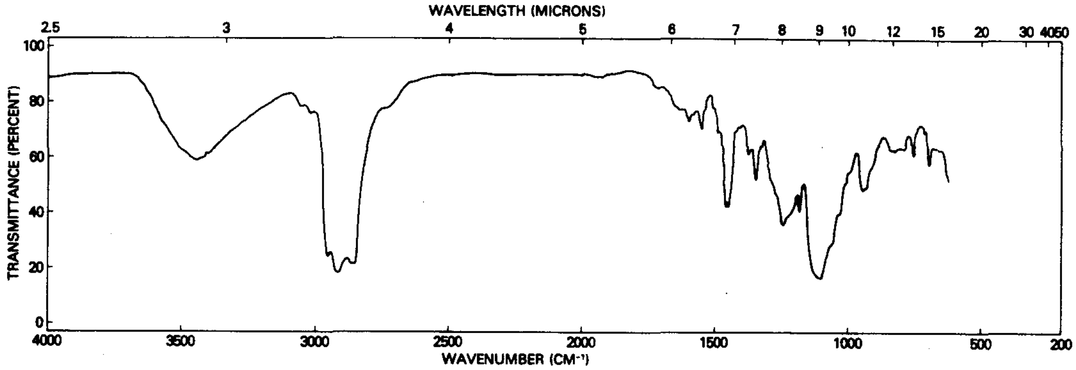


FIGURE 10A. DOW MODIFIER A

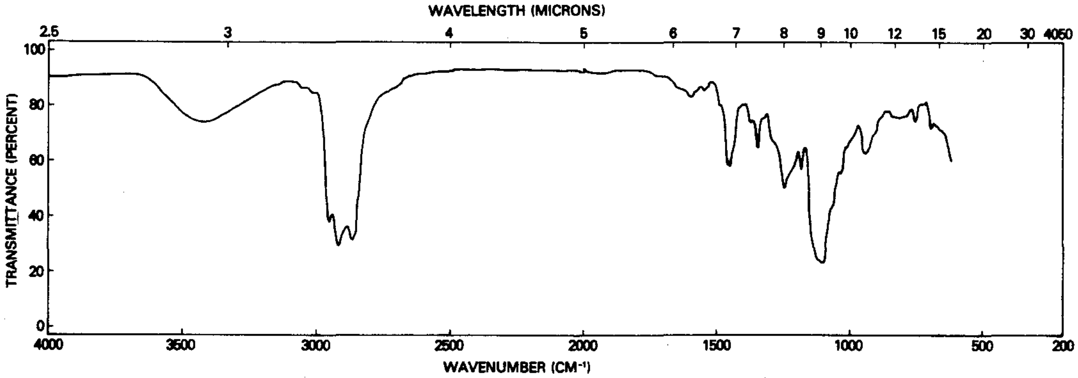


FIGURE 10B. DECO REZ 4776

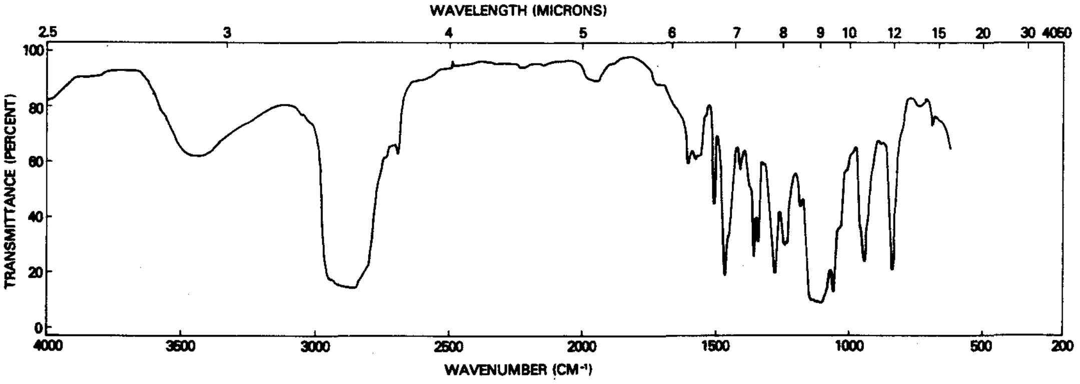


FIGURE 10C. THERMOFLEX 8002

FIGURE 10. INFRARED SPECTRA OF ALCOHOL SOLUBLE PORTIONS OF LATICES.

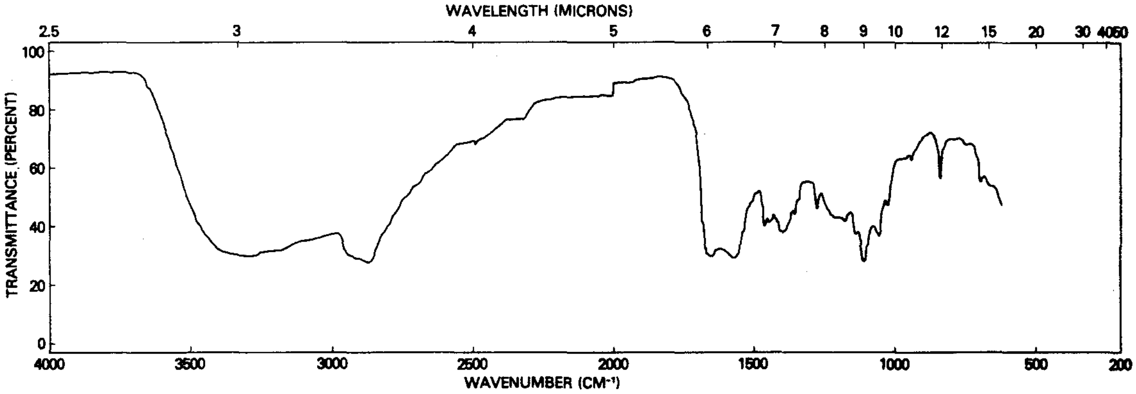


FIGURE 10D. ARCO DYLEX 1184.

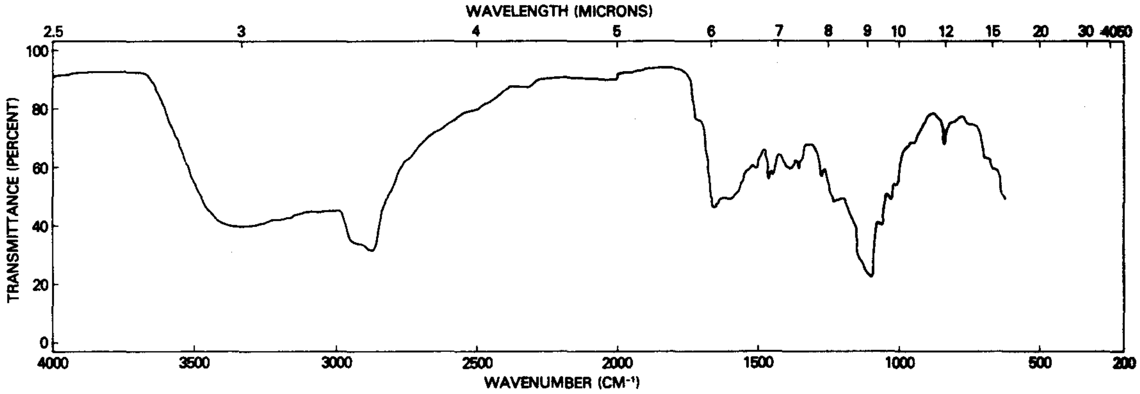


FIGURE 10E. ARCO DYLEX 1186

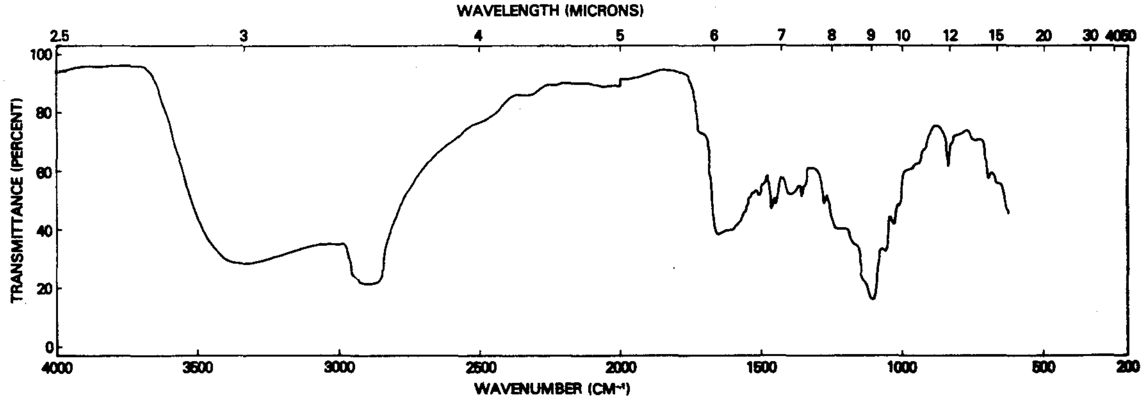


FIGURE 10F. ARCO DYLEX 1186

FIGURE 10 (Cont.). INFRARED SPECTRA OF ALCOHOL SOLUBLE PORTIONS OF LATICES.

within the limits permitted by the transmittance criteria in Sections 3.2.2 and 3.3.2 will not significantly affect the frequencies.

(3) Compare the corresponding peak heights of both spectra. If they are qualitatively equal, this is positive evidence that the materials are similar. However, if a few of the minor peaks are reversed in relative height, this may be due to sample thickness variations and/or test variation rather than actual differences between the two samples.

As examples of the comparison process, the spectra from the two Thermoflex 8002 latex films, given in Figures 9E and 9F, and the spectra of the two Arco 1186 alcohol soluble residues, given in Figures 10E and 10F, are compared below:

(1) Comparison of spectra of latex films (Figures 9E and 9F)

Figures 9E and 9F have the same peak positions and the same major peak shape in all instances. The frequencies of all major peaks in Figures 9E and 9F are equal. Also, the peaks in the groups at 775, 910, 965, 1450, 1490; those at 905, 1030, 1100, and 1600; and those at 1240, 1300, 1340, 1550, 1580 cm^{-1} have the same relative heights. Thus, it is concluded that the two spectra are essentially identical and the two batches of Thermoflex 8002 have essentially the same latex film composition.

(2) Comparison of spectra of emulsion alcohol solubles (Figures 10E and 10F)

Figures 10E and 10F have the same peak positions and the same major peak shape in all instances. The frequency of each major peak in Figure 10E is equal to the frequency of the corresponding peak in Figure 10F. Although the peaks at frequencies of 3400 and 1100 cm^{-1} and 1505 and 1350 cm^{-1} have the same general shape and positions in both figures, the shape of the two peaks at 2870 and 1650 cm^{-1} show minor differences. Similarly, the relative heights of the peaks from 1100 to 1000 cm^{-1} are the same, whereas the peaks at 690 and 1505 cm^{-1} show minor relative height differences. In all cases, however, these minor differences are undoubtedly due to variations in sample thickness within the limits in Section 3.3.2, and thus the spectra are essentially identical.

Additional insight in the area of fingerprint interpretation can be obtained by comparing Figure 10A to 10B (Dow Modifier A to Deco-Rez 4776) and 10D to 10E (Arco 1184 to Arco 1186). In Figures 10A and 10B, the same peaks are present in each spectrum and all corresponding peaks have the same general shape (Criterion 1). Further, the frequencies of the tips of all corresponding peaks are essentially identical (Criterion 2). However, there are variations in the relative heights and exact shape of the corresponding peaks at 3450 and 1600 to 1550 cm^{-1} (Criterion 3). Because Criteria 1 and 2 are met, these two spectra must be judged essentially identical for product certification purposes. Further study of additional spectra would be necessary to obtain insight into whether or not the differences under Criterion 3 were real or caused solely by film thickness and test variations.

A comparison of Figure 10D to Figure 10E indicates the following:

> Criteria 1 and 2--Peaks at 3400, 2870, 2350, and 1650 cm^{-1} are present in both spectra and have the same general shape. However, in the frequency range of 1650 to 700 cm^{-1} there are significant differences in number of peaks, general peak shape, and the frequency of peaks. For example, a large peak, present at 1500 to 1300 cm^{-1} in Figure 10D, is not prominent in Figure 10E, and the general shapes of the peaks from 1300 to 900 cm^{-1} are significantly different in the two figures.

Therefore, these major differences identified when Criteria 1 and 2 are applied are grounds for questioning the similarity of the two emulsions.

2. Determination of the Butadiene/Styrene Ratio in a Latex Emulsion

Determination of the butadiene/styrene ratio within a latex emulsion is commonly done in the latex industry using quantitative infrared spectroscopy. Study of the various procedures used by manufacturers indicated wide variations in both film preparation and interpretation procedures. All the manufacturers followed the general procedures of preparing a reference graph of butadiene content versus infrared spectra (butadiene to styrene) peak ratio and, after determining the B/S ratio of an unknown, using the graph to determine the butadiene content of the unknown. However, requirements for film preparation and thickness, for sample holders and calculation procedures, varied widely. Since the reference curve is the key to accurate determinations, the effect of the various methods on curve positioning and confidence limits was studied in detail. The reference samples of known butadiene content were supplied by Reichhold Chemicals, Inc., and the procedure for sample preparation is given in Appendix A. In all, eight methods for curve preparation primarily involving differences in sample preparation and calculation procedures were studied:

(A) Latex films between sodium chloride plates

The procedures in Section 3.3.1.1 of the Prequalification Program were used to prepare films which were subsequently sandwiched between sodium chloride plates, and the infrared spectra were run.

(B) Latex films formed directly on sodium chloride plates

A circular wire loop (approximately 1 mm thick) was dipped into the latex sample to form a latex liquid film within the loop. The latex loop was then positioned such that the liquid film touched the flat side of a sodium chloride plate and left a film adhering to the plate. The film was dried at room temperature or with the aid of an infrared heating lamp until all tackiness disappeared. The film on the NaCl plate was then subjected to infrared analysis.

Wire loops of two sizes were used with this technique: (1) a 20-mm diameter loop was used to form 15- to 20-mm diameter films on 25-mm diameter NaCl plates; (2) a 35-mm diameter loop was used to form 25-mm diameter films on 25-mm diameter NaCl plates. Because of the two different film diameters, the data for each diameter were analyzed separately.

(C) Latex films formed directly on silver bromide plates

The latex sample was applied directly to a silver bromide plate with a spatula and spread until the coating was smooth. The coating was dried as described in procedure (B) above and the film on the AgBr plate was then subjected to infrared spectroscopy.

(D) IR beam passing only through the film

In this technique, films were formed using the procedures described for the fingerprinting films but with a larger spacer thickness (0.12 mm). The films were then placed on cardboard cell holders and the IR spectra were obtained while the infrared beam passed only through the latex film.

Two calculation methods, each coupled to specific transmittance criteria, were used to define the reference curve:

(a) Transmittance criteria for spectra from 1000 to 650 cm^{-1}

The distance between the tip of peak at 970 cm^{-1} and the base line of the spectrum shall be greater than 50 percent transmittance; and the tip of the peak at 970 cm^{-1} shall be greater than 10 percent transmittance.

Calculation method - Use the 970 and 760 cm^{-1} peaks and the method shown in Figure 23.

(b) Transmittance criteria for spectra from 1000 to 650 cm^{-1}

See Section 4.A.1.1 of the Prequalification Program, Section VI of this report.

Calculation method - Use the 910 and 760 cm^{-1} peaks and the method shown in Figure 23.

By comparing the results of Methods A to E above, it was found that Method (A), sandwiching a fingerprinting-type film between two sodium chloride plates, could be eliminated from consideration because the amplitudes of the styrene and butadiene peaks were too small. The films were too thin and thus the peaks were too small for accurate height measurement, and quantitative interpretation of the results was virtually impossible.

The remaining film preparation procedures (B through D), where coupled with the two calculation procedures and the following seven procedures, were studied in detail:

B1-a - Wire loop, small sample on NaCl plate, 970 cm^{-1} butadiene peak.

B1-b - Wire loop, small sample on NaCl plate, 910 cm^{-1} butadiene peak.

B2-a - Wire loop, large sample on NaCl plate, 970 cm^{-1} butadiene peak.

B2-b - Wire loop, large sample on NaCl plate, 910 cm^{-1} butadiene peak.

C-b - Film on AgBr plate, 910 cm^{-1} butadiene peak.

D-a - IR beam passing only through film, 970 cm^{-1} butadiene peak.

D-b - IR beam passing only through film, 910 cm^{-1} butadiene peak.

Figure 11 shows the reference curves which were obtained with the various procedures. The best-fit reference line is in each instance a straight line of the form $\log y = a + bx$, where y is the butadiene/styrene ratio and x is the percent butadiene in the latex. Obviously, the reference line is dependent on test method in that no two methods resulted in equal best-fit regression lines. However, procedures B2-b, C-b, and D-b did result in very similar reference lines, especially in the 30- to 40-percent butadiene content range,

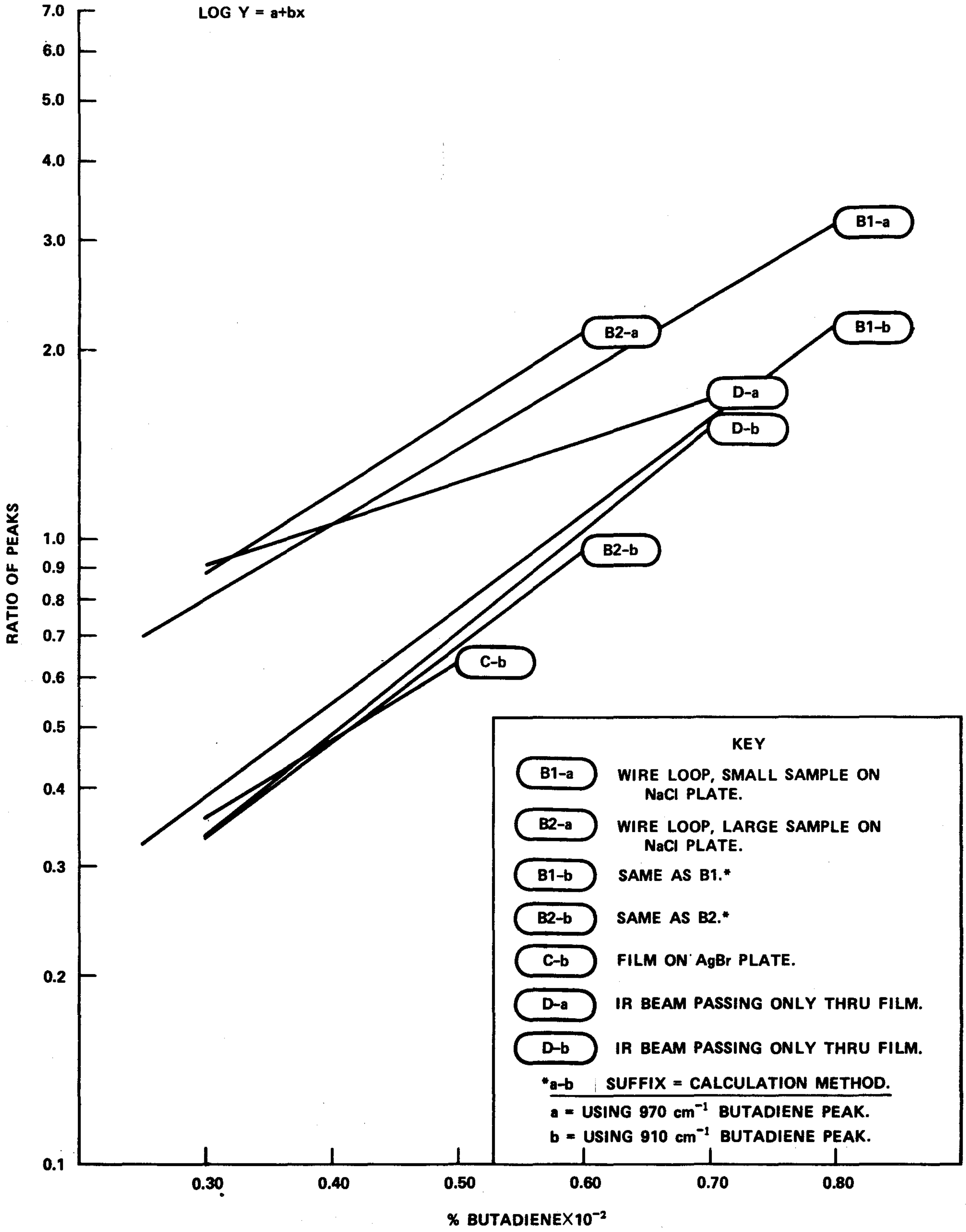


FIGURE 11. COMPARISON OF THE REFERENCE CURVES OBTAINED BY DIFFERENT INFRARED TECHNIQUES.

the area of interest herein. The seven reference lines with 95 percent confidence limits, calculated in accordance with the W_1 procedures given in Reference 12, are shown in Figures 12 through 15. The confidence limits indicate that the best film preparation procedures are (1) procedure B2, wire loop, large sample on NaCl plate, and (2) procedure D, IR beam passing only through the film. The limits also indicate that the best calculation procedure is procedure b, 910 cm^{-1} butadiene peak using the transmittance limits given in Section 4.A.1 of the Prequalification Program. As a result, procedures B2-b and D-b are presented as alternates in the Prequalification Program and procedure D-b was used in this study to yield the following results (Table 11) for the five emulsions of unknown butadiene content.

Table 11. Percent butadiene in the latex emulsions.

Emulsion	Number of Films	Peak Ratio Average	Peak Ratio Standard Deviation[2]	Ave.[1] Percent Butadiene
Dow Modifier A	9	0.469	0.02730	39.1
Deco-Rez 4776	7	0.424	0.01809	36.4
Thermoflex 8002	7	0.429	0.01718	36.7
Arco Dylex 1184	5	0.401	0.02103	34.9
Arco Dylex 1186	6	0.365	0.00813	32.4

[1] Determined using the reference curve in Figure 15.

[2] Average standard deviation = 0.018346; standard deviation of the mean = $0.018346/\sqrt{7} = 0.006934$; t_{95} for 34 samples = 2.033. These figures were used in the accuracy calculations discussed below.

The accuracy of the above average percent butadiene values can be estimated by considering both the 95 percent confidence limits of the reference curve and the mean standard deviation of the average peak ratio for an unknown sample, in accordance with the procedures described on pages 36 to 38 of Reference 13. The result for Procedure D-b is a 90-percent confidence range of ± 2.9 percent butadiene. The requirement in the Prequalification Program that six samples per butadiene content be used to formulate the reference curve, rather than the three used herein, should reduce the above confidence range. Procedure B2-b (wire loop) exhibited slightly narrower 95 percent confidence limits for the reference curve, and thus, a similar analysis using the same mean standard deviation of peak ratios resulted in a 90-percent confidence range of ± 2.1 percent butadiene. These confidence ranges were considered in defining the 30 to 40 percent butadiene limits given in the prequalification and certification programs.

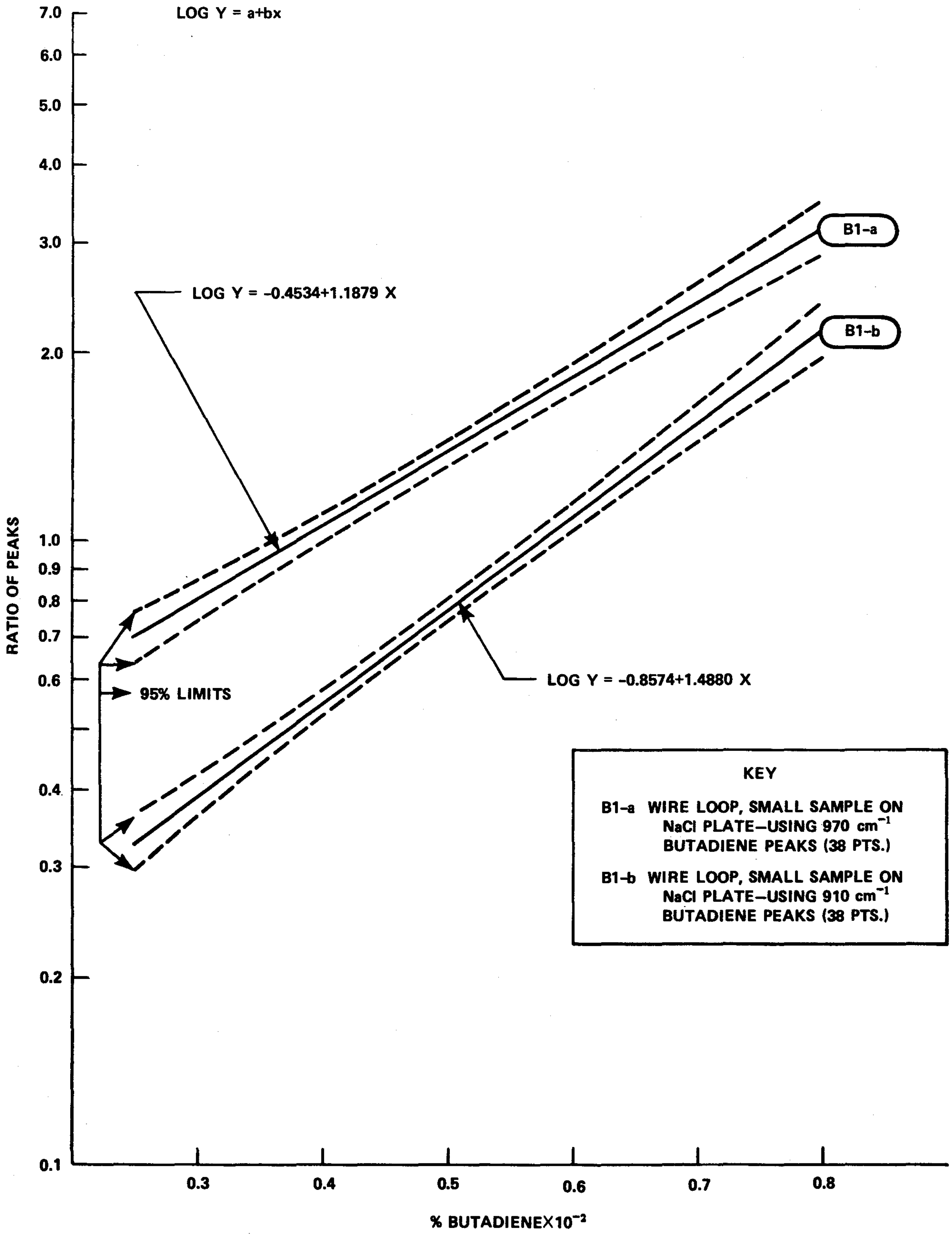


FIGURE 12. REFERENCE CURVES AND CONFIDENCE LIMITS FOR PROCEDURE B1.

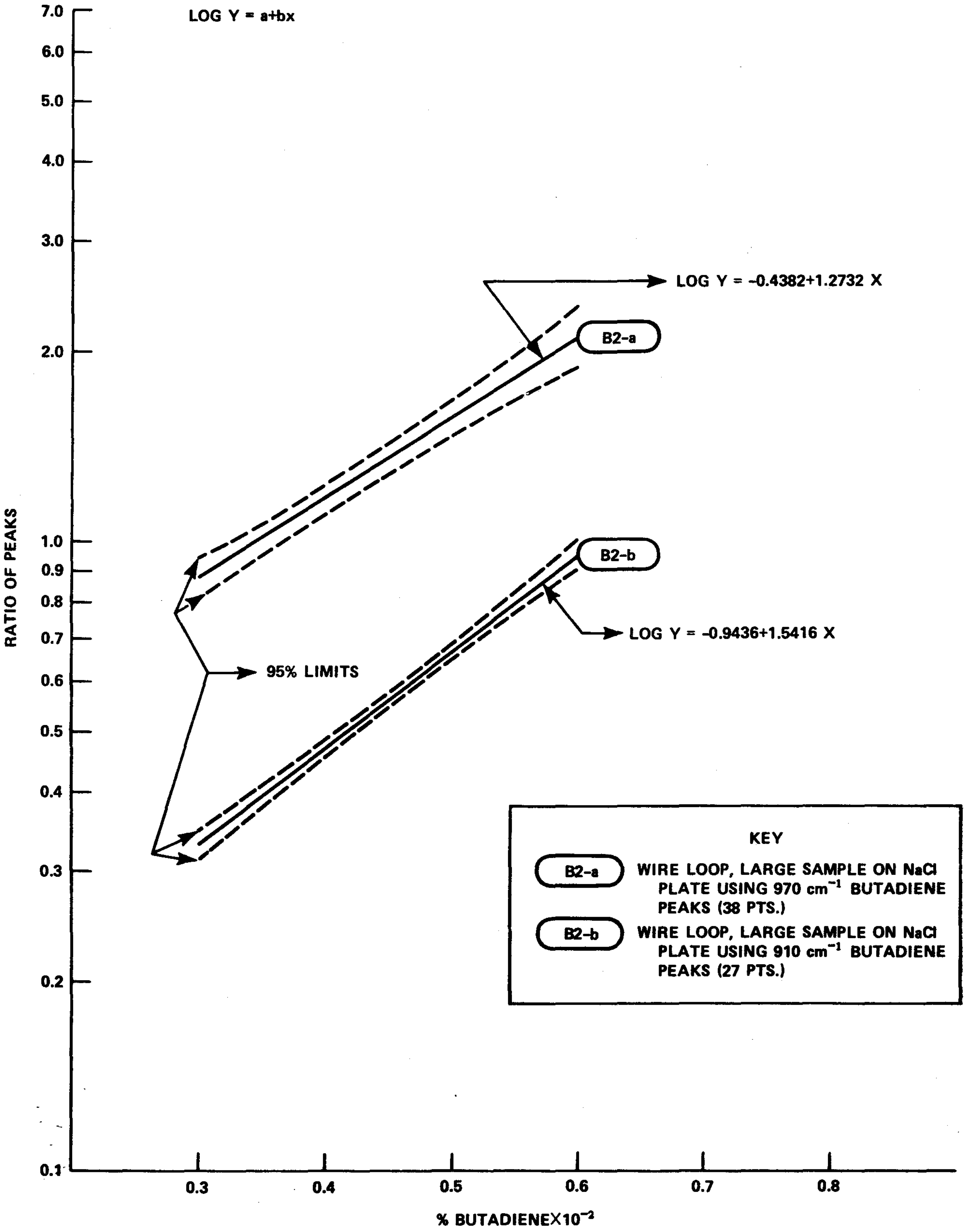


FIGURE 13. REFERENCE CURVES AND CONFIDENCE LIMITS FOR PROCEDURE B2.

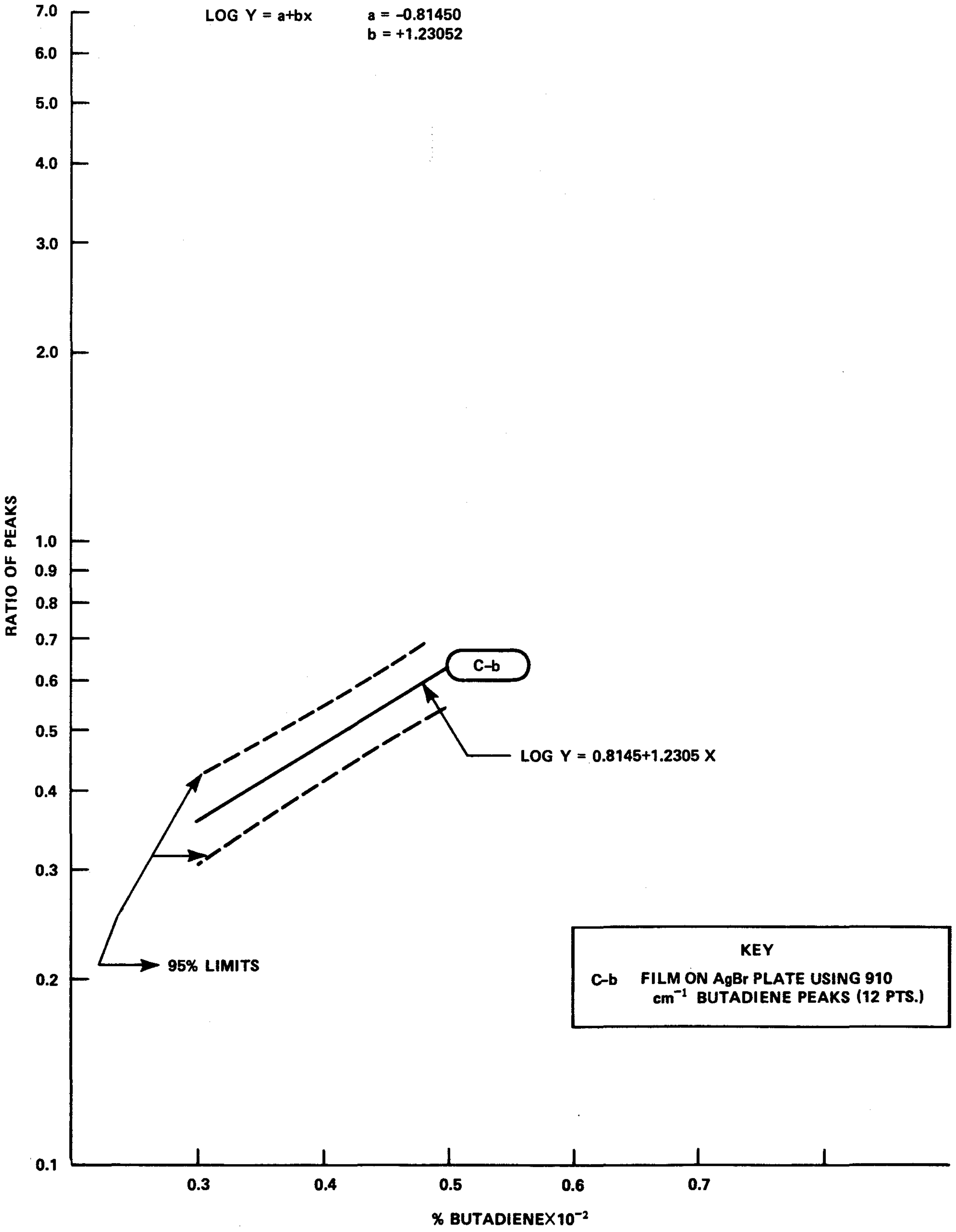


FIGURE 14. REFERENCE CURVE AND CONFIDENCE LIMITS FOR PROCEDURE C.

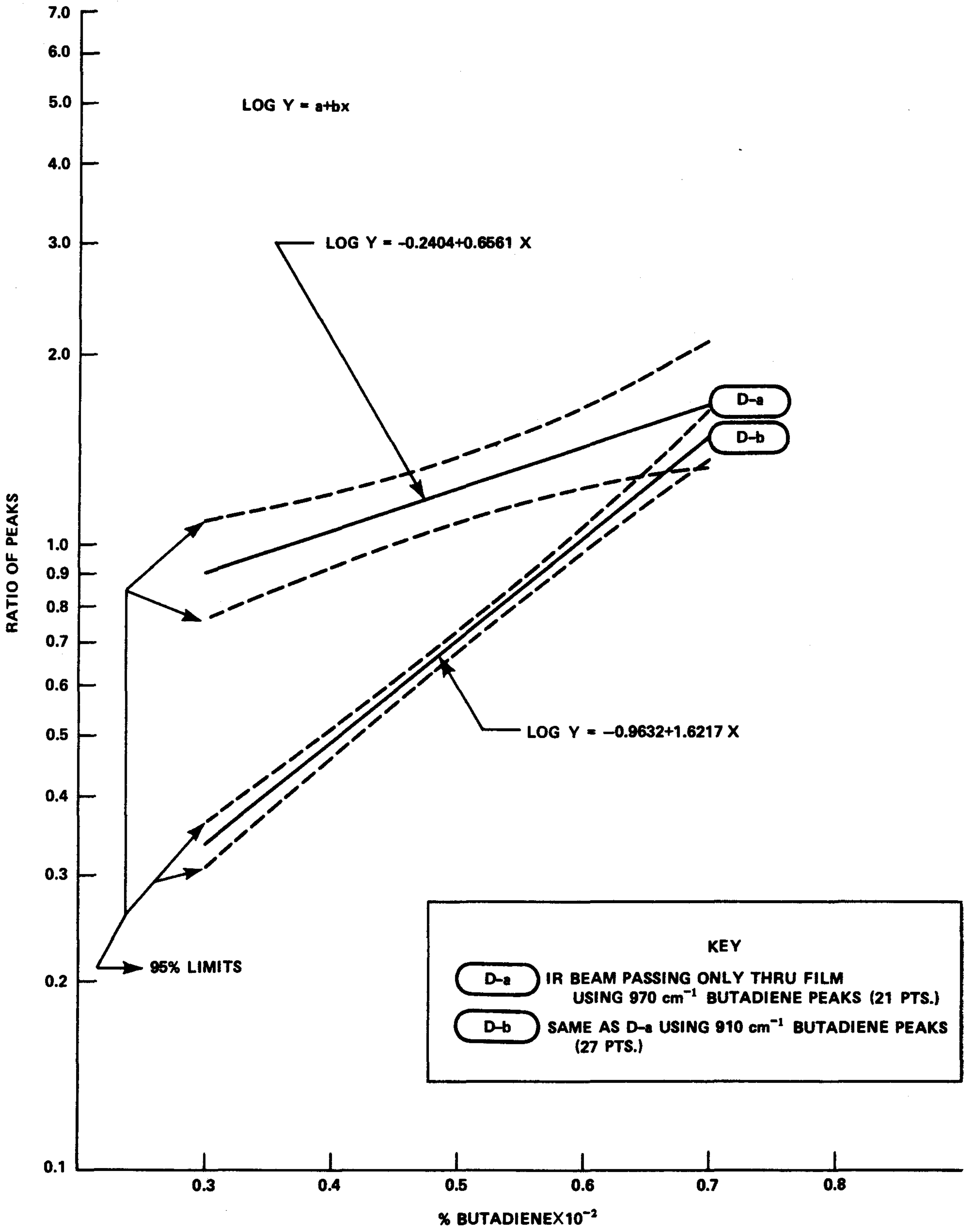


FIGURE 15. REFERENCE CURVES AND CONFIDENCE LIMITS FOR PROCEDURE D.

Weight Percent Solids

Samples of each latex were heated at 120°C for 2 hours to remove the water and volatile materials from the latex. The residue was weighed, and the percent solids were calculated following the procedure in Section 4.A.2. The results are as follows:

Sample	Weight Percent Solids
1. Dow Modifier A	47.7
2. Deco-Rez 4776	47.3
3. Thermoflex 8002	52.6
4. Dylex 1184	51.4
5. Dylex 1186	51.5

The latex from both Dylex 1184 and Dylex 1186 darkened upon heating, while the others did not discolor. The percent solids in Thermoflex 8002 and Dylex 1184 and 1186 were higher than that of the other emulsions but still within specifications. Other batches of these two latices, submitted recently, contained between 47 and 49 percent solids. The solids content of each latex was adjusted to 46 percent by the addition of water (see Section 4.A.3) for use both in the concrete tests (Section II) and for the remaining latex tests in this section.

pH

The pH was determined with each latex at 25°C, using a standard pH meter and a glass electrode. The results are as follows:

Sample	pH
1. Dow Modifier A	10.4
2. Deco-Rez 4776	10.3
3. Thermoflex 8002	10.6
4. Dylex 1184	10.0
5. Dylex 1186	9.5

The pH of Dylex 1186 is lower than the other latices but within specification limits. This is due mainly to the production process the manufacturer uses in emulsion formulation and the type of additives used to stabilize the emulsion. Arco Dylex 1186 does not contain carboxylates as stabilizers and thus, the material should be stable at the lower pH (11).

Weight Percent Coagulum

This was obtained by filtering a measured amount of latex through a 200 mesh screen, measuring the amount of latex retained on the screen, and calculating the percent coagulation using the percent solids content in the latex (see Section 4.A.5). The procedure was also run using a 100 mesh screen. The results are as follows:

Sample	Weight Percent Coagulum Retained 200 mesh screen	 100 mesh screen
1. Dow Modifier A	0.021	0.014
2. Deco-Rez 4776	0.041	0.022
3. Thermoflex 8002	0.130	0.032
4. Dylex 1184	0.070	0.032
5. Dylex 1186	0.171	0.043

Dylex 1186 and Thermoflex 8002 have more coagulum than the other materials. However, this did not adversely affect the physical properties of the concretes containing these materials. For this reason, a 100 mesh screen is specified for use in the Prequalification and Certification Programs.

Viscosity

The viscosities of the latices were measured at 25°C at two different shear rates (velocities) using a Brookfield Viscometer. The results are as follows:

Sample		Viscosity, centipoises 10 rpm	 20 rpm
1. Dow Modifier A	(47.7 percent solids)	26	30
	(46.0 percent solids)	20	23
2. Deco-Rez 4776	(47.3 percent solids)	66	59
	(46.0 percent solids)	38	39
3. Thermoflex 8002	(52 percent solids)	57	58
	(46 percent solids)	26	29
4. Dylex 1184	(51 percent solids)	185	153
	(46 percent solids)	37	37
5. Dylex 1186	(51 percent solids)	70	66
	(46 percent solids)	26	28

Two different velocities were used to measure any possible shear dependent properties of the latices. All the latices showed some shear rate susceptibility. The Arco 1184 material showed significant susceptibility at 51 percent solids and exhibited a very high viscosity at that solids content.

Surface Tension

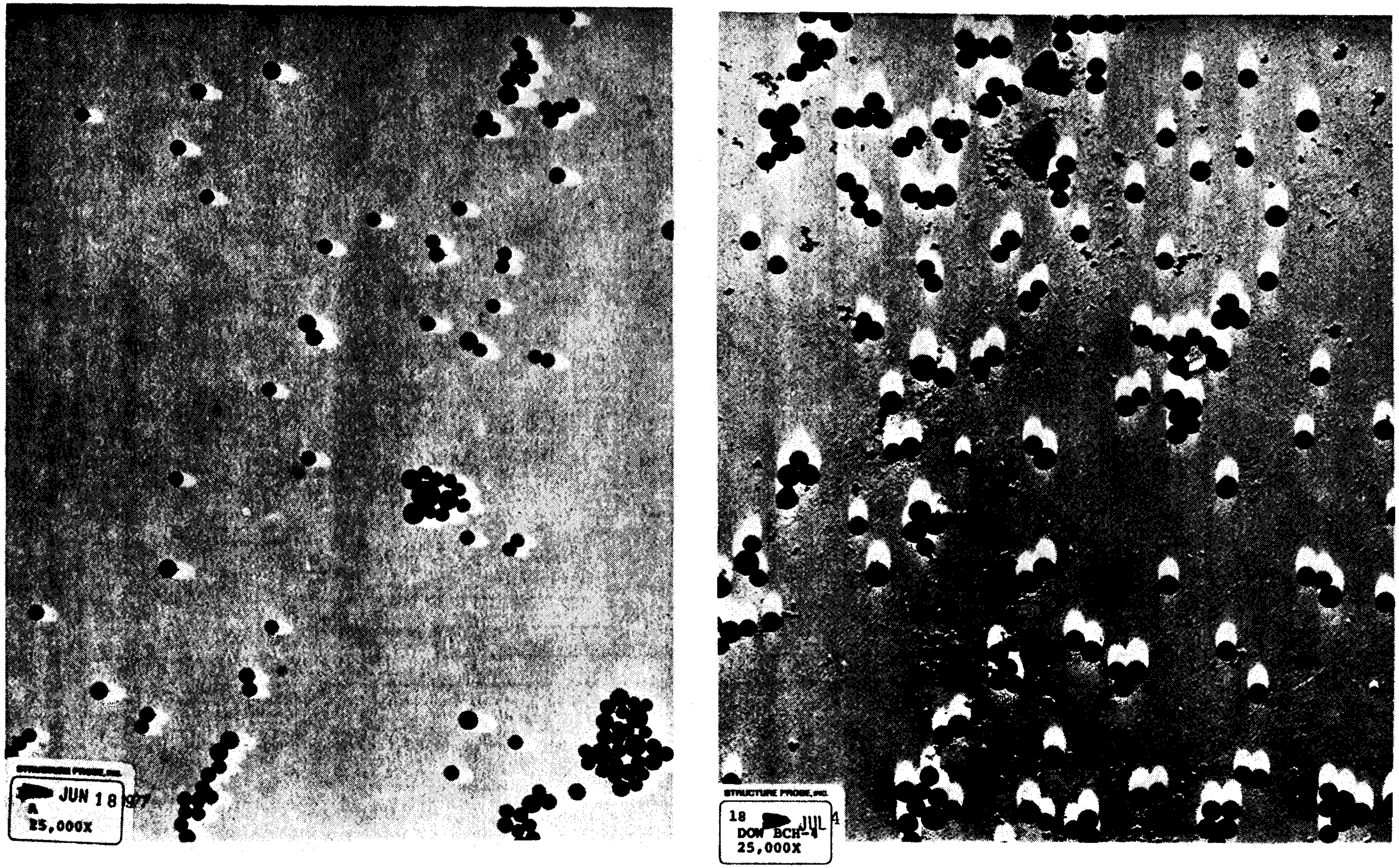
The surface tension of each latex (at 46.0 percent solids) was determined using a Cenco Tensiometer and the procedure described in Section 4.A.8 of the Prequalification Program. The results are as follows:

	Sample	Surface Tension (dynes/cm)
1.	Dow Modifier A	26.4
2.	Deco-Rez 4776	25.8
3.	Thermoflex 8002	39.7
4.	Dylex 1184	44.8
5.	Dylex 1186	42.2

The surface tensions of all the latices are within the prequalification acceptance criterion (Section 4.A.8.6).

Particle Size Distribution

Particle size determinations were conducted for the latices using a scanning electron microscope operated in the transmission mode (see Section 4.A.9 of the Prequalification Program). Latex particles were hardened by an osmium tetroxide reaction technique. Bromine vapor may also be used to harden the latex particles. Photographs were taken of the hardened and "shadowed" particles at 25,000 times magnification (see Figure 16). Histograms were developed from the particle size data; these histograms, representing frequency distributions of the particle sizes in each sample, are in Figures 17 through 21. The median particle size and the 95-percent range of particle sizes were calculated from these histograms. The mean and standard deviation of particle size were also calculated assuming normal particle size distribution, and the results are given in Table 12. The difference between the mean and median particle sizes indicates deviation from a normal distribution. For most of latices, particles normally do not occur in normal distributions and their actual distributions are skewed (14). Thermoflex 8002 and Dylex 1184 latex particle distributions are skewed more than the distributions for the other latices in that the histograms of both of these latices indicate a disproportionate number of particles of one particle size. Both the standard deviation and the 95 percent range indicate the normal spread (dispersion) of the particle sizes. Particle sizes of Dylex 1184 had a larger spread than those of the other latices, and the 95 percent range was outside the specification limits given in the acceptance criteria in Section 4.A.9.4 of the Prequalification Program. The other latices have approximately the same particle size spreads and all are within the specification limits. All the particle size distributions are unimodal.



THERMOFLEX 8002

DOW MODIFIER A

FIGURE 16. LATEX PARTICLE SIZE PHOTOGRAPHS.

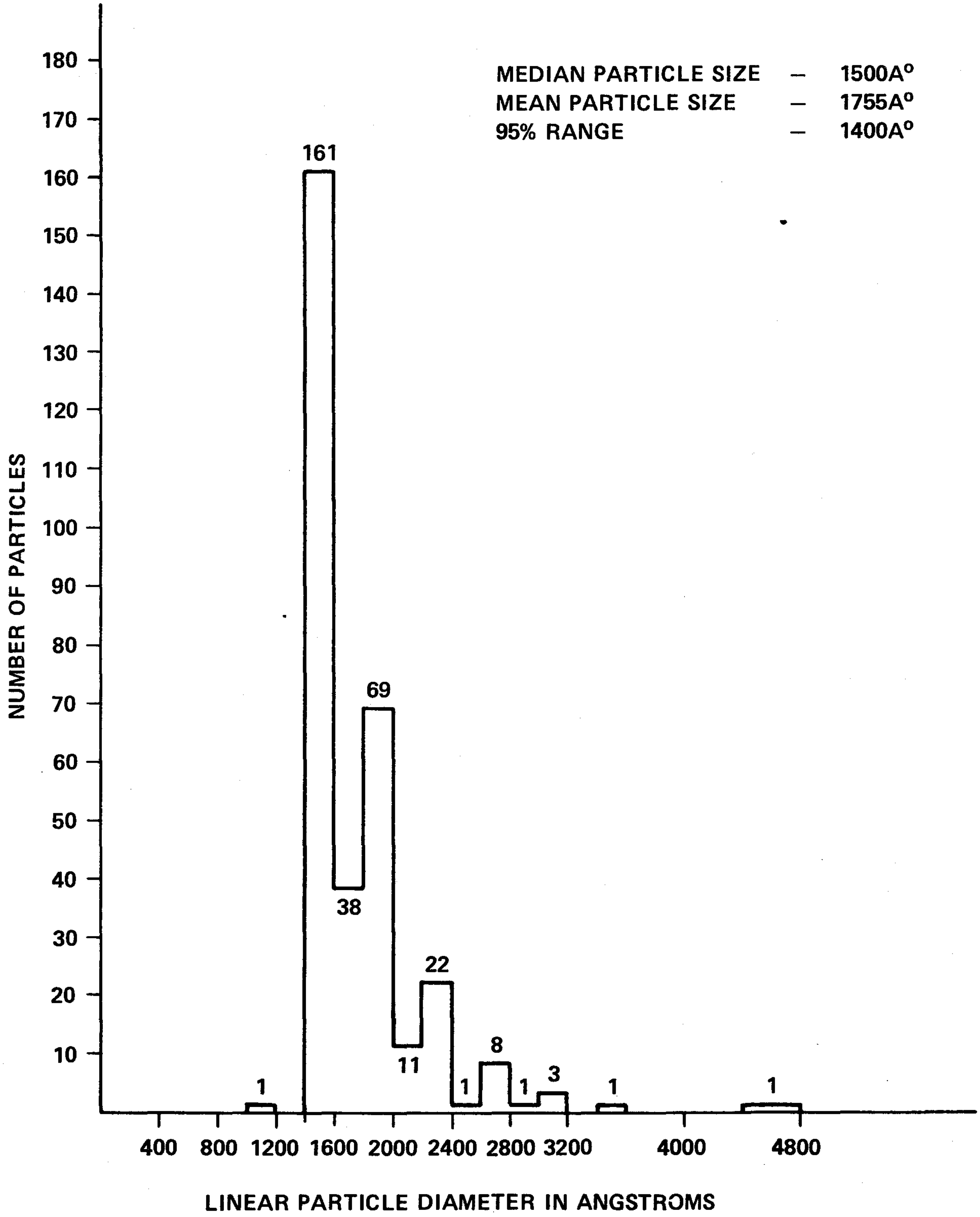


FIGURE 17. HISTOGRAM REPRESENTING FREQUENCY DISTRIBUTION OF THERMOFLEX 8002

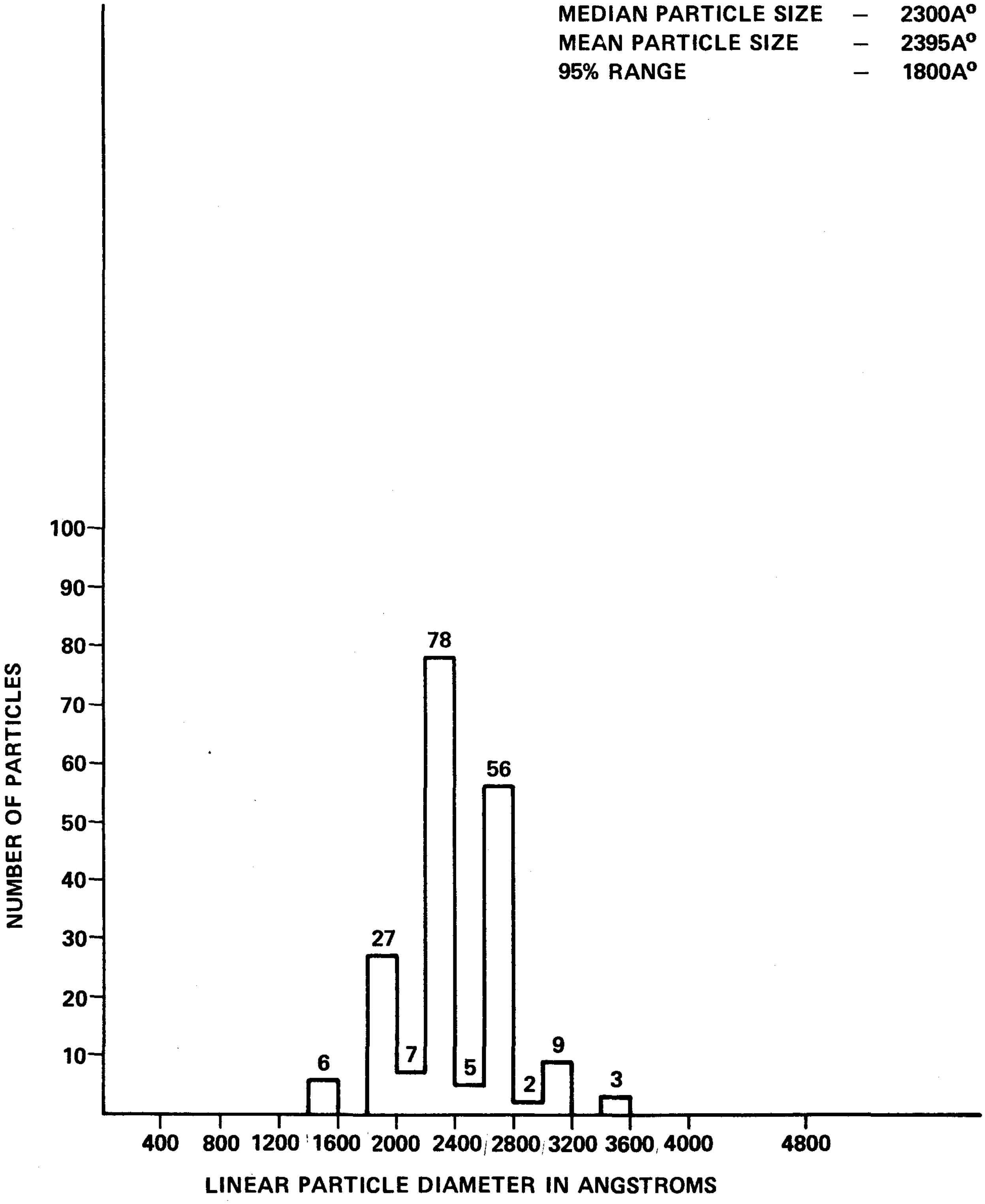


FIGURE 18. HISTOGRAM REPRESENTING FREQUENCY DISTRIBUTION OF DECO-REZ 4776

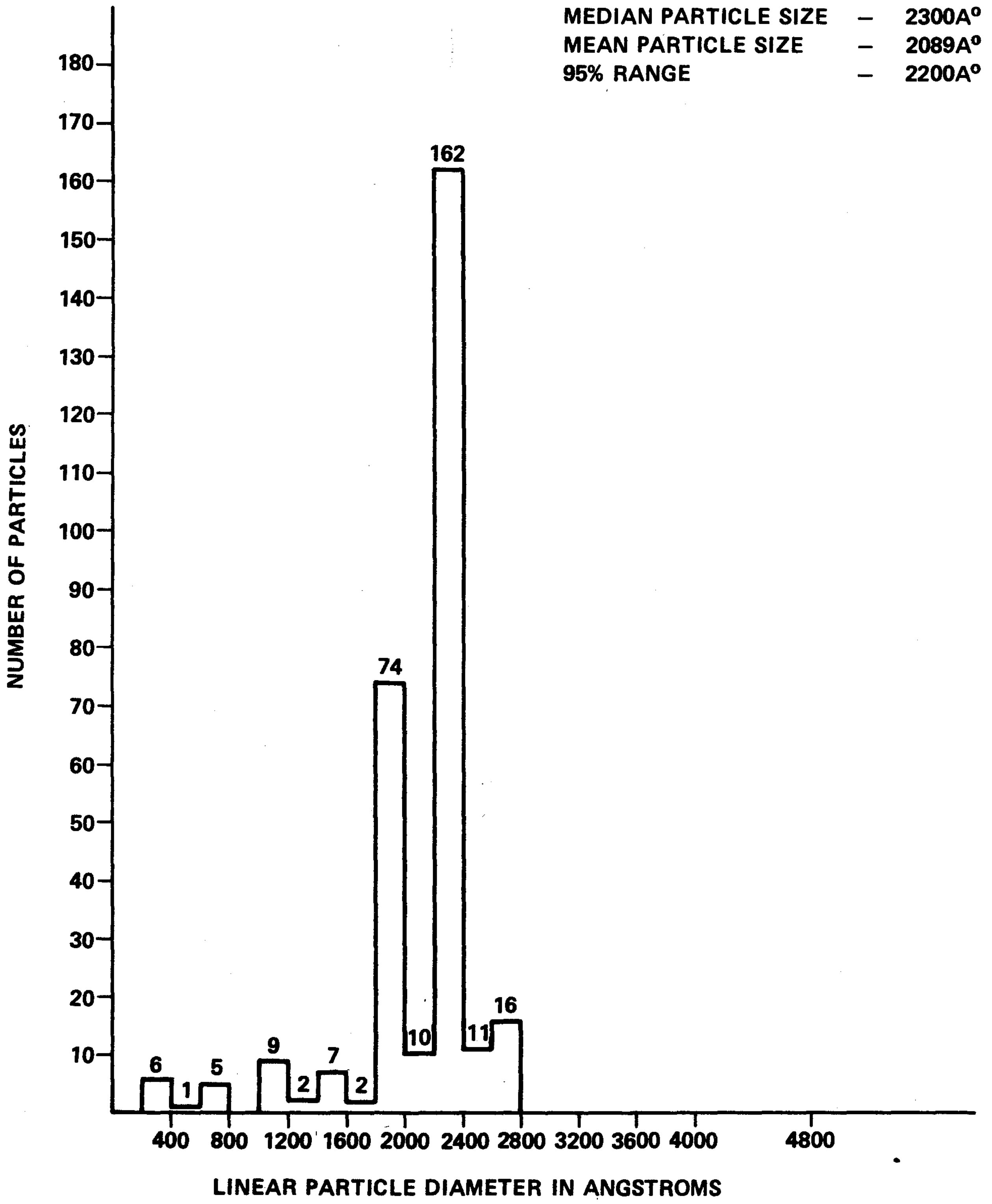


FIGURE 19. HISTOGRAM REPRESENTING FREQUENCY DISTRIBUTION OF ARCO DYLEX 1184

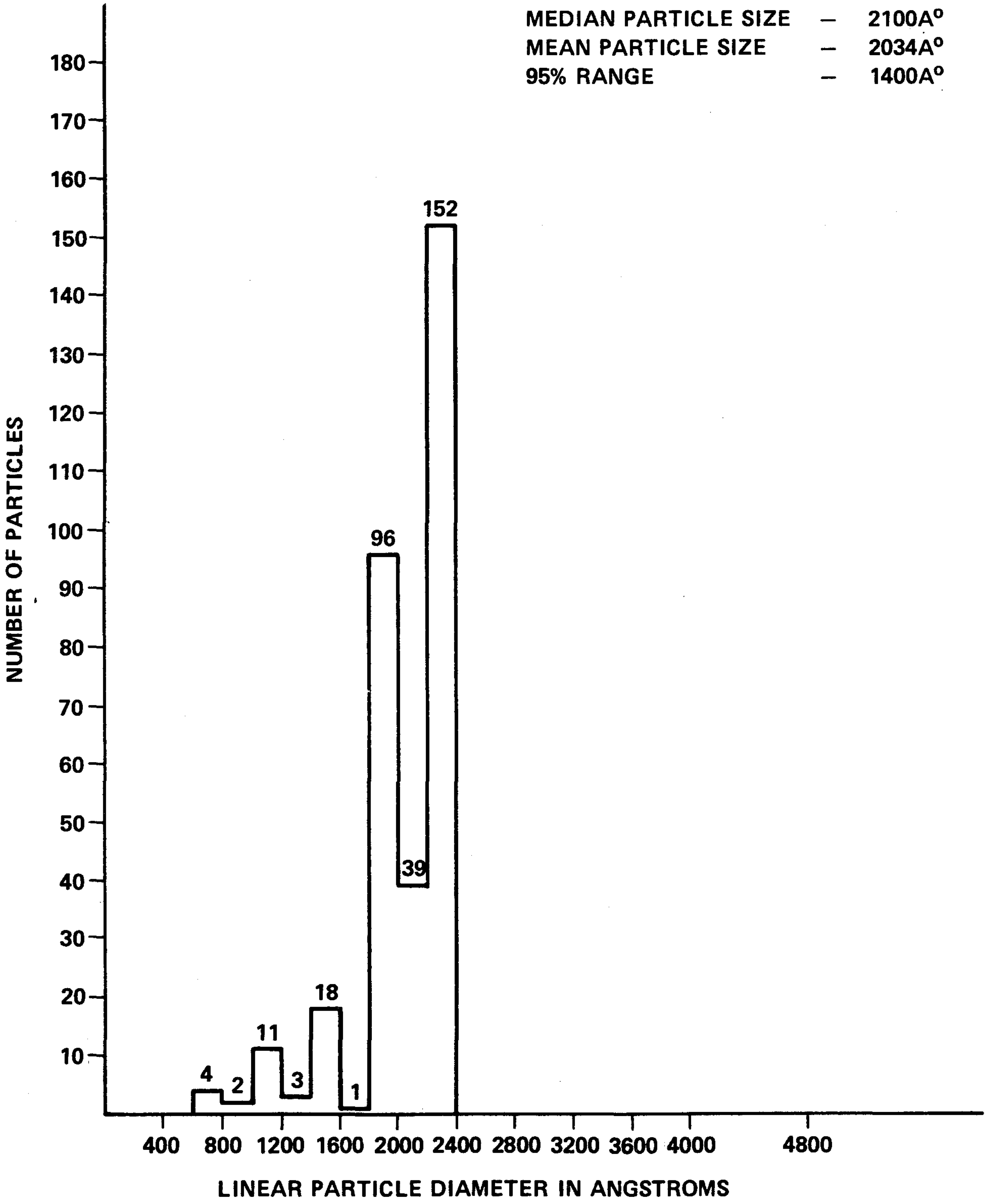


FIGURE 20. HISTOGRAM REPRESENTING FREQUENCY DISTRIBUTION OF ARCO DYLEX 1186

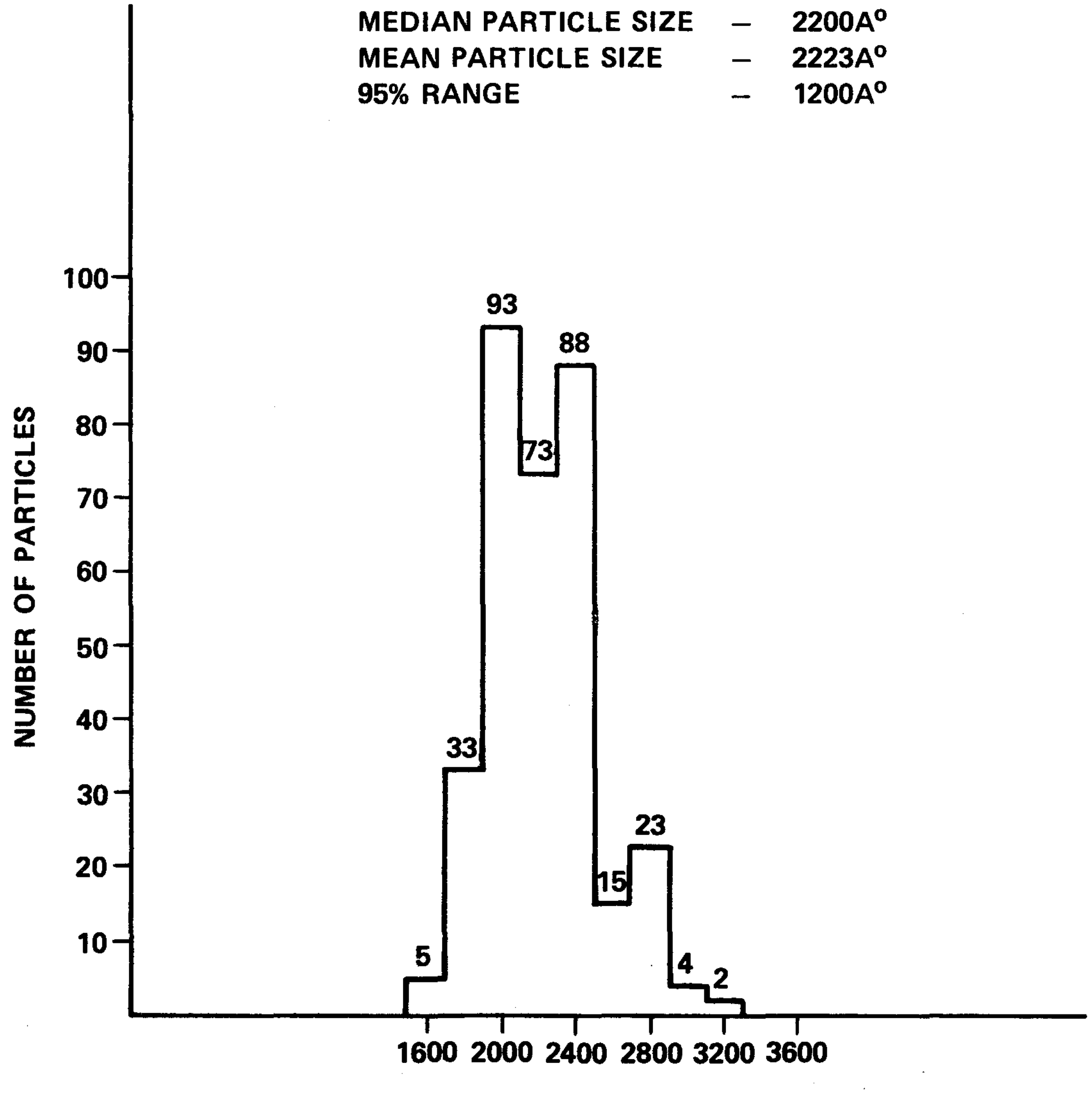


FIGURE 21. HISTOGRAM REPRESENTING FREQUENCY DISTRIBUTION OF DOW MODIFIER A

Table 12. Summary of latex particle sizes.

Particle size in Angstroms[1]

					95 Percent Range		
Latex	Arithmetic Mean	Median	Difference	Standard Deviation	Upper	Lower	Difference
Dow Modifier A	2,223	2,200	23	296	2,900	1,700	1,200
Deco-Rez 4776	2,395	2,300	95	373	3,200	1,400	1,800
Thermoflex 8002	1,755	1,500	255	381	2,800	1,400	1,400
Arco Dylex 1184	2,089	2,300	211	464	2,800	600	2,200
Arco Dylex 1186	2,034	2,100	66	346	2,400	1,000	1,400

[1] See Section 4.A.9 of the Prequalification Program in this report for the calculation procedure.

Both the mean and median particle sizes of the Thermoflex 8002 latex particles are smaller than those of the other latices. This is due to the manner in which the Thermoflex 8002 latex was formulated and, as a result, it would be expected that concrete containing this latex would have a lower compressive strength than concrete containing the other latices studied herein.

Latex Freeze-Thaw Stability

Samples of each latex were subjected to two freeze-thaw cycles, freezing each latex in a plastic container at a temperature of 0°F (-18°C) for 24 hours and thawing each in room temperature air (75°F, 24°C) for 24 hours, following the procedure in Section 4.A.10 except that a 200 mesh screen was used. A procedure using an 8-hour thawing period in air was attempted, but discarded. In the 8-hour thaw test, Dow Modifier A, Dylex 1184, and Dylex 1186 were completely thawed, showed no coagulum on the sides of their containers, and filtered well through a 200 mesh screen. However, Thermoflex 8002 and Deco-Rez 4776 were not completely thawed in this time period. These two latices were very viscous and very hard to filter. Some coagulum formed on the sides of the container containing Deco-Rez 4776 after one freeze-thaw cycle. However, after a 24-hour thawing period, both samples filtered well, and very little coagulum was found on the sides of the container with Deco-Rez 4776. Thus, it appears that the longer thaw period is advantageous, and since it more realistically simulates freezing in the field, that procedure was adopted.

The coagula present after the two freeze-thaw cycles (48 hours each) were:

Sample	Percent Coagulum, 200 mesh screen
1. Dow Modifier A	0.001
2. Deco-Rez 4776	0.063
3. Thermoflex 8002	0.039
4. Dylex 1184	0.005
5. Dylex 1186	0.005

All results are within the limits for an acceptable latex as given in Section 4.A.10.4. The pH's of all of the samples, except Thermoflex 8002, taken after the test were from 0 to 0.3 unit below that of the original latices. That of the Thermoflex 8002 emulsion was from 0.6 to 0.8 unit below that of the original latex. The larger pH change for the Thermoflex emulsion is not completely understood.

IV. Summary and Conclusions

1. The following styrene-butadiene latex emulsions are similar (but not identical) chemically and result in similar polymer-modified concretes when utilized at a dosage rate of 24.5 gal/yd^3 (121 l/m^3):

   Modifier A - Dow Chemical Company
   Thermoflex 8002 - Reichhold Chemicals, Inc.
   Deco-Rez 4776 - General Polymers Corp.
   Arco Dylex 1186 - Arco Polymers, Inc.

   The laboratory evaluation performed herein indicates that the materials should perform equally well as low permeability bridge deck overlay concretes.

2. Arco Dylex 1184 latex emulsion does not appear to be a suitable material for use in bridge deck overlay concrete because of the high water demand and high rate of slump loss exhibited by concrete made with this material.

3. An adequate indication of the chloride permeability of rigid concretes can be obtained by subjecting rigid concretes to 90 days of continuous ponding with a 3-percent sodium chloride solution after curing, sandblasting the surface, and conditioning in laboratory air (72°F (23°C), 50 percent Rh). However, for the materials studied herein, chloride ingress will increase as conditioning time prior to ponding is increased, and thus, specific conditioning procedures and times must be specified.

4. The chloride permeability of latex-modified concrete is significantly less than that of conventional concretes having water/cement ratios of 0.40 by weight or more. However, the latex-modified concretes are not impermeable and thus will allow chloride ingress at a reduced rate.

5. Comparative laboratory tests indicate that internally sealed (wax bead) concrete (w/c = 0.55) is significantly less permeable than all the latex-modified concretes (w/c = 0.36 to 0.40) studied herein.

6. Air contents in fresh latex-modified concrete as high as 9 percent had no significant adverse effect on the properties of the material. In fact, high air contents (6 to 9 percent) may have a beneficial effect on the early age freeze-thaw durability and/or deicer scaling resistance of latex-modified concrete. However, it does not appear that the 0.008-inch (0.20-mm) maximum air void spacing factor ($\bar{L}$) criterion, commonly believed necessary for freeze-thaw resistant conventional concrete, is applicable to the latex-modified concretes studied herein.

7. Concretes made with all the latex emulsions tested herein are somewhat water-susceptible in that a reduction in compressive and slant shear composite-compressive (an indirect measurement of bond) strengths occurred when the air-cured concretes were soaked in saturated limewater for 14 days. Similarly, reductions in flexural strength upon limewater soaking occurred with concretes made with all emulsions except Thermoflex 8002. As a result, the highly acclaimed increased flexural strength of S/B latex-modified concrete over that of a similar conventional concrete virtually disappeared during the limewater soaking from 28 to 42 days of age.

8. Styrene-butadiene latex emulsions can be fingerprinted using the infrared spectroscopy procedures described herein. Two infrared spectra, one on a latex film and the other on the residue of the alcohol solubles in the emulsion, are required. Strict adherence to specific infrared transmittance criteria and qualitative interpretation of the resultant spectra using the procedures discussed in this report are necessary.

9. Although a general chemical specification for S/B latex emulsion was developed, detailed qualification testing of the emulsion and concrete made using the emulsion is necessary to insure that a product in acceptable. This seemingly redundant testing is needed to assure adequate performance because emulsion additives whose chemical compositions are normally unknown to the user are primary determinants of performance. The additives prevent foaming (very high air contents), provide stability in the alkaline portland cement concrete environment, and control the colloidal and rheological properties of both the latex emulsion and the latex-modified portland cement concrete.

10. Infrared spectroscopy can be used to quantitatively determine the butadiene content of a styrene-butadiene latex emulsion if specific film preparation, infrared transmittance, and calculation procedures are followed. The resulting 90 percent confidence range in the percent butadiene will be about ± 2 or 3 percent.

11. A prequalification program for other S/B latex emulsions is given in Section VI of this report. The program consists of the following chemical and physical tests of the latex emulsion:

    a) Infrared Fingerprint of Latex Film.
    b) Infrared Fingerprint of Alcohol Solubles in the Emulsion.
    c) Percent Butadiene in the Solids.
    d) Weight Percent Solids in the Emulsion.
    e) pH.
    f) Weight Percent Coagulum.
    g) Viscosity.

h) Surface Tension.
i) Polymer Particle Size and Size Distribution.
j) Freeze-Thaw Stability.

In addition, the following tests of latex-modified concrete made using the emulsion are suggested:

a) Workability and Slump Loss Versus Time.
b) Compressive Strength.
c) Flexural Strength.
d) Slant Shear Composite Strength.
e) Deicer Scaling Resistance.
f) Chloride Permeability.

Conventional concrete controls are required in many of the concrete tests for comparison purposes.

12. A certification program, consisting of chemical and physical tests on the latex emulsion, is defined in Section VII of this report. It is suggested for use to insure that each production batch of a prequalified S/B latex emulsion is similar to the emulsion on which the prequalification tests were performed. The tests included in the certification program are:

a) Fingerprint of Latex Film.
b) Fingerprint of Alcohol Solubles in Emulsion.
c) Weight Percent Solids.
d) Mean Particle Size.
e) pH.
f) Viscosity.
g) Surface Tension.
h) Percent Coagulum.
i) Freeze-Thaw Stability.
j) Percent Butadiene in the Emulsion.

13. Because the various latex emulsions from different manufacturers are chemically dissimilar, S/B emulsions from various manufacturers should not be mixed. Placement of fresh concrete containing one emulsion adjacent to that containing another emulsion, however, would not be expected to present problems.

V. References

1. Kriegh, James D., "Arizona Slant Shear Test: A Method to Determine Epoxy Bond Strength," Journal of the American Concrete Institute, Proceedings Vol. 73, No. 7, July 1976, pp. 372-373.

2. Clear, K. C. and Hay, R. E., "Time-to-Corrosion of Reinforcing Steel in Concrete Slabs - Vol. 1: Effect of Mix Design and Construction Parameters," Report No. FHWA-RD-73-32, Federal Highway Administration, Interim Report, April 1973, 103 pp.

3. Clear, K. C., "Time-to-Corrosion of Reinforcing Steel in Concrete Slabs, Vol. 3: Performance After 830 Daily Salt Applications," Report No. FHWA-RD-76-70, Federal Highway Administration, April 1976.

4. Clear, K. C. and Harrigan, E. T., "Sampling and Testing for Chloride Ion in Concrete," Federal Highway Administration, Report No. FHWA-RD-77-85, August 1977.

5. Clear, K. C. and Forster, S. W., "Internally Sealed Concrete: Material Characterization and Heat Treating Studies," Report No. FHWA-RD-77-16, Federal Highway Administration, Interim Report, March 1977, 73 pp.

6. Attachment to correspondence from Jim Stone, Supervisor, Concrete Section, Division of Materials, Kentucky Department of Transportation, to Kenneth C. Clear, Federal Highway Administration, August 11, 1977.

7. "Latex-Modified Portland Cement Concrete Technology Seminar," given by Douglas Eash, Dow Chemical, U.S.A., Midland, Michigan 48640, November 11 and 12, 1976.

8. Private communication between Dr. Douglas Eash, Dow Chemical, U.S.A., and Brian Chollar, Materials Division, FHWA, September 6, 1977.

9. Private communication between Jerry Walters of Reichhold Chemicals, Inc., and Brian H. Chollar, Materials Division, FHWA, September 6, 1977.

10. Maron, S. H. and Prutton, C. F., "Principles of Physical Chemistry," Third Edition, The MacMillan Company, New York, New York, 1959.

11. Private communication between Gerald Burkhart of ARCO Polymers, Inc., and Brian H. Chollar, Materials Division, FHWA, March 3, 1978.

12. Natrella, Mary Gibbons, "Experimental Statistics," National Bureau of Standards Handbook 91, U.S. Department of Commerce, October 1966, pp. 5-36.

13. Arni, H. T., "Impact and Penetration Tests of Portland Cement Concrete," Federal Highway Administration, Final Report, FHWA-RD-73-5, February 1972.

14. Orr, Clyde, Jr., and Dallavalle, J. M., "Fine Particle Measurement," The MacMillan Company, New York, 1959, p. 10.

15. Steel, Robert G. D. and Torrie, James H., "Principles and Procedures of Statistics, McGraw-Hill Book Company, New York, 1960.

16. Harkins, W. D. and Jordan, H. F., Journal of the American Chemical Society, 52, 1930, pp. 1764-68.

17. Fox, H. W. and Chrisman, C. H., Jr., Journal of Physical Chemistry, 56, 1952, p. 284.

18. Private communication between L. P. Finnegan of Reichhold Chemicals, Inc. and Kenneth C. Clear, Materials Division, FHWA, March 30, 1978.

19. "Part 14: Concrete and Mineral Aggregates," 1977 Annual Book of ASTM Standards, American Society for Testing and Materials, 1977.

VI. Prequalification Test Program

PREQUALIFICATION OF STYRENE-BUTADIENE LATEX EMULSIONS FOR BRIDGE DECK OVERLAY CONCRETE

1.0 Scope

1.1 This specification covers the qualification requirements for styrene-butadiene latex emulsions for use in concrete for thin-bonded bridge deck overlays. A styrene-butadiene emulsion is defined as a latex emulsion in which at least 90 percent of the non-volatiles are styrene-butadiene polymers.

1.2 The specification is divided into two parts:

(A) Chemical and physical requirements of the latex emulsion, and

(B) Physical requirements of concrete made using the latex emulsion.

In part (A), specific chemical composition and physical property requirements are listed. In part (B), both specific physical property requirements and comparative requirements (i.e., a property of the latex-modified concrete is compared to another property of the same concrete or to the same property of a control conventional concrete made with the same cement, water, and aggregates) are used.

2.0 Material for Test

Twenty gallons of the latex emulsion shall be submitted to the testing agency along with the following information: generic description; fingerprint of the latex solids in the emulsion and the alcohol solubles in the emulsion, performed in accordance with Section 3.0 below; a general description of production procedure and production date; production lot size (i.e., laboratory or full-production batch) and lot identification number; and a listing of all monomers and polymers charged during emulsion manufacture and the relative percentages of each used during batch charging.

3.0 Infrared Fingerprint of Material

3.1 Instrumentation

An infrared spectrophotometer exhibiting the following capabilities shall be used to obtain the fingerprinting spectra of the solid latex films and the alcohol soluble portion of the latex emulsion:

Range = 4000 to 500 cm^{-1}
Resolution = 0.3 cm^{-1}
Readout = linear in wave numbers (i.e., frequency)
Frequency Accuracy = 0.5 cm^{-1}
Frequency Repeatability = 0.25 cm^{-1}

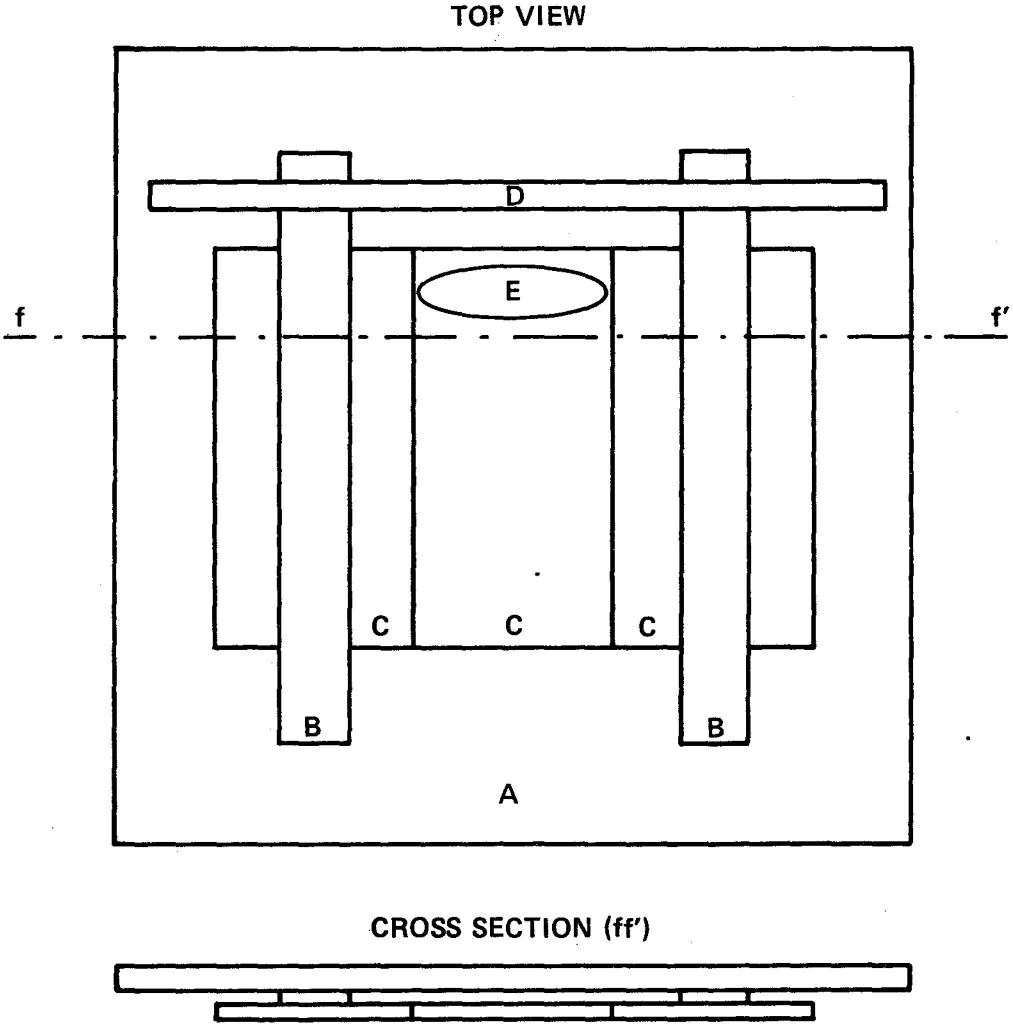
Two types of cell holders may be used with the spectrophotometer; one that holds a latex film only and one that holds a silver or sodium chloride plate containing a coating of latex film. Both cell holders shall be designed in such a way that the infrared beam passes through only the latex film or through the plate and film coating while the spectrum is being obtained. Sodium chloride plates in a conventional cell holder shall be used to obtain the spectrum of the alcohol soluble portion of each latex.

3.2 Infrared Fingerprint Spectra of the Latex Films

3.2.1 Preparation of the Films. Two methods are given to prepare latex films. Only the first method (3.2.1.1) may be used to obtain complete IR spectra (4000 to 600 cm^{-1}) of latices (i.e., the fingerprint). Either method (3.2.1.1 or 3.2.1.2) may be used to obtain spectra used for percent butadiene content of latices (Section 4.A.1 below).

3.2.1.1 Method 1. Prepare a coating assembly as shown in Figure 22. The spacing thickness (b) shall be 0.003 inch (0.075 mm). Apply 1 millilitre of the emulsion to the top of the center slide as shown in Figure 22 and draw the rod slowly down over the center slide to spread the latex evenly over the slide. Let the coated slide dry for 15 minutes. After drying, place the coated slide under hot water (35° to 45°C) and carefully remove the dried film from the plate using a razor blade and tweezers, making sure the film surface does not overlap or adhere to itself. Dry the film by placing it between absorbent paper towels and applying hand pressure for 5 seconds. Place this latex film on a cell holder equipped to hold films so that the infrared beam passes directly through the film.

3.2.1.2 Method 2. Prepare a latex wire sample holder made of a wire (1 mm thick) bent into a circle of 35 cm in diameter with part of the wire used as a handle extending perpendicular from the plane of the circle. Pour about 10 ml of the latex into an aluminum weighing dish (4 cm in diameter and 1 cm deep). Dip the wire sample holder (the wire loop)



A. GLASS PLATE (≈20 X 20 cm.)

B. SPACING MATERIAL TO ADJUST THICKNESS OF COATING (TRANSPARENT MENDING TAPE USED)

C. GLASS MICROSLIDES (≈2½ X 7½ cm.)

D. SMOOTH METAL OR GLASS ROD FOR COATING GLASS PLATE WITH LATEX (≈0.6 cm. IN DIAMETER)

E. INITIAL POSITION OF EMULSION SAMPLE

FIGURE 22. APPARATUS FOR PREPARING LATEX FILMS

into the latex and draw it out carefully. Note, latex film should be adhering to the wire loop. If difficulties arise in obtaining the film within the wire loop, the surface tension for that latex is too low to support a film by this technique. Use the alternative procedure (Section 3.2.1.1) to prepare these films. Place the film adhering to the wire loop on a clean, polished, circular sodium chloride or silver chloride plate of 25 ± 5 cm diameter by slowly lowering the wire loop with the latex sample to the plate holder (plane of the loop is parallel to the flat side of the plate) until the latex film is placed on the flat plate and the plate has passed through the circular part of the wire. The film should cover the plate and not be adhering to any portion of the wire loop. Dry the film on the plate using an infrared heating lamp until the film is transparent (usually about 2 to 5 minutes). Place this plate, containing the film, in a cell holder made to hold plates so that the infrared beam passes directly through the film and the plate. Obtain the spectrum of this film using the procedure given in 4.A.1.1 below. Remove the film from the plate using chloroform as a solvent and polish the plate until its original transparency is regained.

3.2.2 Obtaining the Infrared Fingerprint Spectrum of the Latex Film. Place the latex film on a cell holder equipped to hold films and position this holder in the instrument so that the infrared beam passes directly through the film. Position the pen to read between 90 and 95 percent transmittance at 2,100 to 2,000 cm^{-1} by adjusting the 100 percent dial of the instrument. Examine the spectrum of this film between 3,000 and 2,800 cm^{-1}. The tip of the peak at 2,920 cm^{-1} should be between 10 and 25 percent transmittance. If it is, run the complete spectrum of this film between 4,000 and 600 cm^{-1}. If the peak at 2,920 cm^{-1} extends to a lower transmittance percentage, gently stretch the film to approximately 1¼ times its length, replace the stretched film into the holder, and reexamine the spectrum of the stretched film. If the peak at 2,920 cm^{-1} still extends to a lower transmittance percentage than 10 percent, prepare another film (per Section 3.2.1.1) using a smaller spacer thickness and obtain its spectrum using the above procedure.

3.3 Infrared Fingerprint Spectrum of the Alcohol-Soluble Portion of Latex Emulsions

3.3.1 Preparation of the Alcohol Soluble Portion of the Latex. Add 10.0 grams of the latex emulsion dropwise to 100 ml of an isopropanol solution in a 250-ml beaker. Stir the mixture continuously, both during addition and for 5 minutes after completion of addition, using a magnetic stirring bar and motor at room temperature. Filter the mixture through a moderately retentive filter paper of medium filter speed (e.g., Whatman No.2 or equivalent) and wash the residue in the funnel with ethanol (2 x 20 ml portions), allowing the ethanol washings to combine with the isopropanol filtrate. Evaporate the alcohol filtrate to dryness and dissolve the residue in boiling ethanol (250 ml). After letting the hot mixture settle for 1 minute, decant the hot solution from any insoluble particles and evaporate the liquid to dryness to obtain the viscous, gelatinous residue.

3.3.2 Obtaining the Infrared Fingerprint Spectrum of the Alcohol Soluble Portion. Add approximately 5 mg of the alcohol soluble residue to a sodium chloride infrared plate of 1 inch square or equivalent size so that the complete infrared beam will pass through only this salt plate. Sandwich this residue between that plate and a second sodium chloride plate of the same size so that only a thin coating of material exists between the two plates. Mount the plates in an infrared cell holder and insert the holder into the infrared instrument. Position the pen between 90 and 95 percent transmittance at 1,800 to 1,900 cm^{-1} by adjusting the 100 percent dial of the instrument. Examine the spectrum of this film at 1,100 cm^{-1}. If the tip of the peak at 1,100 cm^{-1} is between 10 and 25 percent transmittance, run the complete spectrum of the film (4,000 to 600 cm^{-1}). If this peak extends to a lower transmittance percentage at this wavelength, repeat the above procedure using less sample.

4.0 Testing Program

Part A. Chemical and Physical Testing and Requirements of the Latex Emulsion

4.A.1 Weight Percent Butadiene in Butadiene-Styrene Latices

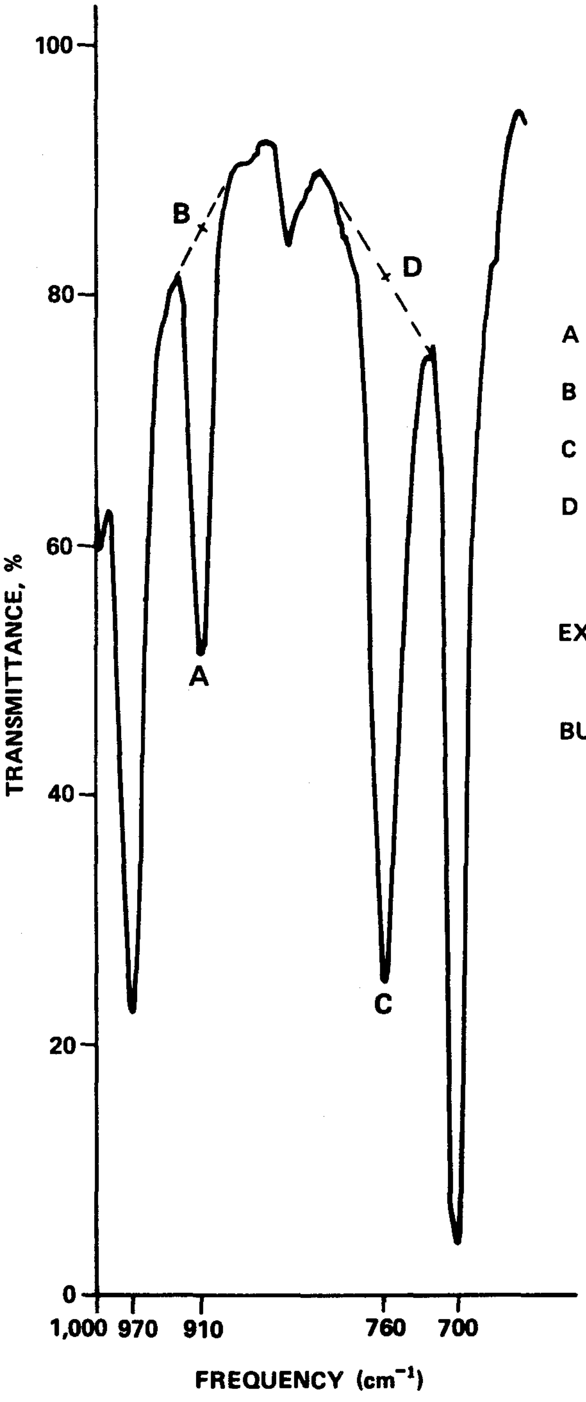
4.A.1.1 Obtaining the Infrared Spectra of the Films Containing Known Weight Percent Butadiene. Prepare films of six replicate samples of a latex containing a known weight percentage of butadiene in the solids of 20 to 50 percent. Appendix A defines a suggested reference (known butadiene content) sample preparation procedure. For film preparation, use either the procedure described in Section 3.2.1.1 with the spacer thickness of 0.0045 inch (0.12 mm) or the procedure described in Section 3.2.1.2. Position one of the six films (or plates containing films) on a cell holder and insert the holder into the instrument so that the infrared beam passes directly through the film. Position the pen to read between 90 and 95 percent transmittance at either 805 or 855 cm^{-1}, the frequency having the greater transmittance value, by adjusting the 100 percent dial of the instrument. Examine the spectrum at 760 cm^{-1}. If the transmittance of the peak at 760 cm^{-1} is above 40 percent, discard the film and prepare another using the procedure in Sections 3.2.1.1 with a larger spacer or 3.2.1.2. If the transmittance of the peak at 760 cm^{-1} is below 20 percent, discard the film on the plate if the procedure in Section 3.2.1.2 was used and prepare another film using that same procedure. If the procedure in Section 3.2.1.1 was used, gently stretch the film to 1¼ times its original size, insert the stretched film into the cell holder, position the holder into the instrument, and again examine the transmittance of the peak at 760 cm^{-1}. If it is still below 20 percent, discard the film and prepare another using the procedure in Section 3.2.1.1 with a smaller spacer. Repeat the above procedure until films are obtained having transmittance values of 90 to 95 percent at 805 or 855 cm^{-1} and 20 to 40 percent at 760 cm^{-1}. Obtain the spectra of six films of one latex sample between 1,000 to 650 cm^{-1}. Repeat this procedure with at least five samples containing known weight percent butadiene throughout the range of 20 to 50 percent (i.e., for a total of 6 samples per butadiene content times 6 butadiene contents = 36 samples).

4.A.1.2 Calculation of Butadiene-Styrene Peak Ratios and the Preparation of the Reference Chart. Draw a base line for the butadiene peak at 910 cm^{-1} and one for the styrene peak at 760 cm^{-1} on each of the spectra obtained in Section 4.A.1.1 as shown in Figure 23. Calculate the peak ratio of each sample using the equation given in Figure 23.

Convert each ratio to logarithms and calculate the least squares, best fit straight line (see Reference 12 of this report) of the form $\log y = a + bx$ for the butadiene content (X) versus ratio (Y) data. The resulting regression equation plotted on semilog paper (ratio (Y) on the log scale) is the reference chart. Show each individual data point on the chart as an indication of data scatter.

The reference chart or the equation of the reference line shall be used to determine the butadiene content of latices of unknown composition (see 4.A.1.3 below).

4.A.1.3 Determination of the Weight Percentage Butadiene in the Subject Latex. Obtain an infrared spectrum of six samples of the subject butadiene-styrene latex using the procedures described in Section 4.A.1.1.



$$\text{BUTADIENE/STYRENE} = \frac{\text{LOG}\ \frac{B}{A}}{\text{LOG}\ \frac{D}{C}}$$

A = TRANSMITTANCE AT THE TIP OF THE PEAK AT 910^{-1}

B = TRANSMITTANCE AT THE BASE OF THE PEAK AT 910 cm.$^{-1}$

C = TRANSMITTANCE AT THE TIP OF THE PEAK AT 760 cm.$^{-1}$

D = TRANSMITTANCE AT THE BASE OF THE PEAK AT 760 cm.$^{-1}$

EXAMPLE:

$$\text{BUTADIENE/STYRENE} = \frac{\text{LOG}\ \frac{85.3}{51.2}}{\text{LOG}\ \frac{81.2}{25.0}} = \frac{0.2216}{0.5117}$$

$$= 0.433$$

FIGURE 23. CALCULATION OF THE BUTADIENE/STYRENE RATIO FROM THE INFRARED SPECTRUM OF THE LATEX FILM

Calculate the butadiene-styrene peak ratio for each of the six samples and determine the average ratio as described in Section 4.A.1.2. Using the average ratio, determine the percent butadiene of the subject latex using the reference chart or the equation of the reference line defined in Section 4.A.1.2.

4.A.1.4 Reporting Procedure. The average weight percentage of butadiene in the latex sample as derived in Section 4.A.1.3 shall be reported. The six infrared spectra, with each calculated ratio shown thereon, and the reference chart, with reference sample data points plotted thereon, shall also be included in the report.

4.A.1.5 Acceptance Criteria. The average weight percentage of butadiene for an acceptable latex shall be 30 to 40 weight percent.

4.A.2 Total Weight Percent Solids in the Latex

4.A.2.1 Procedure. Mix the latex sample by inverting the sample container 5 to 10 times. Weigh three aluminum cups (2 gm or less in weight and with enough capacity to hold 2 ml of liquid) and record the weight of each. Weigh approximately 1 gm of the latex emulsion (room temperature) into each tared aluminum cup. Place the three samples in an oven to dry for 2 hours at 285 ± 2°F (140 ± 1°C). Remove the samples from the oven and immediately place them in a desiccator to cool. After cooling to room temperature, reweigh each sample.

Notes: 1. All weighings shall be to the nearest milligram.

2. If jobsite sampling and testing for solids content is being performed, care should be exercised to insure that a representative sample is obtained. Samples should not be taken from cleanout lines or hoses which may contain trapped wash water.

4.A.2.2 Calculations. Calculate the total solids in weight percent as follows:

$$\text{Total solids} = \frac{C - A}{B - A} \times 100.$$

A = weight of the empty aluminum cup.

B = weight of the aluminum cup and the wet sample.

C = weight of the aluminum cup and the dried sample.

Example:

$$\text{Total solids in percent} = \frac{1.840 - 1.374}{2.356 - 1.374} \times 100 = 47.5 \text{ percent.}$$

4.A.2.3 Reporting Procedure. Report each individual result and the average of the three samples as the weight percent solids.

4.A.2.4 Acceptance Criteria. The average weight percent solids content for an acceptable latex shall be 46 to 53 weight percent.

4.A.3 Adjustment of Percent Solids

If the weight percent solids content of the latex is above 46.0 percent solids (see Section 4.A.2), add the appropriate amount of distilled water to the latex as calculated below:

$$Q = \frac{M \times P}{46} - M$$

Q = quantity of distilled water to add (ml).

M = quantity of latex to be diluted (ml).

P = solids content of the latex (percent).

Allow the diluted emulsion to stabilize at room temperature for at least 24 hours prior to use. Latices adjusted to 46.0 weight percent shall be subjected to the remaining prequalification procedures.

4.A.4 pH of Latices

4.A.4.1 Procedure. Use a Beckman or equivalent pH meter equipped with glass and calomel electrodes exhibiting a maximum measuring sensitivity of 0.1 pH unit. Standardize the electrode against a known buffer solution at 77°F (25°C), following the directions on the instrument. Equilibrate the latex sample (46.0 percent solids, 50 ml in a 100-ml beaker) to 25 $\pm$ 1°C by conditioning in a constant temperature water bath. Immerse the glass and calomel electrodes of the pH meter into the latex and read the pH directly from the scale of the pH meter. After each determination, wipe the electrodes with absorbent tissue, immerse them in distilled water, and again wipe clean. Repeat this cleaning procedure until no visible traces of latex remain on the electrode.

4.A.4.2 Reporting Procedure. Report the pH reading to the nearest 0.1 unit.

4.A.4.3 Acceptance Criterion. The pH of an acceptable latex emulsion shall be between 8.5 and 12.0.

4.A.5 Percent Coagulum

4.A.5.1 Procedure. Pour 900 gm of latex (46.0 percent solids) through a 40 mesh screen and collect the screened latex by means of a funnel and a sample bottle. Equilibrate the collected latex to 25 ± 1°C by conditioning in a constant temperature water bath and weigh it. Pour the latex through a 100 mesh screen and collect the screened latex by means of a funnel and a sample bottle. Wash the residue on the 100 mesh screen with cool water followed by a warm (35° to 40°C) water rinse. (Warm water aids in the coagulation of the residue.) Collect any residue from the 100 mesh screen and quantitatively transfer it to a tared round aluminum dish (lighweight, disposable, of at least 2 cm in diameter). Place the dish in a 250°F (120°C) oven for 30 minutes to dry. Cool the dish to room temperature in a desiccator and reweigh it.

4.A.5.2 Calculation. Substitute the weights in the following equation and calculate the weight percent of coagulum:

$$\text{Weight percent coagulum} = \frac{\text{weight of coagulum} \times 10{,}000}{\text{weight of latex used} \times \text{percent solids in the latex}}$$

Example: Weight of coagulum = 0.18 gm
Weight of latex used = 904 gm
Percent solids content = 46.0 percent

$$\text{Weight percent coagulum} = \frac{0.18 \times 10{,}000}{904 \times 46.0} = 0.093 \text{ percent}$$

4.A.5.3 Reporting Procedure. Report the calculated value as weight percent coagulum to the nearest 0.001 percent.

4.A.5.4 Acceptance Criteria. The weight percent coagulum shall not exceed 0.10 percent for an acceptable latex.

4.A.6 Brookfield Viscosity

4.A.6.1 Instrument. A Brookfield Synchro Lectric Viscometer, model RVF, of the type shown in Figure 24 shall be used with spindle No. 1, the largest spindle supplied with the instrument (56 mm in diameter and 22 mm in depth).

4.A.6.2 Procedure. Filter approximately 600 ml of the latex sample (adjusted to 46.0 percent solids) through a 60 mesh screen into an 800-ml beaker. Condition this for 1 hour in a constant temperature water bath at 25 ± 0.1°C. Place this beaker of latex into a 2-litre beaker containing 400 ml water at 25 ± 0.1°C. Connect spindle

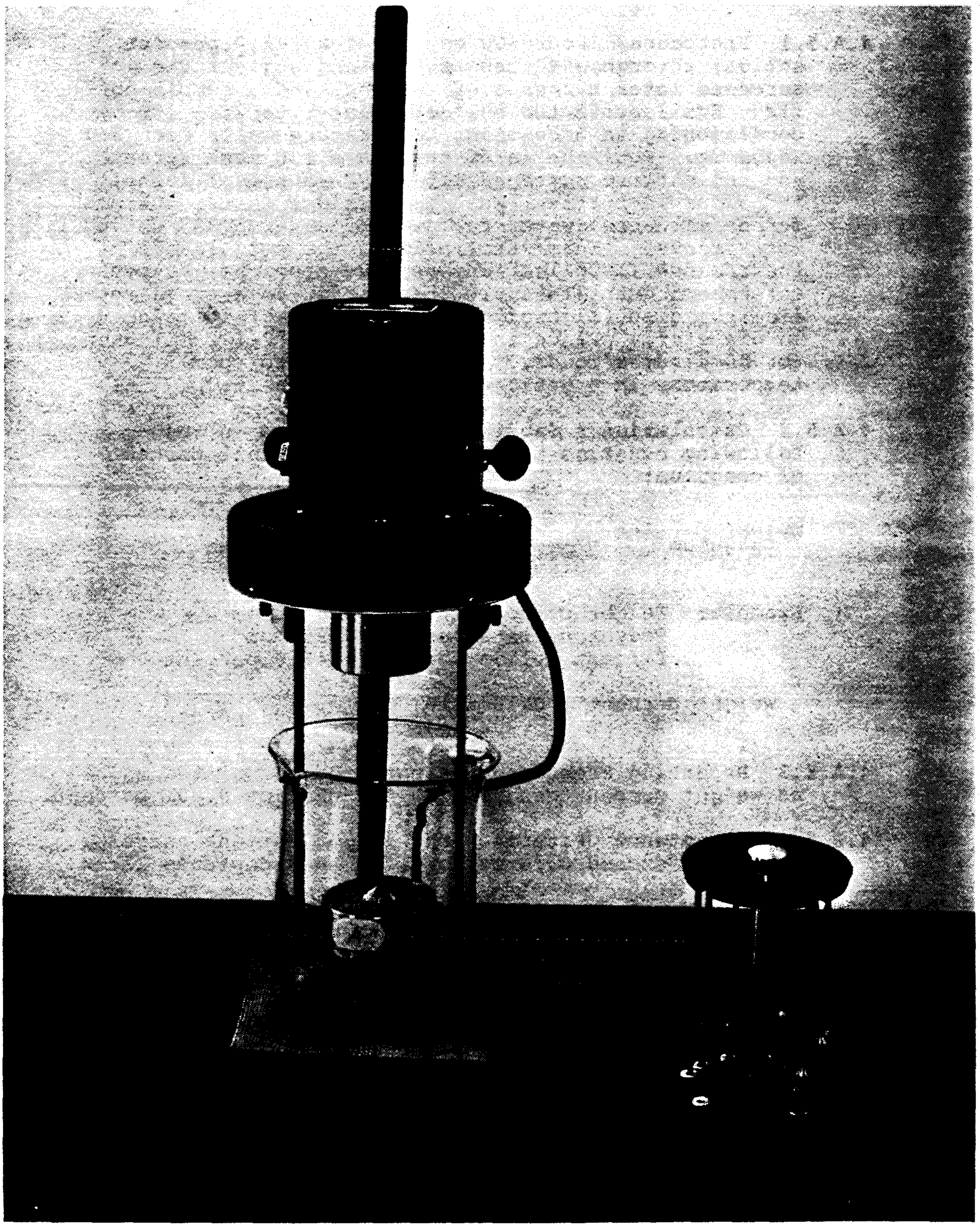


FIGURE 24. BROOKFIELD SYNCHRO-LECTRIC VISCOMETER, MODEL RVF, WITH SPINDLE NO. 1 ATTACHED.

No. 1 from the viscometer kit to the Brookfield Synchro Lectric Viscometer and immerse the spindle into the latex (in the 800-ml beaker) to the indicated depth on the spindle. Start the viscometer motor and adjust the spindle speed to 20 rpm. Allow the pointer on the viscometer to reach a constant value (at least 30 seconds will be required for equilibrium), press the brake and turn off the motor switch simultaneously, and take the reading on the proper scale. Take three readings at this speed and record the average reading. Adjust the speed to 10 rpm and repeat the above procedure. Take three readings at this speed and record the average reading.

4.A.6.2 Calculations. Multiply the average reading at each speed by the conversion factor supplied with the viscometer to obtain the viscosity.

4.A.6.3 Reporting Procedure. Report the average viscosity at each speed in centipoises at 25°C.

4.A.6.4 Acceptance Criteria. None.

4.A.7 Determination of Latex Density

Record the weight (C in gm) of a clean 100-ml volumetric flask. Filter the latex sample through a 100 mesh screen and add 100 ml of the filtrate to the flask, filling it to the indicated 100-ml mark on the flask. Reweigh the filled flask (F in gm) and calculate the latex density using the following equation:

$$\text{Density (D)} \ \frac{\text{gms}}{\text{ml}} = \frac{F - C}{100}$$

Report the density of the latex emulsion at 46-percent solids (for use in Section 4.A.8). Use this latex, filtered through a 100 mesh screen, for the determination of surface tension.

Note: Weighings shall be made to the nearest milligram.

4.A.8 Surface Tension

4.A.8.1 Apparatus.

Cenco-du Nouy Tensiometer, No. 70545, or equivalent. Such an instrument is shown in Figure 25. It should be enclosed in a housing to protect it from air flows.

Platinum-iridium ring, No. 70537, 4 cm in diameter, supplied with the tensiometer.

Drying dish, 4 cm in diameter, 1 cm deep, to hold the latex sample. A disposable aluminum or a reflamable platinum dish may be used.

V	TORSION ARM
I	INDEX
Q	VERTICAL ARM
X	CLAMPING JAWS
T	SAMPLE TABLE
B	SAMPLE TABLE ADJUSTMENT SCREW
E	COUNTER WEIGHT
J	VERTICAL ARM RESTRICTIONS
K	POINT OF RING DETACHMENT FROM VERTICAL ARM
G	TENSIOMETER ADJUSTMENT SCREW
Y	FINE ADJUSTMENT SCREW
S	DIAL
Z	VERNIER
C	DIAL CLAMP
O	BASE LEVELING SCREW
H	SAMPLE TABLE ASSEMBLY ADJUSTMENT SCREW

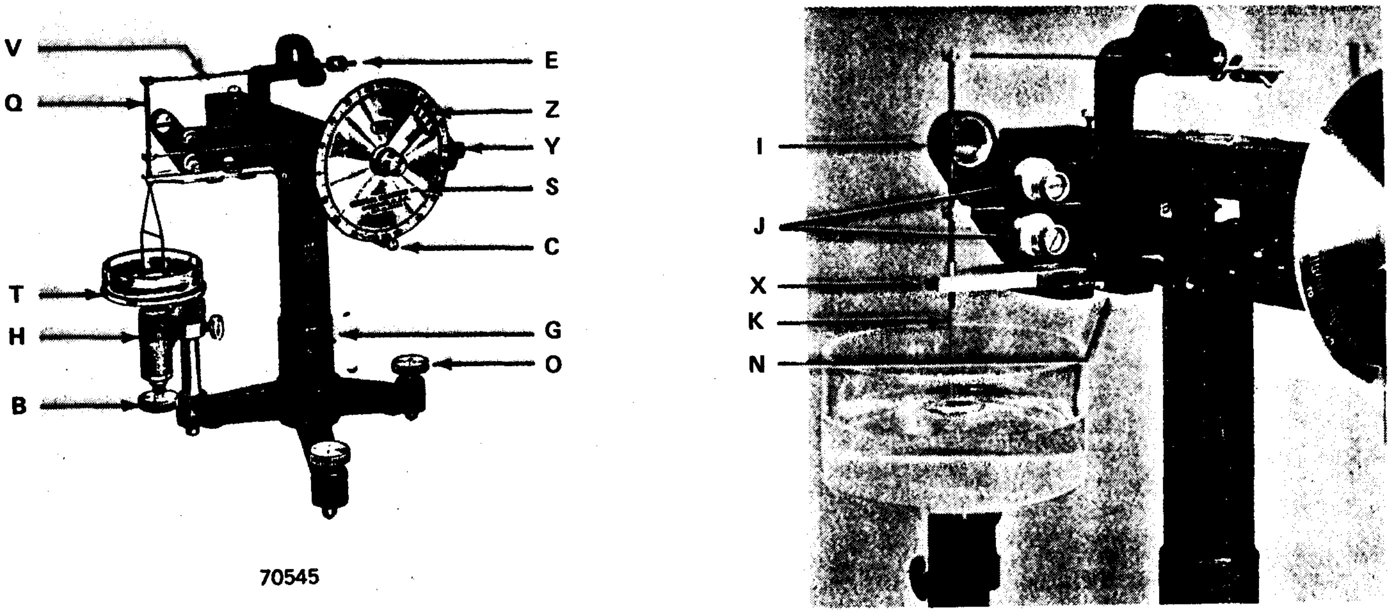


FIGURE 25. CENCO-DU NOUY TENSIOMETER, NO. 70545

Constant temperature bath.

Bunsen burner.

4.A.8.2 Calibration of the Instrument.

4.A.8.2.1 Procedure. Level the tensiometer by adjusting screw O. Check to make sure that the vertical arm is secured by closing the vertical arm restrictions and clamp using screws, and that the dial S is at zero by adjusting screws Y, J, and N. Remove the platinum-iridium ring from the vertical arm at K. Record its ring radius (R in centimetres) and circumference (L in centimetres) and the radius (r in centimetres) of the wire used to form this ring. Calculate and record R/r and R^3. Attach the ring back to the torsion arm at K and bring the sample table assembly to its highest position by adjusting screw H. Bring the sample table to its lowest position by adjusting screw B. Adjust screw G until the ring is approximately 2 to 5 mm above the sample table surface. Release the vertical arm at positions J and N and adjust the counter weights at E until the needle lies on the hairline at I. Clamp the vertical arm using screw N and insert a preweighed, narrow strip of paper on the ring as a platform for weights used in the calibration. Place a 500-mg weight on the paper platform and release the vertical arm using screw N. Apply torsion to the system by adjusting screw Y until the needle lies on the hairline at I. Record the dial reading Z. Clamp the vertical arm using screw N and adjust the torsion screw Y until the dial S is back to zero. Take similar readings using this procedure with several smaller weights. Record the value of each known weight (plus the weight of the paper) and the corresponding dial reading.

4.A.8.2.2 Calculation and preparation of a reference curve. Convert the value of the weights used in the calibration into their corresponding value in dynes/centimetre by the following equation:

$$P = \frac{Mg}{2L} = \text{Calculated surface tension (dynes/cm)}$$

M = weight of the paper strip plus accurately known weights in grams.

g = 980.3 cm/sec^2 (acceleration of gravity).

L = average circumference of the ring in cm.

Example:

$$\text{Weight applied} = 0.600 \text{ gm}$$

$$\text{Weight of paper strip} = \underline{0.018 \text{ gm}}$$

$$0.618$$

$$L = 4.00 \text{ cm}$$

$$g = 980.3 \text{ cm/sec}^2$$

$$P = \frac{Mg}{2L} = \frac{0.618 \times 980.3}{2 \times 4} = 75.7 \text{ dynes/cm}$$

On arithmetic graph paper, plot the values of the weights used (know weights plus weight of the paper platform) against the dial readings and also against the calculated surface tension. Draw straight lines through the origin and through the two sets of data points. These two calibration curves (Figure 26 as an example) and Table 14 of Appendix B are used to convert the dial readings into corrected surface tension values in dynes/centimetres (see Section 4.A.8.4).

4.A.8.3 Measurement of Surface Tension. Check the level of the sample table and adjust screw B (see Figure 25) to position the table to the highest position if necessary. Bring the sample table assembly down to the lowest position by adjusting screw H. Position the dial S at zero by adjusting screw Y. Release the vertical arm restrictions, J, still holding the arm in place by the clamps, X, using screw N. Bring the latex used in Section 4.A.7 (100 ml) to constant temperature of 25° ± 0.5°C by conditioning for 30 minutes in a constant temperature bath at 25.0°C. Pour 25 to 30 ml of the latex into a 4- to 6-cm diameter drying dish until approximately half full. Remove the platinum-iridium ring from its vertical arm at K and heat it in the oxidizing portion of the flame of the bunsen burner until red hot, allow to cool and then attach the clean ring to the vertical arm at K. Place the drying dish containing the latex on the sample table and raise the sample table assembly using screw H until the ring is immersed below the liquid, 2 to 5 mm, and approximately centered with respect to the containers. Release the vertical arm clamp by releasing screw N. Lower the sample table by adjusting screw B until the ring is on the surface of the liquid and the needle lies approximately on top of the hairline at I. Increase the torsion of the wire by rotating screw Y and simultaneously lower the sample table by adjusting screw B while maintaining the needle on top of the hairline at I. The

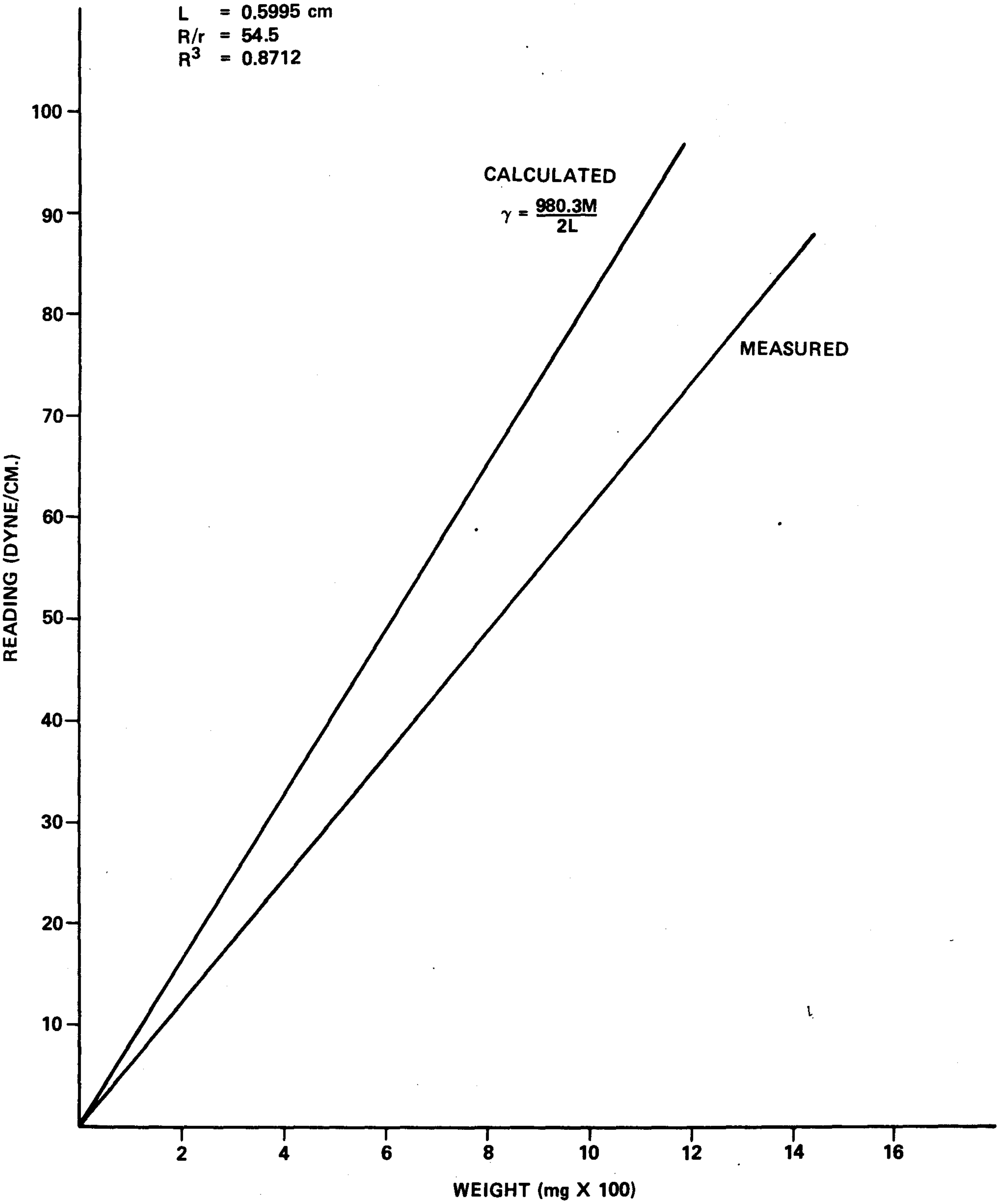


FIGURE 26. SAMPLE TENSIOMETER CALIBRATION CURVE.

needle is to be kept on the hairline at I as the torsion on the wire becomes stronger and the surface of the liquid becomes more distended. As the film adhering to the ring approaches the breaking point, proceed more slowly with the adjustment (B and Y) to make certain that the needle lies directly on the hairline at I when the rupture at the surface occurs and the ring becomes free of the surface. Record the dial reading at Z at the time of the surface rupture. Bring the dial back to zero by adjusting Y. Lower the sample table assembly by adjusting screw H, position the table to its highest position by adjusting screw B, and clamp the torsion arm using screw N. Remove the ring, rinse it with distilled water, and heat it in the oxidizing portion of the flame of the bunsen burner until red hot. Flame only that position of the ring that was and will be immersed in the sample. The ring must be cleaned between each determination. Replace the ring on the torsion arm at K and obtain another dial reading by repeating the above procedure for the same latex sample.

4.A.8.4 Calculations. Average the two values obtained for the sample and round off to the nearest 0.1 dial reading. From the calibration chart, find the corresponding uncorrected surface tension (γ_{un}, dynes/centimetre) and the weight (W in grams) using the average tensiometer dial reading for each latex sample. Using the density of the latex (see Section 4.A.7) and the weight obtained from the chart corresponding to the dial reading. Calculate the volume (V in litres) of the latex. Using the radius (R) of the platinum-iridium ring, calculate R^3/V in cm^3/l. Find the F factor corresponding to R^3/V from Table 14 (Appendix B) and multiply the uncorrected surface tension (γ_{un}) by this factor to obtain the corrected surface tension (γ_{corr}) in dynes/centimetre.

Example: R/r = 54.5

Dial reading = 35
γ_{un} (from chart) = 46.5
W (from chart) = 0.575
Density (D) of Latex (Section 4.A.7) = 1.02
V (W/D) = 0.564
R^3 = 0.871
R^3/V = 1.54
F (from table) = 0.907
γ_{corr} ($F \times \gamma_{un}$) = 42.3

4.A.8.5 Reporting Procedure. Report the corrected average surface tension in dynes/centimetre.

4.A.8.6 Acceptance Criteria. The corrected average surface tension shall be 50.0 dynes/cm or less for an acceptable latex.

4.A.9 Particle Size Distribution

4.A.9.1 Procedure for Obtaining Particle Photographs. Add 1 ml of a latex sample containing 46 percent solids to 100 ml distilled H_2O. Add one to three drops of a 1-percent osmium tetroxide-water solution to the diluted mixture, shake the sample, and store it at room temperature for 1 day. Alternately, place 3 to 5 drops of the diluted latex mixture onto a spot plate, and place the spot plate into a covered container on the bottom of which is a small amount (3 drops) of bromine. A beaker should be used to hold the spot plate off the bottom of the closed container away from the liquid bromine. Remove the spot plate after 5 minutes. Prepare the specimen by spraying a thin film of this diluted latex-osmium tetroxide mixture (or the latex mixture exposed to bromine vapor), approximately one drop, on a round grid (1/8 inch in diameter, or equivalent). The coated grid is then "shadowed" with Pt/C by standard vacuum evaporation techniques to enhance contrast between the particles and background. Photograph the "shadowed" specimen using high resolution transmission electron microscopy at a magnification of 25,000 times. There should be at least 200 individual particles on the photograph completely separated so that precise size measurements can be taken for each particle. If the particles are not well separated, repeat the above procedure with a more diluted latex sample until photographs of well separated particles are obtained (see Figure 16, Section III).

4.A.9.2 Particle Size Measurement and Calculations. Measure and record the "horizontal" diameter in millimetres to the nearest half millimetre of all latex particles on each photograph. Convert each particle size from millimetres (in half-millimetre increments) to Angstrom units by the following formula:

$$\text{Particle size (A°)} = \frac{\text{Particle size (mm)} \times 10^7}{25{,}000}$$

where 25,000 is the magnification factor.

Prepare a histogram of the number of particles of each size for each latex sample tested (see Figures 17 through 21, Section III). The midpoint of the distribution (50 percent of the particles above and 50 percent of the particles below the size) is the median particle size. Calculate the mean particle size by summing the individual particle sizes and dividing by the total number of particles measured (see Table 13). Also calculate the standard deviation from the mean particle size as follows: Multiply the square of the difference between mean particle size and each class interval midpoint by the number of particles of that class interval. Add all the products and divide by the total number of particles measured minus one (see Table 13 for a sample calculation). The square root of this quotient is the standard deviation. Define the 95 percent particle diameter range as follows: Multiply the number of total particles measured by 2.5 percent and round the result to the nearest whole number. Delete each class interval from the histogram in which the sum of the particles in that inteval plus those smaller (on the lower end of the original histogram) or plus those larger (on the upper end of the original histogram) is less than the number of particles calculated above. Subtract the lower limit of the smallest remaining class interval in the histogram from the upper limit of the largest class interval remaining in the histogram to obtain the 95 percent range.

4.A.9.3 Reporting Procedures.

4.A.9.3.1 Report the mean particle size, the median particle size, the standard deviation, and the 95 percent range of particle sizes, all in Angstrom units.

4.A.9.3.2 A histogram containing the number of particles versus particle size shall be included with the test report. Particle sizes in this histogram shall be grouped in class intervals of 200 A°, with the number of particles in each interval plotted at the interval midpoint.

4.A.9.4 Acceptance Criteria.

4.A.9.4.1 The mean and the median particle sizes shall be between 1,400 and 2,500 Angstroms.

4.A.9.4.2 The particle diameter distribution shall be unimodal (i.e., two or more major particle size classes separated by 600 A° or more shall not be present).

4.A.9.4.3 The 95 percent range of particle sizes shall not exceed 2,000 A°.

4.A.10 Latex Freeze-Thaw Stability

4.A.10.1 Procedure. Pour 900 grams of latex (46.0 percent solids), that has been equilibrated to 25° ± 1°C by conditioning in a constant temperature water bath, through a 100 mesh screen and collect the screened latex in a

Table 13. Sample calculation of median and mean particle size standard deviation and the 95 percent range.

Class Interval (A°)	Interval Midpoint (A°)	Number of Particles	Midpoint X Number of Particles	Difference Between the Mean and Interval Midpoint	Difference Squared X Number of Particles
1,500 to 1,700	1,600	5	8,000	623	1,940,845
1,700 to 1,900	1,800	33	59,400	423	5,904,657
1,900 to 2,100	2,000	93	186,000	223	4,624,797
2,100 to 2,300	2,200	73	160,600	23	38,617
2,300 to 2,500	2,400	88	211,200	177	2,756,952
2,500 to 2,700	2,600	15	39,000	377	2,131,935
2,700 to 2,900	2,800	23	64,400	577	7,657,367
2,900 to 3,100	3,000	4	12,000	777	2,414,916
3,100 to 3,300	3,200	2	6,400	977	1,909,058
		336	747,000		29,378,944

Midpoint of the distribution; i.e., 168th particle = median particle size = 2,200 ± 100 A°

747,000/336 = arithmetic mean particle size = 2,223 A°

$(29,378,944/335)^{\frac{1}{2}}$ = standard deviation from the mean = 296 A°

336 x 0.025 = 8.4, rounded = 8 = number of particles to delete from each end of histogram to obtain 95 percent range

95 Percent Range = 2,900 - 1,700 = 1,200 A°

preweighed plastic volumetric flask. Discard the residue on the screen. Alternatively, collect the screened latex used in Section 4.A.5.1 in a preweighed plastic volumetric flask. Weigh the flask containing the latex and calculate and record the weight of the latex. Cover the flask with metal foil such that no dust particles can enter but air can escape from the flask. Place the volumetric flask in a freezer held at 0°F (-18°C). Hold the flask in this environment for 24 hours. No air drafts should be circulating in this environment. Remove the flask with the now frozen latex and set it at room temperature (75°F, 24°C) for 24 hours. At the end of this time, examine the flask and the latex by tilting at a 45° angle and rotating the flask. Note whether any coagulum has formed on the sides of the flask.

Repeat this freeze-thaw procedure a second time and note after the second cycle if any coagulum has formed. After this latex has been at room temperature 24 hours, swirl the latex in the flask to loosen any coagulum adhering to the flask and pour this latex through a 100 mesh screen. Collect the screened latex by means of a funnel and sample bottle and obtain the weight of the residue collected on the 100 mesh screen using the procedure in Section 4.A.5.1.

4.A.10.2 Calculation. Calculate the weight percent of coagulum using the procedure in Section 4.A.5.2.

4.A.10.3 Reporting Procedure. Report the calculated value as weight percent coagulum to the nearest one-thousandth percent. Also report if any significant amount of coagulum was observed adhering to the volumetric flask after either freeze-thaw cycle.

4.A.10.4 Acceptance Criteria. The weight percent coagulum after two freeze-thaw cycles shall not exceed 0.10 percent for an acceptable latex.

Part B. Physical Requirements of the Latex-Modified Concrete

4.B.1 Concrete Mixes

4.B.1.1 Materials for all concrete mixes shall be as follows:

Portland Cement - Type I conforming to ASTM C150-77,

Fine Aggregate - conforming to ASTM C33-77 and having a fineness modulus of 2.6 to 2.9 (ASTM C125-76),

Coarse Aggregate - conforming to ASTM C33-77, 1/2-inch (13-mm) maximum size with the gradation conforming to ASTM C33 (Table 2) size number 7. Further, the coarse aggregate fraction shall have at least 75 percent by weight of particles with at least two fractured faces and 90 percent with one or more fractured faces.

Note: An angular coarse aggregate insures a high water demand for workability.

Water - conforming to 4.1.3 of ASTM C94-74a and having a chloride content of not more than 0.010 percent by weight as measured using the applicable portions of the procedure in Report FHWA-RD-77-85, "Sampling and Testing for Chloride Ion in Concrete," August 1977.

Latex Emulsion - conforming to all requirements of Item 4.A of this specification and having a solids content (4.A.2) of 46.0 percent. If the solids content of the emulsion, as submitted, exceeds 46.0 percent, it shall be reduced to that value prior to use by the addition of distilled water and thorough mixing (see 4.A.3). The deluted emulsion shall be allowed to equilibrate for at least 24 hours before it is used in concrete.

Admixtures - the air entraining admixture for use with the conventional concrete shall conform to the requirements of ASTM C260-74. No other admixtures shall be used.

4.B.1.2 Concretes. Two types of concrete, made using the same cement, water, and aggregates, will be required:

(a) A Latex-Modified Concrete and

(b) An Air-Entrained (AE) Conventional Concrete.

4.B.1.3 Mix Designs. The following mix designs shall be utilized when the latex-modified concrete or the conventional concrete is referenced.

	Solid Volume in $\text{feet}^3/\text{yard}^3$	
Component	Latex-Modified Concrete	AE Conventional Concrete
Cement (658 lb/yd^3)	3.35	3.35
Air	1.35 (5%)	1.62 (6%)
Water	2.43	5.48
Fine Aggregate (SSD*)	9.95	9.93
Coarse Aggregate (SSD*)	6.64	6.62
Latex Emulsion (S.G = 1.01)	3.28	-
Total	27.00 feet^3	27.00 feet^3

*Saturated surface dry condition

To convert ft^3 to m^3, multiply by 0.02832.

To convert lb/yd^3 to kg/m^3, multiply by 0.5933.

Adjustments in the mix designs may be made only in the following instances:

A. Latex-Modified Concrete

(1) To compensate for latex-modified concrete air content other than 5 percent.

(2) The water in the latex-modified concrete mix may be reduced if the slump (ASTM C143-74 measured 4 minutes after completion of mixing) exceeds 6.0 inches (152 mm). The revised water content shall be sufficient to yield a 4- to 6-inch (102- to 152-mm) slump measured 4 minutes after mixing.

In both of the above instances, the adjustment shall not involve a change in the quantity of cement or latex emulsion per cubic yard of concrete. To accomplish the adjustment, the volume of fine and coarse aggregate may be changed, but the fine to coarse aggregate ratio shall be maintained at 60:40 by volume. In no instance shall the water/cement ratio of the latex-modified concrete exceed 0.40 by weight.

B. Conventional Concrete

If the slump of the conventional concrete is less than 3 inches (76 mm), the sand/stone ratio shall be reduced to 50:50 by volume.

The air content (ASTM C231-75) of all air entrained conventional concrete shall be 6 ± 1 percent.

4.B.1.4 Curing Procedures. The curing procedures for each test are presented in the section on that test. However, the following fully describes the phases used and shall be followed when referenced.

1 day wet burlap - a cure for 24 hours after placement in specimen molds covered with wet burlap

lab air - storage at 23 ± 1.1°C and 50 ± 4 percent Rh in a drying room of the type specified in ASTM C157-75, "Length Change of Hardened Cement, Mortar, and Concrete"

moist cure - storage at 23 ± 1.7°C and 95 to 100 percent Rh in a moist room or cabinet of the type specified in ASTM C511-75, "Moist Cabinets and Rooms Used in the Testing of Hydraulic Cements and Concretes"

saturated limewater - storage completely submerged in 73 ± 3°F (23 ± 2°C) water which contains 2 g/l or more lime ($Ca(OH)_2$) in solution

Note: Thus, if a curing procedure is listed as 1 day wet burlap, 27 days lab air, and 14 days saturated limewater, the concrete shall be cured 24 hours in the molds, then in lab air from 24 hours through 28 days, and then in saturated limewater from the 29th through the 42nd day of age. If testing of some specimens is specified during the overall cure period, those particular specimens are simply cured in the manner described until their test age (for example, if half the specimens with the above specified cure were to be tested at 28 days, those particular specimens would be cured 1 day wet burlap, 27 days lab air, and then tested).

4.B.1.5 Mixing Procedures and Equipment. The following batching and mixing sequence shall be used with laboratory pan type or rotary drum mixers:

(a) Latex-Modified Concrete. Add coarse aggregate and latex, mix 1/2 minute; add fine aggregate and cement, mix 1 minute; add water, mix 2.5 minutes.

(b) AE Conventional Concrete. Add coarse aggregate and about one-half the water containing the entire amount of air entraining admixture, mix 0.5 minutes; add fine aggregate and cement, mix 1 minute; add remaining water, mix 2.5 minutes.

Batch size shall be at least 1.5 feet^3 (0.042 m^3) in all instances and the mixer shall be buttered as described in item 1 of Note 12 of ASTM C192-76, "Making and Curing Concrete Test Specimens in the Laboratory."

4.B.2 Fresh Concrete Tests

4.B.2.1 Mixes Required. Both latex-modified and conventional concretes (two batches each) are required.

4.B.2.2 Testing Procedures:

4.B.2.2.1 Prepare a 2.0-foot^3 (0.057-m^3) batch of the subject concrete in a controlled mix room (73 ± 2°F (23 ± 1°C); 50 ± 4 percent Rh) and in accordance with the requirements of 4.B.1.

4.B.2.2.2 Four minutes after completion of mixing, determine the slump (ASTM C143-74) and air content (ASTM C231-75) and then discharge the batch into a nonabsorptive container, sized such that the surface area of concretes exposed to laboratory air shall be approximately 5 feet^2 (0.46 m^2). No other agitation or remixing shall be performed (except that required to remove samples for slump determinations).

4.B.2.2.3 Remove fresh concrete from the container at approximately 15-minute intervals and determine the slump (ASTM C143-74) and note the exact time from completion of mixing until each slump determination. Discard the material used for each determination. Continue until all concrete is used or the slump is 25 percent of the initial value determined 4 minutes after mixing (4.B.2.2.2).

4.B.2.2.4 Define the time after completion of mixing required for 50-percent slump loss by plotting slump versus time, drawing a best-fit curve through the points, and selecting the time from the curve which corresponds to a slump reduction of 50 percent from the initial value determined in 4.B.2.2.2.

4.B.2.2.5 Repeat Steps 4.B.2.2.1 through .4 with three additional batches (i.e., for a total of two latex-modified concrete batches and two conventional concrete batches). Average the times to 50-percent slump loss for each type of concrete.

4.B.2.3 Report:

(a) The initial slump for each batch and the average initial slump for each type of concrete.

(b) The air content of each batch and the average air content for each type of concrete.

(c) The slump loss versus time curve for each batch.

(d) The average time to 50-percent slump loss, T_{ave}, for each type of concrete.

(e) The percent difference (sign included) in Item (d) above for the two types of concretes, calculated as follows:

$$\text{Percent Difference} = \frac{T_{\text{Ave.}}\ \text{Latex} - T_{\text{Ave.}}\ \text{Conventional}}{T_{\text{Ave.}}\ \text{Conventional}} \times 100$$

4.B.2.4 Acceptance Criteria:

4.B.2.4.1 The average initial slump of the latex-modified concrete shall exceed that of the AE conventional concrete.

4.B.2.4.2 The air content of each latex-modified concrete batch shall be less than 9.0 percent.

4.B.2.4.3 The average time to 50-percent slump loss for the latex-modified concrete shall differ from that of the conventional concrete by not more than ±25 percent (i.e., Item (e) reported in 4.B.2.2.5).

4.B.3 Compressive Strength

4.B.3.1 Specimens. Both latex-modified and conventional concretes are required. The specimens shall be 3-inch (76-mm) diameter by 6-inch (152-mm) length cylinders fabricated in accordance with ASTM C192-76. A minimum of three specimens per test age shall be used for each concrete.

4.B.3.2 Test Ages. 24 hours, 28 days, and 42 days.

4.B.3.3 Curing Procedures:

(a) Latex-Modified Concrete. One day wet burlap, 27 days lab air, plus 14 days saturated limewater.

(b) Conventional Concrete. One day wet burlap, 27 days moist cure, plus 14 days saturated limewater.

4.B.3.4 Testing and Reporting Procedure. ASTM C39-72, "Compressive Strength of Cylindrical Concrete Specimens."

4.B.3.5 Calculations. Calculate and report the percent change in average latex-modified concrete compressive strength between 28 and 42 days as follows:

$$\text{Ave. \% change} = 100 \times \frac{(\text{Ave. 42-day str.} - \text{Ave. 28-day str.}}{\text{Ave. 28-day str.}}$$

Retain the plus or minus sign.

4.B.3.6 Acceptance Criteria.

(1) The average compressive strength of the latex-modified concrete at 24 hours and 28 days of age shall be at least 75 percent of the average compressive strength of the conventional concrete of equal age.

(2) The average percent decrease in compressive strength of the latex-modified concrete between 28 and 42 days of age shall not exceed 20 percent.

4.B.4 Flexural Strength

4.B.4.1 Specimens. Both latex-modified and conventional concretes are required. The specimens shall be 3- by 4- by 16-inch (76- by 102- by 406-mm) beams fabricated in accordance with ASTM C192-76. A minimum of three specimens per test age shall be used for each concrete.

4.B.4.2 Test Ages. 24 hours, 28 days, and 42 days.

4.B.4.3 Curing Procedures. Same as 4.B.3.3.

4.B.4.4 Testing and Reporting Procedure. ASTM C78-75, "Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)."

4.B.4.5 Calculations. Calculate and report the percent change in average latex-modified concrete flexural strength between 28 and 42 days as follows:

$$\text{Ave. \% change} = 100 \times \frac{\text{(Ave. 42-day str. - Ave. 28-day str.}}{\text{Ave. 28-day str.}}$$

Retain the plus or minus sign.

4.B.4.6 Acceptance Criteria.

(1) The average flexural strength of the latex-modified concrete at each test age shall be greater than the average flexural strength of the conventional concrete of equal age.

(2) The average percent decrease in flexural strength of the latex-modified concrete between 28 and 42 days of age shall not exceed 25 percent.

4.B.5 Slant Shear Composite Strength

4.B.5.1 Specimens. Each specimen shall be a 3- by 6-inch (76- by 152-mm) composite cylinder consisting of a base cylinder of hardened conventional concrete and an upper portion of latex concrete. Also, complete 3- by 6-inch (76- by 152-mm) latex-modified concrete specimens shall be fabricated from the same concrete batch. A minimum of five specimens of each type shall be used per test age.

4.B.5.2 Test Ages. 24 hours, 28 days, and 42 days.

4.B.5.3 Curing Procedure. Same as 4.B.3.3.

4.B.5.4 Fabrication, Testing, and Reporting Procedures.

4.B.5.4.1 Fabricate fifteen 3- by 6-inch (76- by 152-mm) conventional concrete cylinders in accordance with ASTM C192-76. Cure the cylinders 1 day wet burlap and 27 days moist. Store the cylinders in a moist room until ready for use.

4.B.5.4.2 Within 24 hours of placement of the latex-modified portion of the composite cylinders, cut each conventional concrete cylinder along the diagonal (as shown in Figure 5 of Section II of this report) using a diamond saw. Use sufficient cooling water during cutting to prevent significant heating of the concrete. Discard the top (as originally molded) half of each cylinder and return each bottom half to the moist room until ready for use.

4.B.5.4.3 After preparing a batch of latex-modified concrete, remove the half cylinders from the moist room, dry them to a saturated surface dry condition, and place them in 3- by 6-inch (76- by 152-mm) cylinder molds (cut face up).

4.B.5.4.4 Obtain a small amount of the latex concrete and brush it into the saw cut surface of each half cylinder, and then place the remaining half of each cylinder using the latex-modified concrete and the standard consolidation procedures defined in ASTM C192.

4.B.5.4.5 Using concrete from the same batch, fabricate 15 complete (monolithic) latex-modified concrete cylinders in accordance with ASTM C192-76.

4.B.5.4.6 Cure all cylinders as indicated in 4.B.3.2 and test five bond and five standard cylinders at each test age in accordance with ASTM C39-72, "Compressive Strength of Cylindrical Concrete Specimens." Calculate the compressive force required for failure of each cylinder (composite or monolithic) by dividing the maximum load by the cross sectional area (not the area of the bonding plane). Report the results in accordance with ASTM C39-72.

4.B.5.5 Acceptance Criteria. The average compressive force required to fail the composite cylinders at each age shall be at least 45 percent of the average compressive strength of the full (monolithic) latex-modified concrete cylinders of equal age.

4.B.6 Resistance to Deicer Scaling

4.B.6.1 Specimens. Only latex-modified concrete specimens are required for this test. The minimum dimensions of each specimen shall be 12 by 12 inches (305 by 305 mm) by 3 inches (76 mm) thick. A minimum of three specimens shall be fabricated in accordance with Paragraph 4.2 of ASTM 672-76, "Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals."

4.B.6.2 Test Age. All specimens shall be placed under test at the age of 42 days.

4.B.6.3 Curing Prior to Test. One day wet burlap, 27 days lab air, plus 14 days saturated limewater.

4.B.6.4 Testing and Reporting Procedures. Paragraphs 7 and 8 of ASTM C672-76, "Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals," with the following exceptions:

(1) A 3-percent sodium chloride solution shall be used as the deicing agent and

(2) In accordance with 4.B.6.2 above, the age at the initiation of testing shall be 42 days.

4.B.6.5 Acceptance Criteria. The median scaling rating for the three specimens after 50 daily cycles shall be 3 or less and no single specimen shall exhibit a scaling rating of 5.

4.B.7 Chloride Permeability. Ninety-day ponding with a 3-percent sodium chloride solution.

4.B.7.1 Specimens. Four 12- by 12- by 2-inch (305- by 305- by 51-mm) (or larger) hardened conventional concrete base slabs overlaid with a 1.25-inch (32-mm) thick layer of the latex-modified concrete. Control conventional concrete slabs are also required (see 4.B.7.6).

4.B.7.2 Latex concrete - Curing Prior to Ponding. One day wet burlap and 41 days lab air.

4.B.7.3 Testing Procedure:

4.B.7.3.1 Fabricate and cure (1 day wet burlap, 13 days moist minimum) four 12- by 12- by 2-inch (305- by 305- by 51-mm) thick or larger conventional concrete base slabs.

4.B.7.3.2 Overlay the slabs with a 1.25-inch (32-mm) thick layer of latex-modified concrete. Cure the overlays as indicated in 4.B.7.2 with removal only to accomplish the sandblast specified in 4.B.7.3.3.

4.B.7.3.3 When the overlay is 21 days of age, sandblast or abrade away 1/8 ± 1/16 inch (3 ± 1.6 mm) of each slab surface. No water shall be used in the abrading process. Immediately following the abrading process, return the slabs to lab air to complete the curing described in 4.B.7.2 and the 90-day ponding process. Place dams (approximately 3/4 inch high by 1/2 inch wide (19 by 13 mm)) around three of the specimens prior to 28 days of age.

4.B.7.3.4 On the 43rd day of overlay age, subject the three specimens with dams to continuous ponding to a 1/2-inch (13-mm) depth with a 3 percent (by weight) sodium chloride solution. Continue the ponding for 90 days. Glass plates shall be placed over the three ponded specimens (approximately 1/2 inch (13 mm) above the solution line) to retard evaporation of the solution and additional solution shall be added when necessary to maintain the 1/2-inch (13-mm) depth.

4.B.7.3.5 After 90 days of ponding, the solution shall be removed from the slabs. The slabs shall be allowed to dry and then the surfaces shall be wire brushed until all salt crystal buildup is completely removed.

4.B.7.3.6 Samples for total chloride ion analysis shall then be taken from all slabs in accordance with the procedures described in Report FHWA-RD-77-85, "Sampling and Testing for Chloride Ion in Concrete," Federal Highway Administration, August 1977. Three samples shall be obtained from each of the four (three ponded and one unponded) slabs at each of the following depths:

1/16 inch (1.6 mm) to 0.5 inch (13 mm)
0.5 inch (13 mm) to 1.0 inch (25 mm)

4.B.7.3.7 The total chloride content (percent by weight) of each sample shall be determined in accordance with the procedures in Report FHWA-RD-77-85, referenced above.

4.B.7.4 Calculations

4.B.7.4.1 The baseline chloride ion content for the test specimens shall be determined as the average total chloride ion content of the six samples obtained from the slab that was not ponded with 3 percent NaCl solution.

4.B.7.4.2 The absorbed chloride ion content of each sample from the ponded slabs shall be determined as the difference between the total chloride ion content of that sample and the baseline value calculated in 4.B.7.4.1. If the result is less than zero, the result shall be designated as zero.

4.B.7.4.3 Calculate the average absorbed chloride and the standard deviation of absorbed chloride for the samples from each depth within the ponded slabs.

4.B.7.4.4 Calculate the 95 percent absorbed chloride level for each depth using the information calculated in 4.B.7.4.3 and the following formula:

for each depth given in 4.B.7.3.6:

95 percent Cl^- level = Average absorbed Cl^- + 1.645_(standard deviation of absorbed Cl^-)

4.B.7.5 Reporting. Reporting shall include:

(1) Each total chloride ion value determined in 4.B.7.3.7,

(2) The average and maximum baseline chloride ion (4.B.7.4.1),

(3) Each calculated absorbed chloride ion value determined in 4.B.7.4.2,

(4) The average and standard deviation absorbed chloride ion values calculated in 4.B.7.4.3 for each depth and the maximum absorbed chloride value measured at each depth, and

(5) The 95-percent absorbed chloride level for each depth (4.B.7.4.4).

4.B.7.6 Control Concrete. Using conventional concrete rather than latex-modified concrete, repeat steps 4.B.7.1 through 4.B.7.5 except as follows:

(1) The conventional concrete slabs may be 3-inch (76-mm) thick monolithic slabs or 2-inch (51-mm) conventional concrete overlays on hardened base slabs;

(2) Cure the conventional concrete 1 day wet burlap, 13 days moist, plus 21 days lab air prior to ponding; and

(3) Only one sampling depth, 1/2 to 1 inch (13 to 25 mm), is required.

Note: The purposes of requiring a ponding test on conventional concrete are to provide a basis for comparison and to insure that the test is performed properly.

4.B.7.7 Acceptance Criteria.

(1) The 95-percent absorbed chloride levels for the latex-modified concrete shall be less than each of the following:

1/16- to 1/2-inch depth - 0.320 percent Cl^-
(1.6- to 13-mm)
1/2- to 1-inch depth - 0.0640 percent Cl^-
(13- to 25-mm)

(2) The 95-percent absorbed chloride level for the 1/2- to 1-inch (13- to 25-mm) depth within the conventional concrete shall be at least (i.e., greater than or equal to) 0.160 percent Cl^-.

VII. Certification Program

CERTIFICATION OF STYRENE-BUTADIENE LATEX EMULSIONS FOR BRIDGE DECK OVERLAY CONCRETE

1.0 Scope

1.1 This specification covers the certification testing required on each batch of styrene-butadiene latex emulsion produced for use in bridge deck overlay concrete. A styrene-butadiene emulsion is defined as a latex emulsion in which at least 90 percent of the non-volatiles are styrene-butadiene polymers.

1.2 The specification is applicable only to latex emulsions which were produced in an attempt to duplicate an emulsion which has previously been prequalified by meeting all acceptance criteria listed in Section VI of this report entitled, "Prequalification of Styrene-Butadiene Latex Emulsions for Bridge Deck Overlay Concrete," hereafter referred to as the Prequalification Program.

1.3 The purpose of the certification program is to provide positive indication that each batch of emulsion is similar to the emulsion which was prequalified.

1.4 The certification test results and report shall be applicable for only 1 year following the testing date. If the subject emulsion is not utilized in bridge deck concrete within the 1-year period, all certification testing shall be repeated using a new batch sample; and a new test report must be issued and approved by the engineer prior to use of the emulsion.

2.0 Material for Test and Percent Solids

2.1 Approximately 1 gallon of the latex emulsion shall be obtained from the subject batch by sampling at three or more points within the batch.

2.2 The solids content of the emulsion shall be determined in accordance with paragraph 4.A.2 of the Prequalification Program, and the weight percent solids in the emulsion shall be 46 to 53 percent for acceptance purposes.

2.3 However, for the purposes of the other certification tests listed below, the solids content of the sample shall be 46 percent. If the sample solids content is greater than that value, it shall be reduced to 46.0 weight percent using the procedures given in paragraph 4.A.3 of the Prequalification Program.

3.0 Fingerprint of Material

3.1 Infrared fingerprints of the subject latex emulsion and the alcohol solubles in the emulsion shall be obtained in accordance with paragraphs 3.1 and 3.2 of the Prequalification Program.

3.2 These fingerprints shall be compared to the fingerprints of the latex emulsion which met all acceptance criteria listed in the Prequalification Program. The fingerprints from the subject batch shall be essentially identical to those of the prequalified emulsion in that both the location (frequency) and the general shape of all major peaks and valleys shall be essentially the same. Small variations in the infrared traces caused by normal batch-to-batch and testing variations, as discussed in the section of this report entitled "Latex Emulsions--Chemical and Physical Properties," shall not be cause for rejection.

4.0 Particle Size

4.1 The mean particle diameter of the solids in the subject emulsion shall be determined in accordance with the procedure given in paragraph 4.A.9 of the Prequalification Program.

> Note: Other procedures for determining the particle size (such as light scatter) may be used if the manufacturer provides detailed information defining an acceptable correlation between mean particle size measured by the alternate procedure and that measured by the transmission electron microscope procedure given in paragraph 4.A.8 of the Prequalification Program. Said documentation and a detailed alternate test procedure shall be included in the certification test report.

4.2 The subject batch shall be acceptable from a particle size standpoint if the mean particle diameter for the subject batch is within a ±300 Angstrom range of the mean particle size of the prequalified emulsion and the mean particle diameter is from 1,400 to 2,500 Angstroms.

5.0 pH

5.1 The pH of the subject sample shall be measured in accordance with paragraph 4.A.4 of the Prequalification Program.

5.2 The subject batch shall have an acceptable pH if the sample pH is within ±1.0 pH units of the pH of the prequalified emulsion.

6.0 Viscosity

6.1 The viscosity of the subject sample at an equipment speed of 10 rpm shall be determined in accordance with the procedures in paragraph 4.A.6 of the Prequalification Program.

6.2 The subject batch shall have an acceptable viscosity if the viscosity at 10 rpm is within ±20 centipoise of the viscosity at that speed determined for the prequalified emulsion.

7.0 Surface Tension

7.1 The surface tension of the subject sample shall be determined in accordance with the procedures in paragraph 4.A.8 of the Prequalification Program.

7.2 The subject batch shall have an acceptable surface tension if the surface tension is within ±5 dynes/cm of that determined for the prequalified emulsion, and the surface tension is 50.0 dynes/cm or less.

8.0 Percent Coagulum

8.1 The percent coagulum of the subject samples shall be determined in accordance with the procedures given in Section 4.A.5 of the Prequalification Program.

8.2 Acceptance Criterion. The weight percent coagulum shall not exceed 0.10 percent.

9.0 Freeze-Thaw Stability

9.1 The freeze-thaw stability of the subject sample shall be measured in accordance with the procedures in Section 4.A.10 of the Prequalification Program.

9.2 Acceptance Criterion. The weight percent coagulum after two freeze-thaw cycles shall not exceed 0.10 percent.

10.0 Weight Percent Butadiene

10.1 The weight percent butadiene shall be determined in accordance with the procedures in Section 4.A.1 of the Prequalification Program, with the exception that only three films of the subject latex are required.

10.2 The average weight percentage of butadiene in the latex shall be 30 to 40 percent.

10.3 If the average weight percent butadiene determined using three films does not meet the acceptance criteria, three additional films shall be run in accordance with Section 4.A.1 and the results of all six tests shall be averaged to define whether or not the material is acceptable.

11.0 Reporting

11.1 A test report with batch identification shall be prepared, dated, and signed by the individual(s) responsible for performing the tests. A copy of the test report shall accompany all shipments of the subject latex emulsion and shall be submitted to the engineer for approval prior to use of the emulsion in bridge deck concrete.

Note: Some manufacturers use large holding tanks which may contain a mixture of emulsions from several production batches. In such cases, the test report from each production batch contained in the tank at the time of shipment shall be supplied; or the holding tank shall be sampled shortly before shipment and the certification testing shall be performed on the holding tank sample. In the latter case, no additional material shall be added to the holding tank between the times of sampling and removal of the shipment.

11.2 The test report shall be formulated as follows:

Company: ____________________ Product Name: ____________________

Batch or Shipment Identification: ____________________

Sampling and Test Dates: Sampling ______ Testing ______

	Test Findings		
Certification Test	Prequalified[1] Emulsion	Subject Emulsion	Acceptance Criteria for Subject Emulsion
Weight Percent Solids			46 to 53 percent
Fingerprint of Latex Emulsion	Attached[2]	Attached[2]	Compare Prequalified with Subject - Must be Essentially Identical
Fingerprint of Alcohol Solubles	Attached[2]	Attached[2]	
Mean Particle Size			Prequalified Value ±300A and Mean Particle Size of 1,400 to 2,500A
pH			Prequalified Value ±1.0 pH unit
Viscosity at 10 rpm			Prequalified Value ±20 centipoise
Surface Tension			Prequalified Value ±5 dynes/cm and Surface Tension of ≤ 50 dynes/cm
Percent Coagulum			Less Than or Equal to 0.10 percent
Freeze-Thaw Stability			Less Than or Equal to 0.10 percent Coagulum After Two Freeze-Thaw Cycles
Percent Butadiene			30 to 40 percent

[1] The prequalified emulsion results were obtained during prequalification of the emulsion by an independent laboratory and do not change. Detailed information on prequalification testing is available on request.

[2] Copies of IR fingerprints of the prequalified and the subject batches are on file and shall be supplied upon request. A discussion defining similarities and differences, if any, in the fingerprints, is attached.

I hereby certify that the above and attached tests were performed in accordance with the certification procedures given in Report FHWA-RD-78-35, "Styrene-Butadiene Latex Modifiers for Bridge Deck Overlay Concrete," April 1978.

Signed: ____________________

Title: ____________________

Date: ____________________

APPENDIX A

Preparation of Reference Samples of Known Butadiene Content

Twelve S/B latex references samples with charged butadiene in the butadiene-styrene solids of from 20 to 80 percent (varying in 5 percent increments i.e., 20, 25, 30 . . . . 75, 80) were provided for this study by Reichhold Chemicals, Inc. (18). The samples were used to determine a reference line of butadiene content versus infrared spectra peak ratio (see section 4.A.1 of the Prequalification Program). The procedure for sample preparation is given herein.

Reference sample formulations

The basic formulation used to prepare each reference was:

Ingredients	Parts by Weight
Butadiene	Desired butadiene in percent
Styrene	(100 minus Desired butadiene in percent)
Water	180
Soap	5.0
n-Dodecyl mercaptan	0.5
Potassium Persulfate	0.3

The above formulation can be used to obtain samples containing any butadiene content desired. For example, if 35 percent butadiene was desired, 35 parts by weight butadiene monomer would be used with 65 parts by weight (100 - 35) styrene monomer. Any number of soaps or surfactants could be used, but Dresinate 214 (a rosin acid soap) was used for the reference samples studied herein and performed well.

For accurate reference samples, the above ingredients must be virtually completely polymerized. The degree of polymerization is determined by monitoring the percent solids of the latex emulsion and for the above formulation, 100 percent conversion (polymerization) corresponds to a theoretical solids content of 36.74 percent. A complete description of the sample preparation procedures is given below:

Sample Preparation

For each sample, the ingredients were heated while stirring at 50°C. under either a nitrogen or a carbon dioxide atmosphere for at least 20 hours. After 20 hours reaction time, three 3 ml. samples were taken from the heated mixture using a syringe. Weight percent solids content of each of these samples was then obtained using the procedure in Section 4.A.2 of the Prequalification Program. If the solids content of the mixture, as indicated by the average of the three samples, was above 36.5 percent, the reaction mixture was at least 99.3 percent converted to the butadiene-styrene polymer and the reaction was terminated. If the solids content of this mixture was below 36.5 percent, the reaction was continued (i.e., the mixture continued to be stirred at 50°C. under a nitrogen or carbon dioxide atmosphere) and the weight percent solids content of the mixture was obtained using the above procedure, every 1/2 hour until the solids content was higher than 36.5 percent. In most reactions, 100 percent conversion to the polymer was obtained after 20 hours reaction time. After cooling, the material, thus prepared, was used as a reference sample.

Anyone skilled in the art of the emulsion polymerization should be able to reproduce these latex standards and to obtain polymers that have the appropriate butadiene-styrene ratios.

APPENDIX B

Tables of correction factors (F) for the ring method of determining surface tension.[1]

---

[1] W. D. Harkins and H. F. Jordan, J. Am. Chem. Soc., 52, 1764-68 (1930).
H. W. Fox and C. H. Chrisman, Jr., J. Phys. Chem., 56, 284 (1952).

Table 14

Correction Factors (F) for the Ring Method

R^3/V	R/r = 30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60	65	70	75	80
0.30	1.012	1.018	1.024	1.029	1.034	1.038	1.042	1.046	1.049	1.052	1.054									
.31	1.006	1.013	1.018	1.024	1.028	1.033	1.039	1.041	1.044	1.046	1.049									
.32	1.001	1.008	1.012	1.019	1.023	1.028	1.033	1.035	1.039	1.041	1.045									
.33	0.9959	1.003	1.008	1.014	1.018	1.024	1.028	1.030	1.035	1.036	1.040									
.34	.9913	0.998	1.003	1.010	1.014	1.019	1.023	1.026	1.031	1.032	1.036									
.35	.9865	.993	0.999	1.006	1.008	1.015	1.019	1.022	1.026	1.027	1.031									
.36	.9824	.989	.995	1.002	1.005	1.010	1.015	1.018	1.022	1.024	1.027									
.37	.9781	.985	.991	0.998	1.001	1.006	1.011	1.014	1.018	1.020	1.024									
.38	.9743	.981	.987	.995	0.998	1.003	1.007	1.010	1.015	1.017	1.020									
.39	.9707	.977	.983	.991	.994	0.9988	1.004	1.007	1.011	1.013	1.017									
.40	.9672	.974	.980	.986	.991	.9950	1.000	1.004	1.008	1.010	1.013	1.016	1.018	1.020	1.021	1.022				
.41	.9636	.970	.976	.983	.987	.9922	0.997	1.001	1.005	1.007	1.010	1.013	1.015	1.017	1.019	1.019				
.42	.9605	.968	.973	.980	.984	.9892	.994	0.998	1.002	1.004	1.007	1.010	1.013	1.014	1.016	1.017				
.43	.9577	.964	.970	.977	.981	.9863	.991	.995	.999	1.001	1.005	1.007	1.010	1.011	1.014	1.014				
.44	.9546	.961	.967	.974	.979	.9833	.988	.992	.997	0.998	1.002	1.005	1.007	1.009	1.011	1.011				
.45	.9521	.959	.965	.971	.976	.9809	.986	.990	.993	.996	0.9993	1.002	1.004	1.006	1.009	1.009				
.46	.9491	.966	.962	.969	.973	.9779	.983	.987	.991	.994	.9968	1.000	1.002	1.004	1.006	1.007				
.47	.9467	.954	.960	.966	.971	.9757	.980	.985	.988	.992	.9945	0.998	1.000	1.002	1.004	1.005				
.48	.9443	.951	.957	.963	.968	.9732	.978	.983	.986	.989	.9922	.995	0.997	0.999	1.002	1.003				
.49	.9419	.949	.955	.961	.966	.9710	.976	.981	.984	.987	.9899	.993	.995	.997	1.000	1.001				
.50	.9402	.946	.952	.959	.964	.9687	.973	.978	.981	.985	.9876	.991	.993	.995	.997	.9984				
.51	.9378	.944	.950	.956	.961	.9665	.971	.976	.979	.983	.9856	.989	.991	.993	.995	.9965				
.52	.9354	.942	.948	.954	.959	.9645	.969	.974	.977	.981	.9836	.987	.989	.991	.994	.9945				
.53	.9337	.940	.946	.952	.957	.9625	.967	.972	.975	.979	.9815	.985	.987	.990	.992	.9929				
.54	.9315	.938	.944	.950	.955	.9603	.965	.970	.974	.977	.9797	.983	.986	.988	.990	.9909				
.55	.9298	.936	.942	.948	.953	.9589	.964	.968	.972	.975	.9779	.981	.984	.986	.988	.9892				
.56	.9281	.934	.940	.946	.951	.9567	.962	.966	.970	.974	.9763	.980	.982	.984	.986	.9879				
.57	.9262	.932	.939	.944	.949	.9550	.960	.964	.968	.972	.9745	.987	.980	.983	.984	.9861				
.58	.9247	.930	.938	.942	.947	.9532	.958	.963	.966	.970	.9730	.976	.979	.981	.982	.9442				
.59	.9230	.920	.935	.940	.946	.9515	.956	.961	.965	.968	.9714	.975	.977	.979	.981	.9827				
.60	.9215	.927	.933	.939	.944	.9497	.954	.959	.963	.967	.9701	.973	.976	.978	.979	.9813				
.62	.9184	.924	.930	.936	.941	.9467	.951	.956	.960	.964	.9669	.970	.973	.975	.976	.9784				
.64	.9150	.921	.927	.932	.938	.9439	.948	.953	.957	.961	.9643	.968	.970	.972	.973	.9754				
.66	.9121	.918	.925	.930	.935	.9408	.946	.950	.954	.959	.9614	.965	.967	.969	.971	.9728				
.68	.9093	.915	.921	.927	.932	.9382	.943	.948	.951	.956	.9590	.963	.965	.967	.968	.9703				

Table 14 (continued)

R^3/V	R/r = 30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60	65	70	75	80
.70	.9064	.912	.919	.924	.929	.9352	.940	.945	.949	.953	.9563	.960	.962	.964	.966	.9678				
.72	.9037	.910	.916	.921	.927	.9328	.937	.943	.946	.951	.9542	.957	.960	.962	.964	.9656				
.74	.9012	.907	.913	.919	.924	.9303	.935	.940	.944	.949	.9519	.955	.958	.960	.962	.9636				
.76	.8987	.905	.911	.916	.922	.9277	.933	.938	.942	.947	.9459	.953	.956	.958	.960	.9616				
.78	.8964	.902	.908	.914	.920	.9258	.930	.936	.939	.944	.9475	.951	.954	.956	.958	.9598				
.80	.8937	.900	.906	.912	.918	.9230	.928	.933	.937	.942	.9454	.949	.952	.954	.956	.9581				
.82	.8917	.898	.904	.909	.915	.9211	.926	.931	.935	.940	.9436	.947	.950	.952	.954	.9563				
.84	.8894	.895	.902	.907	.913	.9190	.924	.929	.933	.938	.9419	.946	.949	.951	.953	.9548				
.86	.8874	.893	.900	.905	.911	.9171	.922	.927	.932	.936	.9402	.944	.947	.949	.951	.9534				
.88	.8853	.891	.898	.903	.909	.9152	.921	.926	.930	.934	.9384	.942	.945	.947	.950	.9517				
.90	.8831	.889	.809	.902	.907	.9131	.919	.924	.928	.933	.9367	.940	.943	.946	.948	.9504				
.92	.8809	.887	.809	.900	.905	.9114	.917	.922	.926	.931	.9350	.939	.942	.945	.947	.9489				
.94	.8791	.885	.892	.898	.904	.9097	.915	.920	.925	.929	.9333	.937	.940	.943	.945	.9476				
.96	.8770	.883	.890	.896	.902	.9074	.914	.919	.923	.928	.9320	.936	.939	.942	.944	.9462				
.98	.8754	.882	.888	.894	.900	.9064	.912	.917	.922	.926	.9305	.934	.937	.940	.943	.9452				
1.00	.8734	.880	.886	.892	.899	.9047	.910	.916	.920	.925	.9290	.933	.936	.939	.941	.9438				
1.05	.8688	.875	.882	.888	.895	.9007	.906	.912	.916	.921	.9253	.929	.932	.936	.938	.9408				
1.10	.8644	.871	.878	.885	.891	.8970	.903	.908	.913	.917	.9217	.925	.929	.933	.935	.9378				
1.15	.8602	.867	.875	.881	.888	.8937	.900	.905	.910	.914	.9183	.922	.926	.930	.933	.9352				
1.20	.8561	.864	.871	.878	.885	.8904	.897	.902	.907	.911	.9154	.920	.923	.927	.930	.9324				
1.25	.8521	.860	.868	.875	.882	.8874	.893	.899	.904	.908	.9125	.916	.920	.924	.927	.9300				
1.30	.8484	.856	.864	.871	.879	.8845	.891	.896	.901	.905	.9097	.914	.917	.921	.925	.9277				
1.35	.8451	.853	.861	.869	.876	.8819	.888	.893	.898	.903	.9068	.911	.915	.919	.922	.9253				
1.40	.8420	.850	.858	.866	.873	.8794	.885	.891	.896	.900	.9043	.909	.913	.916	.920	.9232				
1.45	.8387	.847	.855	.863	.871	.8764	.883	.888	.893	.898	.9014	.906	.910	.914	.918	.9207				
1.50	.8356	.844	.853	.861	.868	.8744	.881	.886	.891	.895	.8995	.904	.908	.912	.916	.9190				
1.55	.8327	.841	.850	.858	.866	.8722	.878	.883	.888	.898	.8970	.901	.906	.910	.914	.9171				.9382
1.60	.8297	.839	.848	.856	.863	.8700	.876	.881	.886	.891	.8947	.899	.904	.908	.912	.9152	.922	.928	.933	.9365
1.65	.8272	.836	.845	.853	.861	.8678	.874	.879	.884	.889	.8927	.897	.902	.906	.910	.9133	.921	.927	.931	.9354
1.70	.8245	.834	.843	.851	.859	.8658	.872	.877	.882	.886	.8906	.895	.900	.904	.909	.9116	.919	.925	.930	.9341
1.75	.8217	.831	.840	.849	.857	.8638	.870	.875	.880	.884	.8886	.893	.898	.902	.907	.9097	.918	.924	.929	.9328
1.80	.8194	.829	.838	.847	.855	.8618	.868	.873	.878	.882	.8867	.891	.896	.900	.905	.9080	.916	.922	.927	.9317
1.85	.8168	.827	.836	.845	.853	.8596	.866	.871	.876	.881	.8849	.889	.895	.899	.903	.9066	.915	.921	.926	.9305
1.90	.8143	.824	.834	.843	.851	.8578	.864	.869	.874	.879	.8831	.888	.893	.897	.902	.9047	.913	.919	.925	.9291
1.95	.8119	.822	.832	.841	.849	.8559	.862	.867	.872	.877	.8815	.886	.891	.895	.900	.9034	.912	.918	.923	.9281
2.00	.8098	.820	.830	.839	.847	.8539	.860	.865	.870	.875	.8798	.884	.890	.893	.899	.9016	.910	.917	.922	.9270
2.10	.8056	.816	.826	.835	.843	.8502	.856	.862	.867	.872	.8768	.881	.886	.890	.895	.8991	.908	.914	.920	.9247
2.20	.8015	.812	.822	.831	.839	.8464	.853	.858	.864	.869	.8738	.879	.883	.887	.892	.8962	.905	.911	.917	.9226
2.30	.7976	.808	.818	.828	.835	.8428	.849	.855	.861	.866	.8710	.876	.880	.884	.890	.8935	.903	.909	.915	.9206

Table 14 (continued)

R^3/V	R/r = 30	32	34	36	38	40	42	44	46	48	50	52	54	56	58	60	65	70	75	80
2.40	.7936	.804	.814	.824	.832	.8393	.846	.852	.857	.863	.8680	.873	.878	.882	.887	.8910	.900	.907	.913	.9185
2.50	.7898	.800	.811	.820	.828	.8360	.843	.849	.954	.860	.8651	.870	.875	.879	.884	.8884	.898	.904	.910	.9166
2.60	.7861	.797	.807	.817	.825	.8325	.840	.846	.851	.857	.8624	.868	.872	.877	.882	.8859	.895	.902	.908	.9145
2.70	.7824	.793	.803	.813	.822	.8291	.836	.843	.848	.854	.8598	.865	.870	.874	.880	.8837	.893	.900	.906	.9126
2.80	.7788	.790	.800	.810	.818	.8260	.834	.840	.846	.852	.8570	.862	.867	.872	.877	.8813	.891	.898	.904	.9107
2.90	.7752	.786	.796	.806	.815	.8230	.831	.837	.843	.849	.8545	.860	.865	.870	.875	.8790	.889	.896	.902	.9089
3.00	.7716	.783	.793	.803	.812	.8200	.828	.834	.841	.846	.8521	.858	.863	.868	.873	.8770	.887	.894	.900	.9068
3.10	.7677	.779	.790	.800	.809	.8170	.825	.832	.838	.844	.8494	.855	.860	.866	.871	.8750	.885	.892	.899	.9049
3.20	.7644	.776	.787	.797	.806	.8140	.822	.829	.835	.842	.8472	.853	.858	.864	.869	.8730	.883	.890	.897	.9030
3.30	.7610	.772	.783	.793	.803	.8113	.820	.827	.833	.840	.8449	.851	.856	.862	.866	.8710	.881	.888	.895	.9012
3.40	.7572	.769	.780	.790	.800	.8083	.817	.824	.831	.837	.8424	.849	.854	.860	.864	.8688	.879	.886	.893	.8993
3.50	.7542	.766	.777	.788	.798	.8057	.814	.822	.829	.835	.8404	.847	.852	.858	.862	.8668	.877	.884	.892	.8974

TABLE 14
(continued)

Correction Factors, F, for the Ring Method

R^3/V	R/r 40	50	52	54	56	58	60
3.50	0.8063	0.8407	0.847	0.852	0.858	0.863	0.8672
3.75	0.8002	0.8357	0.842	0.848	0.853	0.858	0.8629
4.00	0.7945	0.8311	0.837	0.843	0.849	0.854	0.8590
4.25	0.7890	0.8267	0.833	0.839	0.845	0.850	0.8553
4.50	0.7838	0.8225	0.829	0.835	0.841	0.847	0.8518
4.75	0.7787	0.8185	0.825	0.832	0.838	0.843	0.8483
5.00	0.7738	0.8147	0.822	0.828	0.834	0.840	0.8451
5.25	0.7691	0.8109	0.818	0.825	0.831	0.837	0.8420
5.50	0.7645	0.8073	0.815	0.821	0.828	0.834	0.8389
5.75	0.7599	0.8038	0.811	0.818	0.825	0.830	0.8359
6.00	0.7555	0.8003	0.808	0.815	0.821	0.827	0.8330
6.25	0.7511	0.7969	0.805	0.812	0.818	0.825	0.8302
6.50	0.7468	0.7936	0.801	0.808	0.815	0.822	0.8274
6.75	0.7426	0.7903	0.798	0.806	0.813	0.819	0.8246
7.00	0.7384	0.7871	0.795	0.803	0.810	0.816	0.8220
7.25	0.7343	0.7839	0.792	0.800	0.807	0.813	0.8194
7.50	0.7302	0.7807	0.789	0.797	0.804	0.811	0.8168

☆U.S. GOVERNMENT PRINTING OFFICE: 1978-261-264/120

FEDERALLY COORDINATED PROGRAM OF HIGHWAY RESEARCH AND DEVELOPMENT (FCP)

The Offices of Research and Development of the Federal Highway Administration are responsible for a broad program of research with resources including its own staff. contract programs. and a Federal-Aid program which is conducted by or through the State highway departments and which also finances the National Cooperative Highway Research Program managed by the Transportation Research Board. The Federally Coordinated Program of Highway Research and Development (FCP) is a carefully selected group of projects aimed at urgent. national problems. which concentrates these resources on these problems to obtain timely solutions. Virtually all of the available funds and staff resources are a part of the FCP. together with as much of the Federal-aid research funds of the States and the NCHRP resources as the States agree to devote to these projects.*

FCP Category Descriptions

1. Improved Highway Design and Operation for Safety

Safety R&D addresses problems connected with the responsibilities of the Federal Highway Administration under the Highway Safety Act and includes investigation of appropriate design standards. roadside hardware. signing. and physical and scientific data for the formulation of improved safety regulations.

2. Reduction of Traffic Congestion and Improved Operational Efficiency

Traffic R&D is concerned with increasing the operational efficiency of existing highways by advancing technology. by improving designs for existing as well as new facilities. and by keeping the demand-capacity relationship in better balance through traffic management techniques such as bus and carpool preferential treatment. motorist information, and rerouting of traffic.

3. Environmental Considerations in Highway Design, Location, Construction, and Operation

Environmental R&D is directed toward identifying and evaluating highway elements which affect the quality of the human environment. The ultimate goals are reduction of adverse highway and traffic impacts. and protection and enhancement of the environment.

4. Improved Materials Utilization and Durability

Materials R&D is concerned with expanding the knowledge of materials properties and technology to fully utilize available naturally occurring materials. to develop extender or substitute materials for materials in short supply. and to devise procedures for converting industrial and other wastes into useful highway products. These activities are all directed toward the common goals of lowering the cost of highway construction and extending the period of maintenance-free operation.

5. Improved Design to Reduce Costs, Extend Life Expectancy, and Insure Structural Safety

Structural R&D is concerned with furthering the latest technological advances in structural designs. fabrication processes. and construction techniques. to provide safe. efficient highways at reasonable cost.

6. Prototype Development and Implementation of Research

This category is concerned with developing and transferring research and technology into practice. or. as it has been commonly identified. "technology transfer."

7. Improved Technology for Highway Maintenance

Maintenance R&D objectives include the development and application of new technology to improve management. to augment the utilization of resources. and to increase operational efficiency and safety in the maintenance of highway facilities.

---

* The complete 7-volume official statement of the FCP is available from the National Technical Information Service (NTIS), Springfield, Virginia 22161 (Order No. PB 242057. price $45 postpaid). Single copies of the introductory volume are obtainable without charge from Program Analysis (HRD-2). Offices of Research and Development. Federal Highway Administration. Washington, D.C. 20590.