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Handbook of Test Methods for Evaluating Chemical Deicers

Cecil C. Chappelow
A. Dean McElroy
Robert R. Blackburn
Midwest Research Institute
425 Volker Boulevard
Kansas City, MO 64110

David Darwin
Frank G. de Noyelles
Carl E. Locke
University of Kansas
Center for Research, Inc.
Lawrence, KS 66045

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Strategic Highway Research Program
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The handbook was prepared by Dr. Cecil C. Chappelow, Senior Advisor for Materials Sciences (MRI), Principal Investigator, with assistance from Mr. Robert R. Blackburn, Head of the Engineering and Statistical Sciences Section (MRI), Dr. A. D. McElroy, Research Associate for Applied Science (MRI), Dr. David Darwin, Professor of Civil Engineering (CRINC), and Dr. Frank de Noyelles, Professor of Botany and Systematics and Ecology (CRINC), who were Co-principal Investigators. Other contributors to the handbook were Dr. Carl E. Locke (CRINC), Mr. Gary R. Cooper (MRI), Ms. Diana Dickerson (CRINC), Dr. Steven L. McCabe (CRINC), Ms. Sonia L. Martinez (CRINC), and Mr. Charles S. Pinzino (MRI).

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Abstract

This handbook contains a structured selection of specific test methods for complete characterization of deicing chemicals. Sixty-two specific test methods are defined for the evaluation of chemical deicers in eight principal property performance areas: 1) physicochemical characteristics; 2) deicing performance; 3) compatibility with bare and coated metals in concrete; 5) compatibility with concrete and nonmetals; 6) engineering parameters; 7) ecological effects; and 8) health and safety aspects.

The 62 specific chemical deicer test methods are composed of 12 primary and 50 supplementary test methods. The primary test methods, which were developed for conducting the more important evaluations, are identified as follows: 1) ice melting tests for liquid and solid deicers; 2) ice penetration tests for liquid and solid deicers; 3) ice undercutting tests for liquid and solid deicers; 4) bare metal corrosion test; 5) concrete degradation test; 6) concrete scaling test; 7) friction characterization test; 8) ecological effects tests; and 9) rebar in concrete corrosion test.

Executive Summary

The chemical deicer user community has not previously had available an organized and accepted set of test methods for evaluating deicing materials. The testing protocols needed by the user community generally encompass (a) the quantification of deicer utility in terms of deicing performance parameters—both theoretical and practical—and (b) the assessment of adverse environmental impacts associated with deicer usage. The tests described in this Handbook are applicable to ice rather than to unconsolidated or compacted snow.

This handbook has been written for local and state highway engineers, chemists, materials scientists, technicians, and other laboratory and research personnel. The handbook assumes that the reader has a general understanding of chemical deicer materials, use, and problems. Relevant background information is provided in a synopsis of current deicing materials and test methods presented in Appendix A.

The main purpose of this handbook is to provide an organized collection of test methods for the complete characterization of deicing chemicals. Sixty-two specific test methods have been defined for the evaluation of chemical deicers in eight principal property/performance areas:

- Physicochemical Characteristics
- Deicing Performance
- Compatibility With Bare and Coated Metals
- Compatibility With Metals in Concrete
- Compatibility With Concrete and Nonmetals
- Engineering Parameters
- Ecological Effects
- Health and Safety Aspects

The 62 chemical deicer test methods are composed of 12 new methods and 50 supplementary methods. The new test methods which were developed on this project specifically to evaluate deicing chemicals are described in detail in Appendix B and identified as follows.

- SHRP H-205.1, Test Method for Ice Melting Capacity of Solid Deicing Chemicals
- SHRP H-205.2, Test Method for Ice Melting Capacity of Liquid Deicing Chemicals
- SHRP H-205.3, Test Method for Ice Penetration of Solid Deicing Chemicals
- SHRP H-205.4, Test Method for Ice Penetration of Liquid Deicing Chemicals
- SHRP H-205.5, Test Method for Ice Undercutting by Solid Deicing Chemicals
- SHRP H-205.6, Test Method for Ice Undercutting by Liquid Deicing Chemicals
- SHRP H-205.7, Test Method for Evaluation of Corrosive Effects of Deicing Chemicals on Metals
- SHRP H-205.8, Test Method for Rapid Evaluation of Effects of Deicing Chemicals on Concrete
- SHRP H-205.9, Test Method for Evaluation of Scaling Effects of Deicing Chemicals on Concrete Surfaces
- SHRP H-205.10, Test Method for Evaluation of Frictional Characteristics of Deicing Chemicals
- SHRP H-205.11, Test Method for Preliminary Assessment of Ecological Effects of Deicing Chemicals
- SHRP H-205.12, Test Method for Evaluation of Corrosive Effects of Deicing Chemicals on Steel Reinforcement in Concrete

The supplementary test methods, which were selected from the literature for performing ancillary evaluations, are listed as references in Appendix C. The supplementary test methods are mainly ASTM standard test methods and EPA standard test procedures and should be given equal weight in selecting the tests a user will perform.

The utility of most of the test methods will be enhanced when the test results are compared to the test results obtained for conventional deicing chemicals. Either sodium chloride or calcium chloride should be used as control or reference materials because they are the principal components of the most widely used chemical deicer formulations.

Laboratory tests are designed to evaluate stated characteristics under controlled, specified conditions, and in most cases, to provide comparative data over an accelerated time interval. Although reasonable attempts have been made to reconcile these approaches with actual field materials and conditions, some differences remain. After initial evaluations in the laboratory, field testing is ultimately required to determine acceptable deicer performance and compatibility.

This handbook contains three sections and three appendices: Section 1–Introduction, Section 2–Testing and Evaluation of Chemical Deicers, Section 3–Test Methods for Evaluating Chemical Deicers, Appendix A–Synopsis of Current Deicing Materials and Test Methods, Appendix B–Primary Test Methods for Evaluating Chemical Deicers, and Appendix C–Supplementary Test Methods for Evaluating Chemical Deicers.

1

Introduction

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- SHRP H-205.4, Test Method for Ice Penetration of Liquid Deicing Chemicals
- SHRP H-205.5, Test Method for Ice Undercutting by Solid Deicing Chemicals
- SHRP H-205.6, Test Method for Ice Undercutting by Liquid Deicing Chemicals
- SHRP H-205.7, Test Method for Evaluation of Corrosive Effects of Deicing Chemicals on Metals
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This handbook contains three sections and three appendices: Section 1—Introduction, Section 2—Testing and Evaluation of Chemical Deicers, Section 3—Test Methods for Evaluating Chemical Deicers, Appendix A—Synopsis of Current Deicing Materials and Test Methods, Appendix B—Primary Test Methods for Evaluating Chemical Deicers, and Appendix C—Supplementary Test Methods for Evaluating Chemical Deicers.

2

Testing and Evaluation of Chemical Deicers

The testing and evaluation of chemical deicers are discussed in seven categories: fundamental properties, physicochemical characteristics, deicing performance, materials compatibility, engineering parameters, ecological effects, and health and safety.

2.1 Fundamental Properties

Chemical deicing technology is based on well-established chemical and thermodynamic principles, which include the following:

2.1.1 Ice Melting Potential

The theoretical potential for chemical ice melting capacities can be defined by low-temperature solubilities in water, molecular or ionic weights of solutes in water, freezing points versus concentration, and eutectic temperature and compositions. Theoretical/fundamental principles and relationships are well established. A substantial body of data is available in the literature and in chemical properties handbooks for a considerable number and variety of chemicals which either have been actively used or tested as deicing materials or might be given consideration. This body of information is largely restricted to single compounds; data on mixtures are generally not available. From a fundamental standpoint, potentially useful deicer chemicals are limited to a relatively small number of compounds, or their mixtures, for the following reasons:

The higher theoretical melting capacities are associated with low molecular or average ionic weights. Chloride salts of the lower atomic weight alkali and alkaline earth metals constitute reference materials for evaluating theoretical melting capacities. Sodium chloride, with an average ionic weight of 29.22 (atomic weight = 59.44), is the benchmark reference in this regard for a practical and reasonably priced deicer. Alkali and alkaline earth salts of acetic acid and formic acid generally offer reasonable theoretical melting capacities. Similarly, a relatively small number of water-soluble, lower-molecular-weight organic compounds—alcohols, amides, and amines—possess reasonable theoretical properties; some of these are employed in special-purpose deicers.

The utility of a deicing candidate material is further constrained by low-temperature solubilities, by concentrations in water associated with freezing points, and by eutectic temperatures which define the lowest temperature at which a material can melt ice or snow. In this regard, the actual theoretical melting capacity can be determined only by freezing point-concentration measurements, particularly at temperatures substantially lower than the normal freezing point of water. Magnesium chloride and calcium chloride have higher theoretical melting capacities per unit weight at temperatures of about 0°F (−17.8°C) than sodium chloride. Similarly, eutectic temperatures and compositions must be determined experimentally, although it is clear that very low eutectic temperatures are associated with high miscibility or solubility in water, and with a high degree of association between solute and water molecules at lower temperatures. The chloride salts of magnesium, calcium, ferric iron, cupric copper, and zinc exhibit eutectic compositions and freezing point depressions substantially lower than predicted via the freezing point depression constant for water ($K_F = 1.86$).

2.1.2 Fundamental Thermodynamic Factors

Thermodynamic aspects of ice melting are qualitatively straightforward but are ill defined in quantitative terms. The important factors are (a) the difference between the thermodynamic activity of water in ice and water in the brine or liquid in contact with ice; (b) the energy required to melt ice; (c) the energy of dissolution or dilution of the deicer in water or brines; and (d) the heat capacities and heat transfer coefficients of the ice melting system.

The energy required to melt water is fixed and the same for all deicers, and heat capacities and heat transfer coefficients will differ little from deicer to deicer. The energies of deicer dissolution, however, are a deicer property that is subject to control or variation by selection of deicers, and insofar as practical, by limiting the exposure of deicer to moisture prior to deicer application. Similarly, the thermodynamic activities of water in ice and in deicer brines are subject to control or variation by deicer selection, by limiting ice melting to temperatures substantially above eutectic temperatures at which activity differences are zero, and by measures which favor or promote rapid dissolution of deicers in brines.

The process of selecting a deicer and of defining the physical form and mode of deicer application and use begins with evaluations and analyses based on these fundamental considerations and associated available data. From a fundamental standpoint, the ideal deicer has a eutectic temperature well below the expected ambient temperature range, dissolves rapidly and exothermically in water and brines, has a low molecular or average ionic weight, and depresses the freezing point of water in high proportions relative to its concentrations in water. The deicer selection process, however, is constrained by other factors: actual performance versus theory, cost and availability, storability, handling and application properties, and the severity and significance of environmental impacts, materials impacts, and safety in manufacture, handling, and use.

2.2 Physicochemical Characteristics

The physicochemical characterization of chemical deicers from a chemical and thermodynamic standpoint potentially requires procedures or methods ranging from procurement of representative samples, through analysis for chemical constituents, to determination of heats of solution and eutectic temperatures. The deicing literature per se is notably devoid of discussion of the appropriate methodologies. These are generally available from sources such as ASTM publications, reference works or standard methods of chemical analysis, and instrument supplier instruction manuals. A few characteristics, such as solubility determinations, are described in general terms in chemistry manuals.

A complete physicochemical characterization of a chemical deicer requires two types of analysis and property determinations. The first consists of identification and quantification of chemical constituents, as follows:

- Principal chemical species identification and quantification.

- Minor chemical species identification and quantification. These potentially include additives and impurities.

- Moisture content including, if appropriate, water of hydration.

- Percent water insolubles.

- Identification of hazardous or toxic constituents.
- pH of aqueous solution.

The second type of measurement or analysis involves the properties which in the aggregate define deicing potential. These are:

- Freezing points and associated deicer concentrations in water.
- Eutectic temperatures and eutectic compositions.
- Solubility, chiefly at temperatures between 0°C and the eutectic temperature.
- Heats of solution in water.
- Viscosities of aqueous deicer solutions.

2.3 Deicing Performance

The fundamental or theoretical properties of deicers define and quantify the limits of ice melting and provide some indication of the probable relationships between these limits and the time-dependent aspects of ice melting. Further, fundamental properties are not rigorously related to the mode of interactions between ice or snow and deicers. Accordingly, methods or approaches capable of measuring and quantifying actual versus theoretical performance properties are needed. One cannot reliably predict, for example, how quickly a given deicer in a specific physical form will interact with ice, nor how it will respond with time at varying ambient temperatures. Further, since control of snow and ice with (or assisted by) deicers seldom involves complete melting of snow or ice, it is difficult to predict or design for the optimum combination of deicer and associated practices.

Several test methods which generally address actual time-dependent deicer performance properties are discussed in the following subsections.

2.3.1 Ice Melting

This test consists of the application of a deicer material to ice, at a specified rate and at a controlled constant temperature, and measurement of volumes of brine melt formed at selected time intervals. Several experimental modifications of this basic method are reported in the literature. Numerous application rates have been employed, usually many times greater than normal highway application rates, and never at a highway application rate of the order of about 400 lb per lane mile (115 kg per lane kilometer). Literature results are consistent with regard to general trends and to comparisons between deicer materials but are quite variable from method to method because of variations in methodologies and test conditions.

A preferred ice-melting rate and capacity test has been identified. The method consists of the addition of deicer to a 1/8-in. (3.175-mm) thick layer of ice contained in a Plexiglas® dish and decantation and measurement of brine volumes via a syringe at selected intervals of time. The method has been utilized with approximately 15 deicer materials at temperatures of 0°, 5°, 10°, 15°, 20°, and 25°F (–18°, –15°, –12°, –10°, –7°, and –4°C). Results are satisfactorily reproducible and have served as the basis for comparing deicers and establishing time-temperature relationships. This method, with some modifications, has been selected as a primary test method.

2.3.2 Ice Penetration

Whereas the ice-melting rate and capacity test measures the quantity of ice melted without regard to the mode of melting, an ice-penetration test is designed for measurement of melting in the vertical or downward mode. Ice penetration is a significant performance property in that a major function of a deicer should be disruption of the ice/pavement interface. The time required to penetrate through ice to the pavement and the fraction of the melting capacity available for subsequent melting at the interface generally define the capability of a deicer to assist in ice-removal processes.

Relatively few ice-penetration test methods have been reported in the literature. The methods differ primarily in the configuration of the ice specimen, that is, in the use of a small slab of free-standing ice or the use of ice formed in a cavity in a material such as Plexiglas®. The latter approach has been selected as the basis for a standardized ice-penetration test. Tests conducted with approximately 15 deicer materials have yielded satisfactorily reproducible results. Significant differences between ice melting and ice penetration are observed in many cases. Some deicers have low penetrating capabilities relative to their ice-melting capabilities, and surface melting occurs almost to the exclusion of penetration as eutectic temperatures are approached.

Some modifications of this ice-penetration test method are recommended. These modifications are designed to better represent the surface of ice normally available to deicer particles and to permit the testing of larger deicer particles.

2.3.3 Ice Undercutting

An ice undercutting test addresses the ability of a deicer to melt ice at the interface between a layer of ice and a pavement substrate. An ice undercutting test for solid deicer particles must include a reproducible means for the particle to initially melt ice and become engulfed in brine. For this reason, the ice undercutting test for solid deicers involves placement of single weighed particles on the surface of a 1/8-in. (0.3175-cm) layer of ice frozen on a substrate, and measurement of the area of the melted ice film generated at the ice/substrate interface subsequent to penetration through the ice layer. The undercutting test with solid deicer particles accordingly represents the extent of undercutting which occurs when deicer particles are distributed over a relatively thin layer of ice.

Liquid deicers will tend to become dispersed as thin films over an ice surface, and to melt ice uniformly from the surface downward. For this reason, an ice undercutting test with liquid deicers consists of placement of measured quantities of the liquid in preformed cavities in the ice; the cavities extend to the base substrate. Ice melting occurs in part by enlargement of the cavity, and in part by melting at the interface between ice and substrate.

With both solid and liquid deicers the undercutting results represent the fraction of the ice melting capacity available for disbondment of ice from the substrate. The undercutting test with liquid deicers does not represent a condition which will exist under normal field application conditions. The undercutting test with solid deicers, on the other hand, closely parallels field application conditions.

2.4 Materials Compatibility

A primary driving force in the search for new or improved deicers is the corrosive or materials-degrading characteristics of chloride-based deicers. Major costs associated with chemical deicers are costs of maintaining and replacing bridge structures and pavements, and the costs of vehicular corrosion. Substantial costs are also associated with practices employed to minimize corrosion and other effects on materials, exemplified by priming and coating metal parts, vehicular design to limit exposure to corrosive chemicals, the use of corrosion-resistant metals or metal surfaces, and the use of air entrained in concrete for pavement construction.

The materials compatibility problem may be subdivided into the following principal areas:

- Bare and coated metals (i.e., metals incorporated in bridge structures, highway appurtenances, and vehicles) (discussed in subsections 2.4.1 and 2.4.2).
- Concrete metal reinforcement corrosion, for which the principal effect is degradation of the metal (2.4.3).
- Concrete or pavement degradation attributable to deicing chemicals (2.4.4).
- Effects on nonmetals such as plastic and rubber (2.4.5).

These types of impacts are well documented, and specific causes of materials degradation are usually well defined and have been related to fundamental processes or mechanisms. While it is quite clear that damage to materials is substantial and costly, both the extent and the economic significance of this damage are subject to debate and differing opinions or interpretations. Accordingly, emphasis in the literature review was placed primarily on methods employed to measure materials degradation processes and the suitability of these methods for use in evaluating chemical deicers.

2.4.1 Compatibility with Bare Metals

Tests of bare metal corrosion basically consist of exposure of metal to a potentially corrosive environment, accompanied by a variety of electrochemical diagnostic measurements, and followed by posttest measurements designed for determination of the extent and type of corrosion. While the tests are simple in principle, a laboratory-based test may not adequately represent or duplicate exposures encountered in actual use conditions. For this reason, numerous exposure protocols have been employed: total or partial immersion; intermittent immersion; spray; air exposure; wet/dry cycling; and exclusion or inclusion of oxygen. Some tests have been designed to duplicate various types of exposure in vehicular corrosion. Corrosion tests should be sensitive to the type of metal, its geometry in a structural member, and its surface characteristics including surface treatments.

Although several standard methods have been accepted by various testing and professional organizations, standard tests that are accepted or validated by these organizations for use with deicing chemicals are not available. It has been concluded, however, that a standardized immersion corrosion test is suitable for evaluation of the corrosive properties of deicing chemicals. The test is designed for determination of the comparative corrosiveness of deicing chemicals, relative in particular to sodium chloride, under controlled and reproducible conditions. The test is not designed for determination of the corrosion under various use conditions.

2.4.2 Compatibility with Coated Metals

Metals are coated primarily to protect the metal against corrosion. Evaluations of the compatibility of chemical deicers with coated metals basically address the effectiveness of the coating in preventing corrosion, but at the same time deal with the integrity or stability of the coating in the chemical environment. Methods available for testing coated metals generally focus on determination of the degree of protection provided by the coating, and on the length of time that a coating will provide satisfactory protection. Electrical and electrochemical techniques are applicable to coated metals. However, the principal approaches employed for evaluating coated metals essentially consist of the imposition of extreme and accelerating environments, exemplified by salt fog/spray cabinet tests, and qualitative/semiqualitative evaluation procedures. A large body of reference or background data is available for numerous coatings and metals; sodium chloride solutions have been extensively tested in such systems. These methods are accepted and well documented, and are suitable for evaluation of chemical deicers.

2.4.3 Compatibility with Concrete Metal Reinforcement

Concrete reinforcement corrosion has been accorded a high priority for several reasons: the corrosive action of deicing chemicals is economically quite important; mitigation or prevention is difficult; and evaluation procedures are difficult, time-consuming, and subject to uncertainties of interpretation.

Experimental procedures must cope with the slowness and variability of the permeation of deicing chemicals through concrete and with the difficulties inherent in designing and using electrochemical diagnostic systems in concrete/reinforcement matrices.

An ASTM procedure is available for evaluating the corrosion status of metal reinforcement in concrete. This method consists of measurement of the potential (electromotive force, EMF) of the metal reinforcement at numerous sites in specimens, and comparison of data with EMF values associated with passive, corrosive, and questionable or uncertain corrosive conditions. The procedure is intended for use on existing structures; that is, no protocol is provided for fabricating and exposing test specimens. The procedure is limited in that results do not provide measures of the extent and rate of corrosion.

Research-oriented approaches to the study of concrete reinforcement corrosion include techniques for isolating liquids (pore solutions) from concrete matrices, and for investigating the corrosive properties of simulated pore solutions, including pore solutions modified by deicing chemical constituents.

Test procedures employed to study corrosion of steel in concrete in simulated bridge decks, either indoors or outdoors, have utilized various configurations and methods for exposing specimens to deicing chemicals. The effect of a deicing chemical on the reinforcing steel is determined by visual observations, electrochemical potential measurements, macrocell current measurements, and postmortems. These tests can be very useful in determining the effects of deicers on corrosion of reinforcing steel. With good quality concrete, however, it can take several years for the deicing chemical to reach the steel reinforcing rod mat.

A recently developed ASTM method which was designed primarily for assessing the corrosion inhibiting or corrosion enhancing properties of admixtures incorporated in concrete has been adopted for evaluating the effect of deicing chemicals on steel reinforcement in concrete. The test does not require sophisticated instrumentation or analytical equipment and can be carried out using equipment normally available in most laboratories. Definitive test results can be obtained usually in less than 1 year.

2.4.4 Compatibility with Concrete

Concrete is subject to some degree of degradation in a non-deicer environment, and generally to more extensive degradation when chemicals are present. Chemicals that are quite incompatible with concrete are clearly unacceptable in a deicer. The mechanisms of concrete degradation are not as well understood as the mechanisms of corrosion. The mechanical forces attending freezing and thawing of wetted concrete are clearly the cause of degradation; this effect is more severe when the water contains chemicals, and air-entrained concrete is considerably less subject to freeze/thaw damage than concrete that is not air-entrained. Degradation of concrete also occurs under wet/dry exposure to chemical solutions, that is, in the absence of freeze/thaw cycles.

Tests which have been employed for determination of concrete compatibility are freeze/thaw tests, wet/dry tests, outdoor exposure tests, and penetration/impregnation tests. Diagnostic or evaluation procedures include visual/spectroscopic tests, abrasion tests, mechanical strength tests, and ultrasonic velocity tests.

The rapid freeze/thaw cycle test recommended by the American Society for Testing and Materials (ASTM C 672-91) is suitable for evaluation of the compatibility of deicing chemicals with concrete, and a substantial inventory of reference results obtained with this test is available. This test may require 50 to 100 cycles over a several week test period.

2.4.5 Compatibility with Nonmetals

The compatibility of chloride based deicing chemicals with plastics and rubber has not been singled out as a significant problem. However, new or modified deicing chemicals will be suspect in this regard. Standard procedures are available for determining the impacts of deicing chemicals on tensile strength and related properties of this class of materials.

2.5 Engineering Parameters

Several parameters of deicer materials pertain to storage and handling characteristics, suitability for use in materials-handling equipment and deicer-dispensing equipment, and efficiency factors such as retention on highway surfaces and the physical and chemical modes of deicer interaction with snow and ice. These parameters include flowability, dusting characteristics, bulk densities, and particle size and size distributions. Methods are available for determination of these properties.

A significant aspect of engineering use parameters is the effect of moisture on the storage and handling characteristics of deicers. Chemical deicers are basically sensitive to moisture, and in the as-received condition may contain varying proportions of water.

Calcium chloride deicers are hygroscopic and accordingly require protection from atmospheric moisture. Rock salt absorbs moisture from the air to the extent of about 0.1 percent by weight at relative humidities less than about 70 percent, but can be wetted with liquid water at concentrations of 1 to 2 percent. Water at the higher levels has an impact on material transfer properties, but more importantly can be the cause of caking or agglomeration. These processes tend to become more severe at low winter temperatures. Wetted rock salt particles also become cemented together when excess moisture is lost by evaporation. Appropriate test procedures are identified for evaluating deicer moisture sensitivities or the effect of moisture and temperature variations on storage and handling characteristics.

The frictional characteristics of pavements under various conditions—wet, dry, snow- or ice-covered with and without abrasives and deicers—have been extensively studied. The frictional property most directly related to deicer selection is the frictional characteristics of pavements wetted with deicer brines or deicer residues. One ASTM procedure was modified during the SHRP H-205 program to serve as a primary laboratory method for measuring this characteristic of deicers.

2.6 Ecological Effects

The literature contains many publications in which ecological effects of deicing chemicals are documented, suggested, or implied. Most studies have focused on the effects of sodium chloride and calcium chloride. There is little unanimity in the literature regarding the extent and significance of environmental effects of sodium chloride. Plant growth and crop productivity in the immediate vicinity of highways treated with deicers are known to be affected, and highway deicing is the source of increases in the sodium chloride salinity levels in streams, lakes, and groundwater aquifers. Increased salt concentrations in drinking water supplies pose a human health problem. Definitive studies of long- and short-term effects on aquatic and terrestrial ecosystems have not been conducted.

Evaluation procedures for determining the ecological effects of deicers are in principle available in the general inventory of methods developed by the U.S. Environmental Protection Agency and others. These methods primarily address materials such as pesticides and require adaptation or modification for testing of the effects associated with deicing chemicals and the ways they are introduced to and disseminated into the environment. Some of these tests were examined and adapted to provide a screening protocol to assess the potential ecological effects of deicers.

2.7 Health and Safety

Deicer chemicals are subject to the standards and procedures pertaining to the manufacture, transport, and use of chemicals. The requirements are essentially that the health and safety hazards or properties of these materials be known and that appropriate protective measures be taken. The deicing chemicals currently in use have been so treated. Under these regulations and standards, new or modified chemicals will need to be characterized and qualified for use.

3

Test Methods for Evaluating Chemical Deicers

In this section, 62 specific test methods for the complete laboratory evaluation of solid and liquid chemical deicers are identified. Relevant background information is provided in a synopsis of current deicing materials and test methods presented in Appendix A.

The 62 specific deicer test methods are divided into two broad groups. The first group includes 12 primary test methods which were developed on this project for conducting the more important evaluations (Appendix B). The second group includes 50 supplementary test methods which were selected from the literature for performing auxiliary evaluations (Appendix C).

The test methods selected for the complete characterization of chemical deicers are discussed in terms of principal evaluation categories:

- Physicochemical Characteristics (3.1)
- Deicing Performance (3.2)
- Compatibility With Bare and Coated Metals (3.3)
- Compatibility With Metals in Concrete (3.4)
- Compatibility With Concrete and Nonmetals (3.5)
- Engineering Parameters (3.6)
- Ecological Effects (3.7)
- Health and Safety (3.8)

The principal elements of each cited test method are identified and summarized. These include but are not necessarily limited to: test description, test objective, equipment and facilities requirements, data output, sources of error, and replication.

3.1 Physicochemical Characteristics

3.1.1 Sampling

Representative samples should be employed in the numerous tests designed to characterize deicer materials. Samples should be representative with regard to particle size, chemical composition, moisture content, and similar sources of variability within a material.

Protocols for obtaining representative samples generally involve mixing by procedures appropriate to the container or stockpile, randomized sampling from designated segments of the material, and ultimate isolation of one or more samples from combined random samples.

Sampling from liquid containers essentially involves thorough mixing of materials in containers, isolation of small samples from randomly selected containers, and isolation of test size samples from combined random samples.

The following ASTM procedures are available for these purposes:

• ASTM D 1568-63	Nonhygroscopic solid deicers: drums and bags
• ASTM D 3665-82	Nonhygroscopic solid deicers: trucks
• ASTM D 75-87	Nonhygroscopic solid deicers: stockpiles, bins, railcars, barges
• ASTM C 702-87	Reduction of large (field) samples to testing size or quantity
• ASTM D 2013-86	Nonhomogeneous samples, grinding and mixing

3.1.2 Deicer Analysis

3.1.2.1 *Moisture Content*

Adsorbed or nonchemically bonded moisture contents of solid deicer materials are determined by drying weighed samples at 230°F (110°C) to constant weights, as specified in ASTM E 534-86 for moisture determination in sodium chloride.

Determination of chemically bound water (water of hydration) requires procedures specific to the material and care to ensure that the hydrated material is not partially decomposed. Accordingly, a standardized procedure for determination of bound water is not presented. Common practice consists of complete analysis of a material and designation of water content by difference.

3.1.2.2 *Water-Insoluble Content*

Deicer materials may contain small percentages of water-insoluble matter. Determination of water-insoluble materials is accomplished by stirring samples in excess water, isolating undissolved matter on a filter, drying, and weighing as specified in ASTM E 534-86. The water-soluble content of the material is also indicated by this procedure.

3.1.2.3 *Chemical Composition*

Procedures for identifying the constituents of chemical deicers and for quantitative measurement are available and described in detail in the analytical chemistry literature. In addition, specific procedures which address the important chemical constituents of products important in certain segments of the chemical use industry are often presented in detail. Standard test methods for the chemical analysis of sodium chloride are presented in ASTM E 534-86; the procedure encompasses moisture-content determination, insolubles determination, and sulfate, magnesium, and calcium analysis, with percent sodium chloride being determined by difference.

A similar procedure for analysis of calcium chloride products is presented in ASTM E 449-90.

A new product, or one which is not known to be available in a normal range with regard to quality and chemical makeup, will normally be subjected to more specific and detailed chemical analysis. Analytical requirements may include identification and quantitation of potentially hazardous trace constituents.

Applicable ASTM procedures include the following:

- ASTM D 3177-89 Sulfur analysis
- ASTM D 3178-89 Carbon and hydrogen determination
- ASTM D 3179-89 Nitrogen analysis

In some cases, metal ions may be analyzed by wet chemical methods as exemplified by titrimetric determination of calcium and magnesium (ASTM E 449-90, ASTM E 534-86). Flame atomic absorption spectrophotometry (AAS) is employed for the quantitative determination of many metals and is the only convenient method for direct determination of alkali metals. An AA method for the determination of sodium is described in ASTM D 4191-82. Atomic absorption methods are described in *Standard Methods for the Examination of Water and Wastewater*, 15th edition, 1980, APHA-AWWA-WPCF (Methods 303 A, 303 B, and 303 C). Methods for the determination of metals in ash from coal or coke are presented in ASTM D 3682-87 and ASTM D 3683-78.

Inorganic anions can usually be determined by wet chemistry procedures. Methods are described in *Standard Methods of Chemical Analysis*, Volume 1, 6th edition, N. H. Furman, Editor. Ion chromatographic techniques are available for determination of chloride, fluoride, nitrate, nitrite, phosphate, and sulfate ions (EPA report EPA-600-14-84-017, Method 300.0). Ion chromatographic methods for organic acids and acid salts have not been published but are available from the Dionex Corporation, Itasca, Illinois.

Independent laboratories which are set up to conduct analysis on a routine basis are sometimes the most efficient and reliable option for the elemental analysis of chemical deicers.

3.1.3 Aqueous Solubility

3.1.3.1 *Test Description*

The test method is employed for determining the solubility of deicer materials in water at temperatures ranging from normal ambient down to eutectic temperatures.

Precise or accurate measurements of solubility involve the careful equilibration of an excess of the material with deionized or distilled water at the temperature of interest, withdrawal of supernatant liquid, and chemical analysis for constituents. This operation is repeated at several temperatures.

Less precise or accurate solubility data may suffice in some cases. For such data, a proximate method is recommended. The proximate method uses multiple specimens containing varying proportions of deicer and water. These specimens are equilibrated to a succession of temperatures, and proximate solubilities are determined by observing points of transition from complete solubility to incomplete solubility.

3.1.3.2 *Test Objectives*

The objectives of solubility measurements are to determine whether a material is sufficiently soluble at low temperatures to merit consideration as a deicer and, in conjunction with freezing point determinations, to define the theoretical limits of a material in terms of ice melting capacities over the temperature range from 32°F (0°C) to the eutectic temperature.

3.1.3.3 *Facilities Requirements*

Solubility measurements require refrigerated systems capable of maintaining constant temperatures down to the eutectic temperature of the water-deicer system. Commercially available constant temperature baths are suitable. A normal complement of laboratory glassware is also required.

Support facilities include equipment to analyze and chemically identify the deicer material prior to initiating solubility tests and to measure the quantities of dissolved materials in saturated aqueous solutions.

3.1.3.4 *Data Outputs*

Primary data consist of the weights of the deicer present in saturated aqueous phases, usually expressed as weight percent of the aqueous phase.

Derived data consist of molecular concentrations of the deicer material. Concentrations may be expressed as the number of molecular weights (moles) of the material dissolved in 1,000 g of water; or more appropriately, for mixed materials or materials which yield more than one discrete dissolved species, as the number of equivalent weights of the dissolved species. One mole of $CaCl_2$ thus contains three equivalent weights, and one mole of NaCl two equivalent weights. Molal or equivalent concentration data may be utilized to estimate freezing points, from the relationship:

$$\Delta T = kC = 1.86C$$

where

T = the freezing point depression in °C,
k = the freezing point depression constant (= 1.86 for water), and
C = the deicer concentration (equivalent weight basis) in equivalents per 1,000 g water.

This expression is reasonably accurate in relatively dilute solutions, for example, for freezing point depressions of the order of 5°C. It is less reliable at higher concentrations.

3.1.3.5 *Sources of Error or Uncertainty*

Solubility measurements and the resulting data are straightforward and subject to no interpretive uncertainties when the material is an essentially pure single material. Deicers will usually not be high in purity and may contain substances added to improve handling and storage properties, to minimize corrosion, or to improve ice melting. They may also contain major proportions of two or more substances (e.g., CMA). Impurities or additives may be readily soluble, sparingly soluble, or essentially insoluble.

Such materials will require care both in the measurement of solubilities and in the interpretation of solubility data. Since the objective of solubility measurements will usually be definition of the probable temperature range and ice melting capacity limits of a material, the impact of impurities or additives on the usefulness of solubility data will be of less concern than it would be if the objective were precise delineation of the phase diagram of a pure material.

Certain preliminary and precautionary measures are nevertheless in order in determination of solubilities of deicer materials. These will vary from material to material, and include:

- Chemical analysis for major and minor constituents, moisture content, and percent water insolubles.

- An obvious excess of the solid phase should be present, to ensure that insoluble materials are not mistaken for an excess of soluble substances.

- Alternatively, concentrated aqueous solutions may be filtered to remove insoluble matter and resulting solutions or recovered soluble solids employed in solubility determinations.

- If soluble impurities—or second and third constituents—are present in greater than minor quantities, the solution phase should be analyzed to determine whether materials are soluble in proportions differing from proportions in the bulk material.

- Data outputs (reported data) should clearly indicate the active (water soluble) ingredients of the deicer materials; the percentage of active ingredients in bulk deicers; and the effective (average) molecular or ionic equivalent weights of dissolved species.

3.1.3.6 *Test Replication*

The number of replicates is user discretionary, and is primarily dependent on the use to be made of solubility data. Well-controlled single tests at four to six temperatures will provide data of the extent and quality needed to screen solubilities. Results to be published should be replicated sufficiently to establish data reproducibility and validity.

3.1.3.7 *Additional References*

Additional information on procedures for determining aqueous solubility are discussed in *Experimental Physical Chemistry*, 6th edition (Daniels et al., 1962), Chapter 6, pp. 124-128.

3.1.4 Freezing Point

3.1.4.1 *Method Description*

ASTM D 1177-88 describes a method to determine the freezing point of engine coolants. This method can be used to determine the freezing point of aqueous solutions of deicer materials without modification.

Deicer solutions of known concentrations are cooled slowly and with stirring while time and temperature are recorded. The freezing temperature is the temperature at which the first ice crystals form.

Near the anticipated freezing point, the deicer solution is "seeded" with frozen crystals of the same deicer solution to prevent supercooling. Cooling of the deicer solution is continued for at least 10 min after the apparent freezing point of the deicer solution. The freezing point is taken as the temperature at the point of intersection of temperature vs. time curves before and after the onset of freezing.

3.1.4.2 *Method Objective*

The objective of ASTM D 1177-88 is to accurately determine the freezing point of an aqueous solution of deicer material.

3.1.4.3 *Facilities and Equipment Requirements*

The equipment requirements described in ASTM D 1177-88 are: (1) a cooling bath consisting of a 2-L Dewar containing dry ice/methanol or liquid nitrogen; (2) a 200-mL clear Dewar in which the deicer solution is placed; (3) a stainless-steel wire coil stirrer driven by a windshield wiper motor; and (4) accurate thermometers or thermocouples.

3.1.4.4 *Data Outputs*

Data outputs for ASTM D 1177-88 will be a record of the deicer solution temperature vs. time. The freezing point of the deicer solution is defined as either the temperature at the intersection of the cooling curve and the flat portion of the freezing curve, or the maximum temperature attained immediately after the deicer solution supercools. Freezing points are recorded for several deicer concentrations and typically are displayed in graphical form.

3.1.4.5 *Sources of Error or Uncertainty*

A major source of error arises from supercooling, which can be minimized by stirring and by seeding with ice crystals prepared from test solutions. Freezing point data are also subject to the accuracy of procedures used to establish solution concentrations.

3.1.4.6 *Method Replication*

Tests should be run in duplicate, and individual solutions should be cycled through freezing temperatures until results are in agreement.

3.1.5 Eutectic Temperature

3.1.5.1 *Method Description*

An aqueous solution of deicer is cooled slowly and stirred until further cooling results in a constant temperature which persists until all water is converted to ice and all solutes are converted to solids. The method and equipment utilized in ASTM D 1177-88 may be employed for eutectic temperature determinations. The eutectic temperatures may be approached by traversing either the freezing point or the solubility curve of a deicer. In the first case, water is gradually separated as ice until the eutectic temperature and eutectic concentration are reached. In the second case, the deicer solute is precipitated gradually until the eutectic composition and temperature are reached.

3.1.5.2 *Method Objective*

The objective of the test is determination of the eutectic temperature of a deicer in water.

3.1.5.3 *Equipment*

Equipment requirements specified in ASTM D 1177-88 are: (1) a cooling bath consisting of a 2-L Dewar containing dry ice/methanol or liquid nitrogen; (2) a 200-mL clear Dewar in which the deicer solution is placed; (3) a stainless steel wire coil stirrer driven by a windshield wiper motor; and (4) accurate thermometers or thermocouples.

3.1.5.4 *Data Outputs*

The result of interest is the eutectic temperature. The eutectic temperature is associated with a eutectic composition determined via the procedure described in Subsection 3.1.6.

3.1.5.5 *Sources of Error or Uncertainty*

A eutectic temperature is an invariant property of a solvent-solute system, and is uniquely associated with a eutectic concentration. Eutectic temperatures and compositions are rigorously associated with single, pure solutes and can be identified for multiple solutes only when solutes precipitate from the solution phase in proportion to solution concentrations as water is converted to ice. Accordingly, reliable determination of a eutectic temperature demands either that the solute be high in purity or that impurities (or other constituents) become essentially insoluble as the eutectic temperature is approached.

Because the eutectic temperature is a property of a pure material, it will usually be determined only for the pure material. Conversely, an apparent eutectic temperature can be identified with a specific compound only to the extent that the purity of the sample is known to be high.

The technique for determination of a eutectic temperature may, however, be employed to locate the lowest temperature at which liquid or brine can coexist with ice for multisolute or impure materials.

Eutectic temperature determination is straightforward in principle, but may be experimentally difficult if solution phases become highly viscous or exhibit gel-like properties as the eutectic temperature is approached.

3.1.5.6 *Method Replication*

A minimum of two determinations of eutectic temperature is recommended.

3.1.6 Eutectic Composition

3.1.6.1 *Method Description*

Eutectic compositions and eutectic temperatures normally will be determined together. The final step in the determination of a eutectic composition consists of measurement of the freezing characteristics of a solution with a concentration identified in preliminary tests as the eutectic concentration. Tests involving slight variations in concentration may be necessary to identify precisely the eutectic composition.

Preliminary identification of eutectic compositions may be effected by (a) careful stepwise addition of solute to dilute solutions and determination of freezing characteristics; (b) careful stepwise dilutions of concentrated solutions; or (c) chemical analyses of solutions at equilibrium with ice and precipitated solute at the eutectic temperatures. Concentrations may be measured by analysis of major constituents, for example, chloride ion; by recovering, drying, and weighing dissolved solids; or by dilution of withdrawn liquid and measurement of solution conductivity.

The method for determining eutectic compositions thus consists of determination of solute concentration at the eutectic temperature as determined via the equipment specified in ASTM D 1177-88 for freezing point determination.

3.1.6.2 *Method Objective*

Eutectic compositions and temperatures define the point of transition, for a deicer, from a capability to melt ice to the lack of this capability. At a temperature slightly higher than the eutectic temperature, the deicer will have a theoretical ice melting capacity slightly higher than that indicated by the eutectic composition. At the eutectic temperature, the ice melting capability is effectively zero because the proportions of brine, ice, and solid deicer can be varied only by withdrawing or supplying thermal energy.

3.1.6.3 *Equipment and Facilities*

The equipment specified in ASTM D 1177-88 for freezing point determination is required, plus glassware and instruments needed for chemical analysis of deicer solutions.

3.1.6.4 *Data Outputs*

Eutectic compositions are usually stated as weight percent solute associated with the eutectic temperature. The weight percent of water, obtained by difference, represents the quantity of ice which theoretically can be at equilibrium with the weight of deicer corresponding to the eutectic weight percent, at the eutectic temperature.

3.1.6.5 *Sources of Error or Uncertainty*

Eutectic temperatures and compositions apply rigorously only to pure solutes. Tests with impure solutes or mixed solutes will yield results which should be interpreted with caution, that is, the results may indicate a lowest achievable temperature which does not constitute a eutectic temperature.

Although eutectic property determinations are simple in principle, experimental difficulties may be encountered if solutions are viscous or gel-like. Isolation of solution free of solids or ice may be difficult, and equilibration of solute concentrations may be sluggish. Substantial excesses of either solids or ice should be avoided by initiating tests with solute concentrations at or near the eutectic composition.

3.1.7 Heat of Solution

3.1.7.1 *Test Description*

Heats of solution are measured using solution calorimeters, which may be purchased, or assembled in laboratories. A solution calorimeter consists of a Dewar flask system contained in a controlled-temperature chamber and protected from significant inputs or withdrawals of thermal energy from the surrounding environment. The system is instrumented to permit accurate calibration of system heat capacities, and to permit accurate measurement of changes in solution temperatures. The receiving solution, usually water, and the test material are brought to the same temperature, the test material is discharged with stirring into the liquid or solution, and temperature changes are recorded. Recorded temperature changes are customarily translated to energy changes by comparison with quantities of electrical (resistance heating) energy required to produce equivalent temperature changes.

The first approach to obtaining this type of thermodynamic data involves a search of the thermodynamics literature. Sources of data are chemistry handbooks, the *International Critical Tables*, NBS publications, and *JANAF Tables of Thermodynamic Data.* These sources typically list the heats of formation of solid or crystalline compounds and compounds in aqueous phases; the differences between values for solids and solutions represent the heat of solution, usually for high dilutions. Experimental determination of heats of solution should seldom be necessary.

3.1.7.2 *Test Objective*

Exothermic dissolution of deicers is preferred, because exothermicity should be associated with a greater degree of interaction between ice and deicer, and because the evolved thermal energy partially counterbalances the endothermicity of ice melting. Measurement or literature derivation of heats of solution is of interest relative to the ice- melting characteristics of deicer materials.

3.1.7.3 *Data Outputs*

Heats of solution are customarily expressed as kilocalories per mole of material.

3.1.7.4 *Sources of Error or Uncertainty*

Heats of solution can be readily determined to the accuracies needed to evaluate deicers. Fundamentally oriented interpretation of heat of solution data demands accurate information on the chemical identities and purities. Particularly important for hydroscopic materials is knowledge of the extent of hydration of a tested material.

3.1.7.5 *Additional Reference*

Additional information on procedures for determining heat of solution can be found in *Experimental Physical Chemistry*, 6th edition (Daniels et al.) Chapter 2, pp. 22-29.

3.1.8 Viscosity of Deicer Solutions

3.1.8.1 *Test Description*

ASTM D 445-88, standard test method for kinematic viscosity of transparent and opaque liquids (and the calculation of dynamic viscosity) is recommended for determining the viscosity of aqueous deicer solutions. In this method, the time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. For deicer brines, viscosities at temperature between 32°F (0°C) and about 0°F (−18°C) are of interest. Brine concentrations of interest are (a) saturated brines at test temperatures, and (b) freezing point concentrations at test temperatures.

3.1.8.2 *Test Objective*

Deicer solution viscosities are potentially important from the standpoint of highway safety. Fluid or nonviscous deicer solutions are preferred for their reasonably rapid rates of ice melting and ice penetration.

3.1.8.3 *Equipment*

Calibrated capillary viscometers, regulated temperature baths or enclosures, accurate thermometers or other available temperature measuring devices, and timers capable of measuring within about 0.2 sec are required.

3.1.8.4 *Data Outputs*

Measured flow times are multiplied by the calibration constant of the viscometer to derive kinematic viscosities.

3.1.8.5 *Sources of Error or Uncertainty*

The method and equipment provide viscosity data of the desired precision or accuracy for deicer brines without requiring accurate control of temperature or deicer concentrations.

3.1.9 pH of Deicer Solutions

ASTM E 70-90 presents a method to measure the pH of aqueous solutions using a glass electrode. pH measurements can be made on aqueous solutions containing high concentrations of inorganic salts or water-soluble organic compounds, or both. The pH and alkalinity or acidity characteristics of deicer brines are potentially significant relative to immediate and long-term impacts on materials and long-term impacts on the environment.

3.2 Deicing Performance

3.2.1 Ice Melting Test (SHRP H-205.1 and H-205.2)

3.2.1.1 *Test Description*

A 1/8-in. (3.175-mm) thick layer of ice is frozen in a flat Plexiglas® dish, and the surface is leveled by partial melting and refreezing with the dish on a level surface. The test specimen is placed in a temperature-regulated enclosure and allowed to equilibrate to the test temperature. The solid or liquid deicer is spread over the surface of the ice. At specified time intervals, melted ice or brine is decanted to the edge of the dish, removed, measured via a syringe, and returned to the test specimen. The normal elapsed time period for the test is 60 min. A minimum of four incremental time intervals is employed to obtain data on rates of ice melting.

3.2.1.2 *Test Objective*

The test provides data on the actual or time dependent quantities of ice melted by a deicer, so that these quantities can be compared to theoretical or equilibrium quantities of ice and can be employed to estimate quantities melted under field conditions within reasonable lengths of time. The test may be employed to establish useful temperature ranges, to compare generic classes of deicers or evaluate new or modified deicers, and to ascertain the effects of variables such as particle size or deicer application rates.

3.2.1.3 *Facilities Requirements*

- A temperature-regulated, low-temperature enclosure with provisions for operator handling of specimens. Temperature requirements include control of temperature to ±0.5°F or better, from 30° to 0°F (−1° to −18°C).

- Calibrated temperature indicators; Plexiglas® test dishes, syringes, timers, and balances for weighing to 0.01 g. The recommended dimensions of circular test dishes are 9-in. (22.86-cm) diameter and 3/4-in. (1.91-cm) deep.

3.2.1.4 *Data Outputs*

Data outputs are:

- Recorded data: brine volume, in milliliters, at time and temperature, for a given weight of deicer.

- Data-reporting options:

  * Brine volumes, in milliliters, at time and temperature, for specified deicer weights.

  * Brine volumes per unit weight of deicer in metric or English units.

  * Conversion of brine volumes to quantities of ice melted per unit weight deicer.

3.2.1.5 *Principal Sources of Error or Uncertainty*

The principal sources of error or uncertainty are as follows.

- Specimen temperature: Brine volumes vary by as much as 10 percent per degree Fahrenheit.

- Low brine volumes at low temperatures.

- Deicer sample integrity: Care should be taken to provide test samples which represent the material and are appropriate to the comparisons to be made.

Comparisons of the melting characteristics of generic classes of solid deicers should be based on specific sieve fractions.

Comparisons of bulk or as-used solid materials should utilize samples with bulk proportions of particle sizes. Particularly to be avoided are nonrepresentative proportions of fine particles and large particles.

Solid deicer materials containing additives, wetted deicers, deicers coated on abrasives, and other modifications will usually pose special problems relative to isolating representative test samples.

3.2.1.6 *Test Replication*

Three replicates are recommended. Standard errors range from about 2 to 10 percent of the average of three replicates. The larger standard errors, on a percentage basis, are usually associated with small brine volumes, that is, at short times and at low temperatures.

3.2.2 Ice Penetration Test (SHRP H-205.3 and H-205.4)

3.2.2.1 *Test Description*

Weighed or size-classified individual solid deicer particles or drops of liquid deicer are placed on the surface of an ice specimen and depths of penetration observed and recorded. After some initial surface melting, the majority of ice melting by solid deicer particles yields a more or less cylindrical cavity filled with deicer brine. The depths of penetration are recorded at 5- to 15-min intervals over a period of 1 hr or more. A dye is employed to facilitate observation of the penetration process.

3.2.2.2 *Test Objective*

A principal mechanism for ice control on highways involves the disbondment of ice from pavement that results when a thin film of melted ice (brine) is generated between ice and pavement. The penetration test provides a measure of the time required for penetration through a given thickness of ice, and gives a rough approximation of the fraction of melting capacity available for undercutting. The test serves to identify temperatures below which penetration is slight and no undercutting and disbondment can be expected, and to identify deicers having a marked tendency to melt surface ice without significant penetration.

3.2.2.3 *Facilities Requirements*

The facilities requirements are:

- A temperature-regulated enclosure capable of control to ±0.5°F (±0.3°C) or better.
- Penetration equipment: The equipment recommended consists of a Plexiglas® plate containing 10 to 15 drilled cavities 5/32 in. (4 mm) in diameter, enlarged at the top by countersinking to a diameter of 0.4 in. (10 mm). Deaerated, deionized water is frozen in

the cavity and the protruding ice is melted to provide a surface level with the top of the cavity. A millimeter grid is placed behind the plate to permit observation and recording of depths of penetration.

3.2.2.4 *Data Outputs*

Primary data consist of depth of penetration at several time intervals, for each temperature, with particle weights or sizes identified.

Data of particular interest are: (a) the time required for penetration through a selected thickness of ice, such as 1/8 in. (3.175 mm), and (b) the depth of penetration in excess of the selected depth after 1 hr on longer. The 1/8-in. thickness data indicate time required for penetration to a pavement surface, and the 1-hr penetration data provide an approximate measure of the melting capacity available for undercutting and disbondment. Recommended data reports are:

- Primary data, the averages of replicates, and standard errors.
- Plots of penetration versus time.
- Time for penetration to a depth of interest.

3.2.2.5 *Important Factors and Sources of Uncertainty*

Penetration depths are not linearly proportional to particle weights. Comparisons of deicers should accordingly be made with particles with specified weights, and weight tolerances within 8 to 10 percent of an average weight (e.g., 25 ± 2 mg). The principal uncertainty in interpreting penetration results arises from the fact that penetration cavities differ in geometric configuration from deicer to deicer. Penetration cavities typically are larger near the surface than at depths of several millimeters; some deicer materials yield penetration tendrils with diameters of 40 to 80 mils (1 to 2 mm), and others yield cavities 120 mils (3 mm) or greater in diameter. The practice of recording maximum depth therefore does not necessarily provide an accurate measure of the usefulness of penetration relative to disbonding ice from pavement.

3.2.2.6 *Test Replication and Reproducibility*

The recommended number of replicates is five.

3.2.2.7 *Data Reproducibility*

The minimum readily observable penetration depth is about 80 mils (2 mm), which can be read to within about ±8 mils (±0.2 mm). Maximum penetration depths, at 60 min and 25°F (–4°C), are of the order of 800 mils (20 mm). Potential observation-related errors accordingly range from about 1 percent to 10 percent of recorded penetration depths.

Standard errors calculated from triply replicated tests of pelletized calcium chloride averaged 2 percent and 5 percent of average values at 20° and 25°F (–7° and –4°C), and 9 percent at 5° and 15°F (–15° and –10°C). Triply replicated tests with halite pellets yielded standard errors which averaged 10 percent of average values, for data obtained at four temperature and six time intervals.

Background data thus indicate standard errors of the following magnitudes.

Penetration depth (mm)	Standard error range (mm)
20	1 - 2
10	0.5 - 1.0
5	0.4 - 0.5

3.2.3 Ice Undercutting Test (SHRP H-205.5 and H-205.6)

3.2.3.1 *Test Description*

The ice undercutting test measures the areas of the brine films formed between a layer of ice and a substrate material. The test utilizes a specified thickness of ice and involves a two-step process with solid deicers. Penetration through the ice to the substrate is followed by the second step, in which a fraction of the melting capacity is expended in melting a thin film of ice between ice and substrate. With liquid deicers, the deicer is placed in a cavity in the ice, that is, the deicer is initially in contact with the substrate.

Various substrates may be employed, but the substrate of choice for deicer evaluation is concrete-based, specifically a mortar mix.

In the recommended test, a 1/8-in. (3.175-mm) thick layer of ice is frozen slowly and in a bottom-up mode on a concrete block. Initially frozen ice in test specimens is partially melted at the surface and refrozen; then the specimens are placed in a regulated temperature enclosure containing a suitable camera. Accurately weighed deicer pellets or liquid samples are placed on dye spots (prepared by freezing a drop of dilute aqueous dye solutions), and photographs are taken at selected time intervals, for 60 min, to record dye patterns.

Photographic slides are projected using a system which permits measurement of the area of the undercut patterns.

3.2.3.2 *Test Objective*

Ice control on highways is primarily achieved, with deicers, by weakening or eliminating the bond between ice and pavement, or alternatively by interfering with the formation of adhering layers of ice or packed snow.

The ice-undercutting test provides a means to measure the extent and rate of undercutting at various temperatures and with various deicing materials. The recommended test utilizes a controlled and uniform ice/substrate condition in order to obtain results or data which reproducibly depict the effects of deicer, temperature, and time. The ice-to-pavement bonding utilized in the test limits the undercutting to a fairly symmetrical area, which may be considerably smaller than undercut areas obtained with the less strongly bonded or less perfect ice formed under natural conditions on highway surfaces.

Undercutting test results are useful for comparing the undercutting capabilities of deicers over the range of temperatures experienced in highway deicing, for defining temperature limitations and strengths of deicers, and in conjunction with field experience, determining the most suitable deicer and estimating optimum application rates for given conditions.

3.2.3.3 *Facilities Requirements*

The facilities requirements are:

- A regulated temperature enclosure, for use at 0° to 25°F (−18° to −4°C), with temperature regulation to ±0.5°F (±0.3°C).
- A bottom-chilled, insulated cold box, with air temperature maintained at about 36°F (2°C), for freezing ice on test specimen substrates.
- Equipment for mixing cement, casting in suitable molds, conditioning surfaces to be contacted with ice, and curing specimens.

3.2.3.4 *Data Outputs*

Primary data consist of undercut areas, in square centimeters, associated with a known deicer particle weight, and with specific temperatures and time intervals.

Derived data may be expressed in a format and in units convenient to the user. Suggested formats are as follows, with time, temperature, and particle weight or size specified:

- cm^2 undercut area/g deicer typical range: 20 to 100 cm^2/g
- m^2 undercut area/kg deicer typical range: 2 to 10 m^2/kg
- ft^2 undercut area/lb deicer typical range: 10 to 50 ft^2/lb

3.2.3.5 *Important Factors and Uncertainties*

The test procedure with solid deicers does not permit direct observation of the time at which penetration to the pavement surface occurs. Plots of undercutting versus time should indicate no undercutting prior to the time that the penetration cavity or core has contacted the surface and a rapid increase to the area of the penetration cavity at the time of initial contact. The undercutting test accordingly should be supported with independently conducted penetration tests in which times are determined for penetration through the thickness of ice employed in undercutting tests.

Background tests, obtained with five replicates at each condition, from 0° to 25°F (−18° to −4°C), yielded standard errors which generally were 3 to 10 percent of the average undercut areas. The majority of the standard errors fell within 3 to 6 percent of average values.

3.2.4 Ice Disbondment Test

The literature describes one method, a drop test, for measuring the capability of deicing materials to destroy or disrupt the bond between ice and a pavement substrate. This method is not deemed suitable for a standard test. Related, ongoing studies are addressing the fundamental aspects of ice and substrate interfaces. In unpublished work, a mechanical disbondment apparatus was recently constructed and employed to measure forces and completeness of removal of ice on concrete treated with several chemical deicers. The ice and concrete specimens were prepared so that the ice was uniformly and strongly bonded to concrete, as in the ice-undercutting test. Uniformly sized deicer particles were placed in symmetrical arrays on the ice, in order to obtain uniform, close-to-complete undercutting. Under these conditions undercutting was confined to reasonably symmetrical circular patterns, and complete undercutting was achieved only when deicer application rates were sufficient to fill the void spaces between the circular patterns.

Disbondment force measurements showed clearly that nonundercut areas required vector forces of the order of 40 to 60 lb/in. of blade width, and that completely undercut areas required essentially no removal force. The results are informative in that they indicate that, under idealized conditions for ice and pavement bonding, the effects of individual deicer particles do not extend beyond the areas defined by undercutting, that is, bonding in adjacent ice is not weakened.

The undercutting test constitutes a much simpler approach for evaluating the disbondment capabilities of deicers and should provide the same type of information on less well-bonded ice. An ice-disbondment test is accordingly not recommended for inclusion in a set of standard test methods. A disbondment test, such as the one based on a laboratory-sized set of loaded wheels, should, however, provide useful and practical information about the undercutting and disbondment properties of deicers on systems subjected to thermal, mechanical, and other types of stress.

3.3 Compatibility With Bare and Coated Metals

3.3.1 Corrosion of Bare Metal (SHRP H-205.7)

3.3.1.1 *Test Description*

Bare metal specimens are immersed in corrosion test solutions for specified time intervals. The primary measure of corrosion is weight loss. Instructions are provided for examination of corroded specimens via low power microscopes, by metallographic sectioning, eddy current electromagnetic inspection, and ultrasonic inspection.

Procedures for immersion corrosion testing are provided in the following general reference test methods:

- ASTM G 31-72 (1990), Standard Practice for Laboratory Immersion Corrosion Testing of Metals.
- ASTM G 1-90, Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens.
- ASTM G 46-76 (1986), Recommended Practices for Examination and Evaluation of Pitting Corrosion.

3.3.1.2 *Test Objective*

Immersion corrosion testing of bare metals provides a means to determine the corrosive characteristics of deicer solutions, including evaluations of alternate deicers or modifications that will minimize corrosion.

3.3.1.3 *Equipment*

Minimum equipment needs are glass vessels, analytical balances, micrometers, low power microscopes, and equipment and chemicals for cleaning or preparing test specimens before and after tests. Optional equipment for examining corroded specimens is specialized and normally available in a metallographic laboratory.

3.3.1.4 *Data Outputs*

Primary weight loss data are customarily converted to annual corrosion rates, for example, mils per year. Weight loss data are augmented to the extent possible by observations of pitting or other types of corrosion.

3.3.1.5 *Sources of Error or Uncertainty*

Corrosion tests are subject to numerous sources of error; adherence to practices recommended in ASTM G 31 and ASTM G 1 is essential. Duplicate tests, and control tests with water and a reference material such as sodium chloride, are recommended.

3.3.2 Corrosion of Coated Metal Via Salt Spray

3.3.2.1 *Test Description*

In ASTM B 117-90, coated or bare metal test specimens are exposed to a salt fog (5 wt percent sodium chloride) environment at 95°F (35°C). This test, modified by substitution of deicer materials for sodium chloride, is recommended for investigations of candidate deicers.

Associated procedures are as follows:

ASTM D 1186-87 presents a method for nondestructively determining the thickness of nonmagnetic coatings on magnetic substrates. The specification provides for two types of coating thickness gauges: (1) magnetic pull-off type, or (2) magnetic flux type. The thickness of magnetic coatings or coatings on nonmagnetic substrates must be determined using suitable micrometers.

ASTM D 1654-79 presents methods for (a) preparing coated specimens prior to salt fog exposure, (b) cleaning the coated specimens after salt fog exposure, and (c) evaluating the specimens. A scribe mark is made through the coating to the metal substrate. Damaged coatings are removed either with a compressed air blast, or by scraping with a dull knife. A rating system (O to 10) is specified for evaluation of the specimens after salt fog exposure, by measurement of the average distance of coating failure away from the scribe mark.

In ASTM D 1654-79, provisions are made for other configurations of scribe marks, including no scribe marks at all. A second rating system, also 0 to 10, is provided in the case of no scribe marks, based on estimation of the percent area of the test specimen with underfilm corrosion or blisters.

ASTM F 502-83 presents a method to determine the effects of cleaning and maintenance materials on painted aircraft surfaces. The effects on the paint are determined by conducting pencil hardness tests on the paint before and after the exposure tests. These procedures should be conducted in conjunction with the salt fog exposure tests to determine effects on coatings, in addition to the corrosion tests.

3.3.2.2 *Testing Objective*

The objective of these test methods is to determine the corrosive effects of deicer materials on coated metal substrates by salt fog testing, and in addition to determine effects on coatings or paints.

3.3.2.3 *Facilities and Equipment Requirements*

The basic equipment consists of a salt fog cabinet, as specified in ASTM B 117-90, augmented by scribing tools, thickness gauges or micrometers, compressed air nozzles, and scraping tools.

3.3.2.4 *Data Outputs*

The primary data output of these ASTM methods is a rating (0 to 10) of the corrosive effects of the deicer material on coated metal substrates. The rating of corrosive effects is determined by either:

- Measuring the average distance of coating failure away from a scribe mark.
- Estimating the percent of specimen area with corrosion effects (rust and blisters) for specimens with no scribe marks.

Secondary data outputs are:

- Visual observations of the specimens for color changes, swelling, and chalking.
- Pencil hardness of paints before and after salt fog exposure.

3.3.2.5 *Sources of Error or Uncertainty*

Preparation of the coated test specimens is the most critical step of salt fog testing. The ASTM methods do not specify methods for coating metals. (ASTM F 502-83 describes the application of an aluminum conversion coating/epoxy primer/polyurethane topcoat system to aircraft aluminum test specimens, which may be of little interest to highway maintenance laboratories.) The coated test specimens for salt fog testing should be prepared according to either (a) state highway department specifications or (b) the coating manufacturer's recommendations.

3.3.2.6 *Test Replication*

A recommended number of replicates is not specified in ASTM B 117-90. However, three to five test specimens for each coating system and each substrate type are considered appropriate. If the deicer material causes considerable corrosion or coating degradation, test specimens for each coating system and each substrate type should be withdrawn and examined at one-fourth, one-half, and three-fourths of the anticipated salt fog test period of 500 hr or longer. Test protocols should include reference tests with sodium chloride salt fogs.

3.4 Compatibility With Metals in Concrete

A variant of the ASTM G 109 test method is recommended for evaluation of the corrosive effects of deicing chemicals on metals in concrete. This adapted test method is summarized in Section 3.4.1. Techniques for evaluating corrosion of rebars in concrete are reviewed in ASTM STP 906, 5-14 (1986).

3.4.1 Corrosive Effects of Deicing Chemicals on Steel Reinforcement in Concrete (SHRP H-205.12)

3.4.1.1 *Test Description*

This test method is a variant procedure within the scope of ASTM G 109. Concrete test specimens (11 x 6 x 4.5 in.) are prepared containing one upper and two lower No 4 rebars (14-in. lengths) which are embedded 0.75 in. and 5.0 in., respectively, beneath the surface and extend about 1.5 in. beyond the end of the specimen. The two lower rebars are connected together with a ground wire, and a 100-Ω resistor is placed between the upper and lower rebars. A chemical deicer solution is ponded on the top surface of the test specimen for two weeks; the solution is drained off, and the specimen is allowed to dry for two weeks. The galvanic current is determined by measuring the voltage across the resistor, and the electrode potential is measured by a calomel reference electrode. The current is monitored as a function of time (cycles) until a marked difference is observed between the reference and the test solution.

3.4.1.2 *Test Objective*

Macrocell current data are used to calculate the corrosion rates of steel rebar embedded in concrete test specimens exposed to ponding solutions of various chemical deicer formulations. Comparative evaluation of the corrosion rate data provides a basis for assessing the relative corrosivity of deicers and the relative effectiveness of corrosion inhibitors.

3.4.1.3 *Equipment*

Concrete mixing, casting and curing equipment; voltmeter with a 1-MΩ input impedance; calomel reference electrode.

3.4.1.4 *Data Output*

The average macrocell currents for each set of control and test samples are plotted vs. time in months. Total integrated macrocell currents and time-to-failure values are calculated in control and test samples.

3.4.1.5 *Sources of Error or Uncertainty*

Faulty test specimens containing cracks and/or other imperfections can give erroneous time-to-failure values. The variability in individual current values as the macrocell approaches a highly active corrosive state creating a degree of uncertainty.

3.4.1.6 *Test Replication and Precision or Bias*

Critical values are provided for establishing the 95 percent repeatability limit (within a laboratory) and the 95 percent reproducibility limit (between laboratories). Multiple tests (2 or 3) are generally indicated as being necessary.

3.5 Compatibility With Concrete and Nonmetals

3.5.1 Rapid Method for Evaluation of Effects of Deicing Chemicals on Concrete (SHRP H-205.8)

3.5.1.1 *Test Description*

Small cylindrical concrete test specimens are prepared and cured in accordance with specified procedures and weighed. Specimens are presoaked 24 hr in deionized water and placed on sponges in circular plastic containers, 4 per container. Deicer solution is added to the test container. The container is covered and subjected to 10 freeze-thaw cycles. Specimens are removed from containers and any spalled material is removed manually from the specimen. The specimens are rinsed, acclimated for 24 hr at 50 percent relative humidity, and then weighed. The weight of spalled material is noted for each specimen, and the mean percent weight loss is determined for each test condition.

3.5.1.2 *Test Objective*

The test measures the resistance of concrete to scaling when exposed to deicing chemical solutions and freeze-thaw cycles. The test may be used to compare the effects of different chemicals at varying concentrations, as well as to evaluate the scaling resistance of different types of concrete.

3.5.1.3 *Equipment and Facilities*

Major items include: (a) concrete mixing equipment, scales, molds for casting specimens, tamping rods, and small tools for finishing concrete; (b) a freezer set to operate at 0°F (−18°C); (c) provisions for monitoring and controlling humidity and temperature; and (d) slump cone (ASTM C 143-90a).

3.5.1.4 *Data Output*

Data output consists of recording the weight of spalled material dirt from each specimen and computing the mean percent weight loss of the four test specimens from a given test container. These values are compared to those obtained using sodium chloride under similar test conditions.

3.5.1.5 *Sources of Error or Uncertainty*

The main sources of error in this test are procedural errors associated with performing the evaluation.

3.5.1.6 *Method Replication*

The test should be conducted using duplicate test cells each containing four test specimens.

3.5.1.7 *Applicable Documents and Procedures*

<u>ASTM Standards</u>

C 31-90	Method of Making and Curing Concrete Test Specimens in the Field
C 33-90	Specification for Concrete Aggregates
C 70-79	Test Method for Surface Moisture in Fine Aggregate
C 125-88	Definitions of Terms Relating to Concrete and Concrete Aggregates

C 127-88	Test Method for Specific Gravity and Absorption of Coarse Aggregate
C 128-88	Test Method for Specific Gravity and Absorption of Fine Aggregate
C 138-81	Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete
C 143-90a	Test Method for Slump of Portland Cement Concrete
C 172-90	Method of Sampling Freshly Mixed Concrete
C 192-90a	Making and Curing Concrete Test Specimens in the Laboratory
C 231-90	Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method
C 330-89	Specification for Lightweight Aggregates for Structural Concrete
C 470-87	Specification for Molds for Forming Concrete Test Cylinders Vertically
C 511-85	Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
C 566-89	Test Method for Total Moisture Content of Aggregate by Drying
C 567-91	Test Method for Unit Weight of Structural Lightweight Concrete
C 617-85b	Practice for Capping Cylindrical Concrete Specimens
D 448-86	Specification for Standard Sizes of Coarse Aggregate for Highway Construction

National Bureau of Standards

Handbook 44 Specifications, Tolerances, and Other Technical Requirements for Commercial Weighing and Measuring Devices

3.5.2 Scaling Effects of Deicing Chemicals on Concrete (SHRP H-20.9)

3.5.2.1 *Test Description*

This test method is a modification of ASTM C 672-91. Concrete specimens 3-in. (7.62-cm) thick, with a test surface area of about 47 in.^{2} (303 cm^2), are prepared and cured according to specified procedures. Deicing brine is ponded on the surface via a suitable dike or reservoir, and the specimens subjected to freeze-thaw cycles as follows: 16 to 18 hr at 0° ± 5°F (−17.8° ± 2.8°C), 6 to 8 hr at 73° ± 3°F (22.8° ± 1.7°C), for 10 or more cycles. At intervals of five cycles, specimens are flushed, visually examined, and the results recorded as a 0 to 5 visual rating. Scaled material is also removed from the surface, collected, filtered, washed, and weighed. The total weight of scaled material is recorded.

3.5.2.2 *Test Objective*

The test provides a measure of the resistance of concrete to scaling when exposed to solutions of deicing chemicals, and freeze-thaw cycling. The test may be employed to compare the effects of different chemicals at varying concentrations, as well as to evaluate the scaling resistance of different types of concrete, including concrete taken from existing structures.

3.5.2.3 *Equipment and Facilities*

Major items include: (a) concrete mixing equipment, scales, molds for casting specimens, tamping rods, and small tools for finishing concrete; (b) a freezer set to operate at 0°F; (c) provisions for monitoring and controlling humidity and temperature; and (d) slump cone (ASTM C 143-90a).

3.5.2.4 *Data Outputs*

Data outputs consist of visual ratings of scaling on a scale of 0 to 5 and weight of scaled material removed from the surface, at intervals of five freeze-thaw cycles, combined with identification of type or history of the concrete specimen and the composition of deicing brines. Photographs and descriptions of surfaces should be included.

3.5.2.5 *Sources of Error or Uncertainty*

Visual ratings are subject to operator bias or interpretation and may not be appropriately subjected to statistical analysis for error or deviation. Weight measurements are quantitative and may be subject to statistical analysis.

3.5.2.6 *Method Replication*

Specifications call for at least two replications for each test condition. Multiple tests will normally be required.

3.5.3 Abrasion Resistance

3.5.3.1 *Test Description*

The test is described in ASTM C 944-90a, Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating-Cutter Method. Cored or fabricated concrete or mortar specimens are abraded in a rotating, abrading cutter, and weight loss and depth of wear are measured.

3.5.3.2 *Test Objective*

The test gives an indication of the relative wear resistance of concrete and mortar. Relative to chemical deicer selection and use, the method constitutes an option for determining the wear resistance of concrete specimens subjected to freeze-thaw tests or for evaluating wear resistance of pavement surfaces exposed to deicing chemicals.

3.5.3.3 *Equipment Requirements*

The basic piece of equipment consists of a rotating cutter drill press, as described in ASTM C 944-90a. Auxiliary equipment includes molds and associated equipment for fabricating specimens or equipment for obtaining core samples, balances for weighing samples prior to and after tests, and instruments for measuring depth of wear.

3.5.3.4 *Data Outputs*

Data reports include average weight loss in grams, or depth of wear in millimeters; documentation of sample size, finish, compaction, age, and strength; and documentation of test conditions.

3.5.3.5 *Precision and Accuracy*

Under normal test conditions the single-operator coefficient of variation is 21 percent; therefore, two tests by one operator should not differ by more than 59 percent of the average. Under severe test condition the single-operator coefficient of variation is 12.6 percent. Therefore, two tests by one operator should not differ by more than 36 percent of the average.

3.5.4 Concrete Mechanical Strength Retention

3.5.4.1 *Test Description*

A compressive axial load is applied to molded cylinders or cores of concrete at a rate within a prescribed range until failure occurs. The compressive strength of the specimen is calculated by dividing the maximum load during the test by the cross-sectional area of the specimen.

The method is described in ASTM C 39-86, Standard Test Method for Compressive Strength Determination of Cylindrical Concrete Specimens.

3.5.4.2 *Test Objective*

The objective of this test is determination of the compressive strength of molded or cored concrete specimens. The method is generally applicable for investigations of the effects of chemical deicers on compressive strengths.

3.5.4.3 *Equipment Requirements*

The principal piece of equipment is a screw-driven or hydraulically-operated test machine capable of applying loads at specified loading rates, continuously and without shock.

Auxiliary equipment includes concrete-mixing equipment, molds and finishing tools, or coring equipment.

3.5.4.4 *Data Outputs*

The primary data output is compressive strength, calculated by dividing maximum load by average cross-sectional area. Supporting data or information include specimen history, specimen dimensions, maximum load, type of fracture, and defects in specimens or cap.

3.5.4.5 *Sources of Error or Uncertainty*

Values obtained depend on specimen size and shape, batching and mixing procedures, fabrication methods, and age, temperature, and moisture conditions. Reproducibility of results for fabricated specimens requires adherence to uniform procedures for fabricating and conditioning specimens and conducting strength tests.

3.5.4.6 *Test Replication and Precision or Bias*

Precision and bias have not been determined. Multiple tests are generally indicated as being necessary.

3.5.4.7 *Applicable Documents and Procedures*

- ASTM C 31-90 Method of Making and Curing Concrete Test Specimens in the Field
- ASTM C 39-86 Standard Test Method for Compressive Strength Determination of Cylindrical Concrete Specimens
- ASTM C 42-90 Method of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
- ASTM C 192-90a Method of Making and Curing Concrete Test Specimens in the Laboratory

3.5.5 Effects of Deicers on Nonmetals

3.5.5.1 *Test Description*

ASTM procedures are available for measuring the tensile properties of plastics (ASTM D 638-90) and rubber (ASTM D 412-87). To determine the effects of deicers on nonmetals, these two tests are recommended in conjunction with materials exposure either via a modified salt fog test (ASTM B 117-90 with deicer substituted for sodium chloride), or by immersion in deicer solutions as in the immersion corrosion tests (SHRP H-205.7 procedure).

In ASTM D 638-90 and ASTM D 412-87, the following properties may be measured or calculated:

- Tensile strength at yield
- Tensile strength at break
- Elongation via an extension measuring device
- Elastic modulus (calculated)

3.5.5.2 *Testing Objective*

The objective of these test methods is to determine the effects of exposure of test specimens to aqueous solutions of deicers on mechanical properties of nonmetals.

3.5.5.3 *Facilities and Equipment Requirements*

The equipment requirements are either a salt fog chamber (ASTM B 117-90) or an immersion system (ASTM G 31-72); and equipment requirements for ASTM D 638-90 and D 412-87, including a testing machine capable of constant crosshead motion, grips to hold the specimen, load indicator (load recording device recommended), micrometers, calipers, cutting die or machine to cut test specimens, conditioning chamber at 73.4° ± 3.6°F (23° ± 2.0°C) and 50 percent ± 5 percent relative humidity, compression set apparatus (optional), and extension measuring device (optional).

3.5.5.4 *Data Outputs*

The data outputs will depend on the equipment used to determine the tensile properties. At a minimum, the data outputs will be:

- Visual observation for changes in the test specimen appearance.
- Maximum load applied to the test specimen.
- Average specimen width in the test area.
- Average specimen thickness in the test area.

The elongation of the specimen can be determined if a suitable extension indicating device is used.

The elastic modulus can be calculated if the applied load is recorded. A suitable recording device records the load on one axis and either time, extension, or strain on another axis.

3.5.5.5 *Sources of Error or Uncertainty*

Tensile properties of plastic and rubber materials are dependent on several factors including method of loading, rate of load application, temperature, humidity, and previous history. Test protocols should include control specimens.

3.5.5.6 *Method Replication*

A minimum of five test specimens is required for determining the tensile properties of plastic materials.

3.6 Engineering Parameters

3.6.1 Frictional Characteristics (SHRP H-205.10)

3.6.1.1 *Test Description*

A laboratory test method has been developed based on ASTM E 303-83, Standard Method for Measuring Surface Frictional Properties Using the British Pendulum Tester. A pendulum tester with a standard rubber slider is used to determine the frictional characteristics of deicer materials on a standard test surface. The pendulum slider is adjusted to barely contact the test surface and raised to a locked position. The test surface (sand-blasted glass) is cleaned and then wetted with the solution to be tested. The pendulum is then released, allowing the slider to make contact with the test surface. The greater the friction between the slider and the test surface, the more the swing is retarded. A drag pointer which moves with the pendulum during the forward part of its swing indicates a scale reading.

3.6.1.2 *Test Objective*

The objective of this frictional characteristics test is to determine whether deicing chemical solutions on pavement surfaces constitute a significant safety hazard.

3.6.1.3 *Equipment*

- British pendulum tester
- Sand-blasted glass (profile 1 to 1.5 mils)
- Standard laboratory equipment for making and storing solutions; thermometer

3.6.1.4 *Data Output*

The drag pointer indicates the British pendulum number (BPN). The greater the friction between slider and surface, the larger the BPN reading. The relationship of BPN units to some "true" value of skid resistance or to coefficients of friction has not been established.

3.6.1.5 *Sources of Error or Uncertainty*

- Temperature at which tests are conducted
- Precise adjustment of the rubber slider path contact length to 4 15/16 in. ± 1/16 in.
- Uniform surface cleaning and deicer application procedures
- Use of "control" solutions

3.6.1.6 *Replication*

A minimum of six trials is required for each test.

3.6.2 Particle Size Distribution

3.6.2.1 *Method Description*

The method and procedures are based on ASTM C 136-84a, Sieve Analysis of Fine and Coarse Aggregates. Representative samples of solid deicers are separated into several size fractions via a succession of standard mesh-size screens. A sample size of 250 to 500 g is recommended to ensure a representative sample

3.6.2.2 *Method Objective*

Particle size analyses are employed to determine whether a commercial deicer conforms to specifications (e.g., ASTM D 632-84 for rock salt), or to determine particle sizes of alternate or developmental deicers in order to obtain information related to deicer handling and application properties.

3.6.2.3 *Equipment Requirements*

Five to six standard mesh screens are required. Mesh sizes correspond to sizes designated for material specifications, or alternatively are selected to provide isolated samples which represent the size distribution of the material. Auxiliary equipment includes balances or scales capable of weighing 500 g to the nearest 0.1 g and an oven for drying certain materials at 230°F (110°C). A mechanical sieve shaker is optional. Hygroscopic materials may require dry boxes for sieving and other handling operations.

3.6.2.4 *Data Outputs*

Results are normally expressed as percent passing each screen employed in the sieve analysis. Other options are percentages retained on individual screens and percentages passing a screen and retained by the next smallest screen.

3.6.2.5 *Sources of Error or Uncertainty*

The principal source of error is the representativeness of the sample employed in the sieve analysis. Another potential source of error may result from the degradation of soft materials during sieving.

3.6.2.6 *Replication*

One carefully conducted test will usually suffice for a given sample. Tests with multiple samples may be required to determine sample-to-sample variations or to characterize bulk materials.

3.6.3 Deicer Particle Shape

Deicer particle shape is generally considered to be significant relative to the mode of ice melting, and possibly relative to ice-melting rates. The deicer materials presently in use or available for use range from irregularly shaped flakes (flake calcium chloride) through spherical particles (pellet calcium chloride, urea, and one manufactured form of CMA). Rock salt particles are generally not spherical. The rock salt particles include fine grains and small platelets; square particles with a height dimension of perhaps 50 percent of a length dimension; flat, generally rectangular particles with sharp corners or edges; and variously shaped chunks having several flat or curved faces. The large particles and the fine particles tend to be uniform in shape; intermediate-size particles are more variable in shape. Uncompacted voids in solid materials may also contribute to particle shape.

A review of the literature did not reveal the existence of a relatively simple method for determining particle shape. Approaches to quantifying or describing particle shape have been described. A two-dimensional approach involves measurement of the perimeter of a particle and comparing the perimeter to the perimeter of a circle of equal area. A second two-dimensional approach consists of dividing length by width. Three-dimensional approaches for irregular particles define shapes in terms of the diameter of a sphere having the same surface area or the diameter of a sphere having the same volume or mass. The three-dimensional approaches primarily define size in terms of equivalent spheres, however.

Techniques for measuring particle dimensions for a two-dimensional approach are simple in principle. For a material such as rock salt, the measurement process will be laborious and time-consuming, however. Given the current lack of documented relationships between deicer performance properties and deicer shape, a standard method for determination of particle shape, based on particle-by-particle dimension measurement, is not included in these methods.

3.6.4 Flow Characteristics

3.6.4.1 *Test Description*

A test method prescribed for measuring apparent densities and pourability of plastic materials (ASTM D 1895-89) is recommended.

Apparent or bulk densities are determined by collecting and weighing a specified volume of material discharged into a container through a funnel, with a drop height of 3.8 cm.

Pourability tests will usually be conducted at temperatures in the region of ambient temperatures anticipated during deicer use. Supporting information includes moisture contents of tested deicer materials, in order to establish the effect of moisture and storage at low ambient temperatures on deicer flow properties.

3.6.4.2 *Test Objective*

The objective of pourability tests is determination of the flowability of deicer materials, and particularly the determination of whether flow properties are affected by storage under a range of temperatures and by changes in moisture contents.

3.6.4.3 *Equipment*

Equipment requirements include funnels of specified dimensions, volume-calibrated collection vessels, timers, balances, and thermometers.

3.6.4.4 *Data Outputs*

Control or reference pourability data are obtained with appropriate reference materials, for example, moisture-free rock salt with a normal particle size distribution. The pourability of test samples taken from incoming supplies or from stockpiles is compared to that of the reference material. Test results include observations of bridging, caking particle agglomeration, and similar impediments to flow.

3.6.4.5 *Sources of Error or Uncertainty*

The flow characteristics of test samples will be affected by mechanical disturbances of the material when samples are taken from stockpiles or containers. Surface samples can be expected to have flow properties different from flow properties of samples taken from the interior of a stockpile. Accordingly, uniform, consistent materials-handling procedures are important, as are records of the origin and history of test samples.

3.6.5 Storage Characteristics

Bulk storage of deicers is preferred for economy and convenience. A covered storage facility should protect deicers from precipitation to prevent loss of materials, as well as dispersal into the environment by dissolution in rainfall or snowmelt. Exposure to precipitation will also normally result in substantial changes in handling properties, which become more severe at low winter temperatures. Changes include caking and apparent freezing because of moisture-activated interparticle interactions which tend to cement particles together.

Some deicer materials, most notably calcium chloride products, have an affinity for atmospheric moisture sufficient to mandate storage in airtight, moisture-proof containers.

Standard tests for evaluating storage characteristics and for determining the moisture sensitivity of deicer materials are not available. Potentially required tests include determination of the existence and identity of solid hydrates and associated temperature-water vapor pressure conditions; determination of temperature and humidity conditions associated with deliquescence for essentially nonhygroscopic materials; determination of the uptake of moisture from ambient air at appropriate temperatures and relative humidities; and determination of whether adsorbed or chemically bound moisture causes caking or agglomeration of particles into larger particles or into mechanically intractable masses.

Procedures or methods for obtaining qualitative or quantitative information on moisture sensitivity are presented in the following subsections.

3.6.5.1 *Pure Compounds Present in a Deicer as Major or Minor Constituents*

The following types of literature data or calculations based on literature data can be used to assess moisture sensitivity.

- The vapor pressures of water in saturated solutions of pure compounds are generally available. Alternately, relative humidities of saturated solutions are reported at various temperatures. Such data translate directly to the temperature and humidity conditions associated with deliquescence, that is, the absorption of moisture vapor from the atmosphere followed by eventual liquefaction. Deliquescence can be expected when the vapor tension of moisture in air slightly exceeds the vapor pressure of water over a saturated solution of the chemical.

- Solid hydrates of numerous chemical compounds are reported in the literature, usually in combination with water vapor tensions associated with given temperatures and specified hydrates.

- Moisture contents of commercial products are usually reported. Such data are indicative of the moisture adsorbing and retention capacities of materials under normal conditions of handling and storage.

3.6.5.2 *Experimental Methods For Determining Moisture Uptake*

Desired information will normally address moisture sensitivity at normal storage or use temperatures and associated relative humidities.

A basic procedure suitable for this purpose consists of drying a weighed sample at 230°F (110°C), reweighing to determine the as-received moisture content, exposing the dry material at a fixed relative humidity and temperature, and weighing at selected time intervals until moisture uptake ceases or until evidence has been obtained for deliquescence or the formation of solid hydrates.

Constant relative humidities, ranging from about 40 percent to 98 percent over a temperature range from 32° to 158°F (0° to 70°C), can be provided with glycerin-water solutions.

Instructions for preparation and use of the solutions are given in ASTM E 104-85, Maintaining Constant Relative Humidity by Aqueous Solutions. A reservoir of the appropriate solution is placed in an airtight enclosure, such as a glass desiccator, and open samples of test materials placed in the enclosure. Data of the required accuracy and completeness will usually be obtained with tests conducted at normal laboratory temperature and at perhaps two temperatures in a refrigerator.

Data obtainable from such tests include:

- Rates of moisture uptake.
- Moisture adsorption capacity (that is, adsorbed but not chemically bound water).
- Solid hydrate formation. Identification of specific hydrates and of associated water vapor tensions will, however, require carefully controlled conditions and a greater number of tests.
- Conditions for deliquescence.

3.6.5.3 *Determination of Storability Characteristics*

Laboratory representation of the range of temperature, moisture contents and humidities, and materials packing conditions encountered in bulk storage is clearly not possible. Nevertheless, certain simple tests can be conducted to obtain evidence of changes in deicer materials properties accompanying temperature variations. A recommended test protocol follows.

- Determine the particle size distribution of as-received or candidate deicer materials.
- Characterize the material by chemical analysis and moisture determination.
- Prepare 50- to 100-lb batches of material with a range of moisture contents, packaged in a container designed to retain moisture.
- Cycle the materials between a normal ambient temperature and a specified low temperature for time periods of 5 to 7 days at each temperature.

- After each part of the cycle, examine and test the material to ascertain mechanical properties: resistance to flow, hand scoopability and spreadability, the presence and size of clumps, fracturability under stresses (exemplified by dropping a bag or barrel), and sieving to determine changes in particle size and particle size distribution. Flow characteristics (pourability) can be established via procedures presented in 3.6.4.

The test cycle should include return to warm ambient temperature conditions, in order to determine whether changes reverse on warming.

Adaptations of the test procedure may be employed to evaluate other aspects of storability. Some examples follow:

- An as-received material may contain higher than normal moisture contents. Evaporative losses may be accompanied by the cementing together of particles.

- Exposure of stored materials at high relative humidities may result in significant moisture uptake and in the onset of deliquescence, followed by evaporative losses at low relative humidities and by particle agglomeration.

- Additives introduced for purposes such as corrosion inhibition may significantly affect storage characteristics. Tests of moisture absorbency characteristics, followed by temperature and moisture content cycling, will define storage characteristics.

3.6.6 Dusting Susceptibility

3.6.6.1 *Test Description*

In ASTM D 4331-84 a sample of granular solid material is placed in a vertical fluidizing tube equipped with a vibrator, a controlled air supply, flowmeters, dust filters, and a dust sampling pump. Air is passed through the sample, and suspended dusts collected, weighed, and analyzed as appropriate.

3.6.6.2 *Test Objective*

The objective of the test is determination of deicer dusting tendencies relative to deicer materials loading or transfer and application to highways.

3.6.6.3 *Equipment*

- Vertical-fluidizing tube on ring-stand assembly, as specified in ASTM D 4331-84, with accessories
- Timers
- Analytical balances

3.6.6.4 *Data Reports*

Weights of collected dust are reported, together with a complete description of the sample and test conditions.

3.6.6.5 *Sources of Error or Uncertainty*

Dusting susceptibilities are significantly affected by sample moisture contents and humidity levels in air used to entrain dusts. Precision and bias are unknown.

3.6.6.6 *Test Replication*

A minimum of five replicates is specified.

3.6.7 Specific Gravity of Liquids

ASTM D 1122-90a describes a method to determine the specific gravity of engine coolants, such as antifreeze. The method is applicable for determining the specific gravity of liquid deicer materials. The procedure involves cooling the sample to 60°F (15.5°C) in a transparent cylinder and measuring specific gravities with a hydrometer.

3.6.8 Density of Solids

ASTM D 1895-89 provides standard test methods for determining the apparent density, bulk factor, and pourability of solid materials which can be used for solid chemical deicers.

3.7 Ecological Effects

Numerous relevant test procedures are available for the ecological risk assessment of chemicals, such as pesticides and industrial effluents, which may be introduced into the environment. These tests are employed in the framework of a generalized protocol, with specific tests being selected on the basis of their suitability and responsiveness to the type or magnitude of the impact posed by a material, including the characteristics of potentially affected ecosystems. Such tests have not previously been applied in a systematic evaluation of the impacts of chemical deicer usage. As a consequence, a set of ecological effects tests specifically designed for use with chemical deicers was developed. Summarized in the following subsections are: (a) a testing protocol for assessment of ecological effects, (b) a general method for estimation of environmental concentrations, and (c) a specific set of tests for evaluation of potential ecological effects.

3.7.1 Ecological Test Protocol

3.7.1.1 *Protocol Description*

An ecological protocol consists of a system for preliminary identification of the characteristics of a deicer in relation to potential ecological effects, determining the mode of dispersal into the environment and quantifying quantities or concentrations in ecological receptor systems, selection of appropriate tests, and designation of test sequences, together with guidelines for determining when ecological issues have been adequately addressed.

The principal elements of the protocol are as follows:

- Chemical analysis and identification of chemical species, both major and minor.

- Preliminary hazard analysis, ecological risk identification, and definition of significant systems at risk, together with probable or potential types of impacts.

- Estimation of probable environmental concentrations in receptor systems and of the magnitude or size of affected systems.

- Identification of species at risk and of appropriate test species.

- Identification of appropriate tests and test sequences with specified checkpoints.

- Guidelines for determining when ecological issues have been satisfactorily resolved.

3.7.1.2 *Protocol Objective*

The objective of the protocol is to provide an operating framework for conducting ecological evaluations and tests in a systematic manner, with test procedures or methods suitable, to the extent possible, for use in agency materials laboratories.

3.7.2 Estimated Environmental Concentration (EEC)

3.7.2.1 *Method Description*

The general method for the derivation of the EEC of deicers will consist of the following basic elements:

- An enumeration of data requirements and data sources, or alternatively of estimating techniques for deriving needed data.
- Submodels or estimating procedures for describing and quantifying the dissemination of deicer chemicals into and through the environment.
- Overall models for predicting the concentration of deicer materials in environmental receptor systems, that is, aquatic and terrestrial systems, and additionally for estimating ultimate fate or persistence.
- Guidelines or procedures for relating estimated environmental concentrations to environmental risk potential, for defining species or systems at risk, and for identifying appropriate test procedures.

3.7.2.2 *Method Objective*

The objective of this method is to provide information and data on the expected fate of deicer materials in potentially affected ecosystems, to ensure the effective utilization of ecological test methods.

3.7.3 Ecological Effects Tests (SHRP H-205.11)

These test methods were developed to conduct initial screening of chemical deicers for their potential ecological effects.

3.7.3.1 *Testing Description*

Ecological effects test methods potentially encompass a range of tests designed to establish acute and chronic toxicities of materials with appropriate single species in aquatic and terrestrial habitats, through the determination of long-term effects on balanced, multispecies systems. Tests suitable for use in an agency materials laboratory are single species tests. Basic procedures for such tests are described in the literature in the following publications:

- Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms, EPA/600/4-85/013
- Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, EPA/600/4-85/014
- Standard Practice for Conducting Acute Toxicity Tests With Fishes, Macroinvertebrates, and Amphibians, ASTM E 729-88a
- Test Methods for Assessing the Effects of Chemicals on Plants, EPA/560/5-75/008

The tests selected for inclusion in SHRP H-205.11 involve the exposure of aquatic or terrestrial species to chemical deicers at specific levels or concentrations under controlled conditions and for specific time periods, and the documentation of mortality rates or quantitative/qualitative evidence of chronic effects. Exposure levels range from those required to cause mortality to levels near estimated environmental concentrations.

3.7.3.2 *Testing Objectives*

The overall objectives of the ecological effects testing is to determine whether the ecological effects of a deicer material pose a significant risk, and to provide a basis for determining whether identified risks are sufficient to either preclude use of the material or to prescribe limitations for its use.

3.8 Health and Safety Aspects

3.8.1 OSHA Protocol

The manufacture, import, and use of chemicals are subject to federal regulation via the Hazard Communication Standard (HCS) of the Occupational Safety and Health Administration (OSHA). The HCS covers all employers with employees exposed to hazardous chemicals in the workplace. The protocols and requirements of the HCS are presented in an abbreviated format in this section.

3.8.1.1 *Elements of the HCS*

Five elements are specified in the HCS. These are:

- Hazard Evaluation
- Labels/Forms of Warning
- Materials Safety Data Sheets
- Employee Training
- Written Hazard Communication Program

Activities in all of the above areas are organized to ensure that all hazards are identified and characterized relative to specific hazards; that procedures are in place to ensure that potentially exposed workers are made aware of hazards; that operational procedures for protecting workplaces and ensuring worker safety are developed and integrated in an operating plan; that workers are informed and trained: and that an ongoing hazard communication system is instituted and maintained.

3.8.1.2 *Hazard Evaluation*

Hazard evaluation begins with an inventory of chemicals, developed in accordance with a specified definition of chemicals. Any substance that presents a physical or health hazard as defined by OSHA is considered regulated under the HCS. Four lists, totaling about 1,200 chemicals, comprise the floor of regulated chemicals.

Hazard evaluation is based on available information on health hazards and on information or data which define the physical hazards of a chemical. Compilations of information and lists of chemicals are available in the following sources:

Hazardous Chemicals

- 29 *CFR*, Part 1910, Subpart Z, Toxic and Hazardous Substances
- Threshold Limit Values for Chemical Substances and Physical Agents in the Work Environment, American Conference of Industrial Hygienists (ACGIH), latest edition

Carcinogenic Chemicals

- 29 *CFR*, Part 1910, Subpart Z, "Toxic and Hazardous Substances"
- National Toxicology Program (NTP) Annual Report on Carcinogens, latest edition
- International Agency for Research on Cancer (IARC) Monographs, latest editions

3.8.1.3 *Labels/Forms of Warning*

Warning labels serve to inform workers about potential danger of significant risk from chemicals that are shipped or used in the workplace. Information on such labels includes the identity of the chemical, the types of risks or hazards, and optional identification of protective measures.

3.8.1.4 *Material Safety Data Sheets (MSDS)*

Material safety data sheets are the basic vehicle for organizing and presenting the identity and concentration of chemicals, together with a variety of data and information which define physical and health hazards and indicate appropriate protective measures. The MSDS is the principal means of transferring information from manufacturers to users.

Information in the MSDS includes:

- Chemical identity, including constituents which pose a health hazard and are present in concentrations of 1 percent or more, and carcinogens present at 0.1 percent or more.
- Physical and chemical characteristics such as vapor pressures, flash points, explosivity, and reactivity.

- Physical hazards and health hazards.
- Permissible exposure levels.
- Safe handling and use precautions, such as eye or skin protection.
- Recommended engineering controls.
- Emergency procedures.

3.8.1.5 *Employee Training Program*

The purpose of an employee training program is to reduce chemical-related illness or injury by informing workers of hazards and providing training on safe practices. A written training program is required and must encompass designated responsibilities and schedules, as well as the specific procedures to be employed in communicating hazards to workers and providing training in safe work practices.

3.8.1.6 *Written Hazard Communication Program*

The written hazard communication program consists of a written record of all the elements which must be complied with in the HCS. The program involves the implementation and updating of a system for recording hazard evaluation/identification and following through with MSDS, labeling/warning, and employee training.

3.8.2 Chemical Deicer Testing and Laboratory Safety

Many test methods for evaluating chemical deicers may involve potentially hazardous chemicals or equipment. Persons conducting these tests are responsible for familiarizing themselves with all pertinent safety data and procedures.

Appendix A

Synopsis of Current Deicer Materials and Test Methods

1

Current Deicer Materials

1.1 Chemical Characteristics

The characteristics of potential chemical deicers—excluding for the moment two critical factors, cost and significant adverse side effects—are that they generally consist of ionic or nonionic chemicals or mixtures which (a) are highly soluble in water at low temperatures, (b) dissolve in water to yield ions or molecules of reasonably low average molecular weight, and (c) exhibit a eutectic temperature preferably several degrees lower than the ambient temperature encountered in the ice or snow event of interest.

Accordingly, the inorganic, ionized deicer is usually composed of alkali or alkaline-earth metal chloride salts. Other anions of potential utility include the acetate, formate, phosphate, and organic acid anions which are derived, at low cost, from organic cellulosic matter by chemical or biochemical processing methods. The nitrate, nitrite, and sulfate anions have been proposed as constituents of chemical deicers; the number of acceptable anions is limited, however, by the fact that many of these anions are incompatible with concrete.

Potential nonionic deicers generally are limited to the lower-molecular-weight simple aliphatic alcohols (i.e., methyl alcohol and ethyl alcohol), lower-molecular-weight amines or amides, and polyhydric alcohols (e.g., ethylene glycol and propylene glycol). Water itself is sometimes a key or essential ingredient of a deicing chemical or a deicing formulation.

1.2 Deicing Applications

Although a large number of chemical substances fall within the general framework described above, the number actually used as chemical deicers is relatively small. Their use is described in the following subsections.

1.2.1 Streets and Highways

Rock salt is the workhorse chemical deicer for highways and streets. Flake, pellet, and "liquid" calcium chloride (usually 30 percent to 32 percent $CaCl_2$ in water) are used, usually in combination with rock salt or abrasives (sand), particularly at lower temperatures, that is, less than 20°F (-7°C). The "liquid" calcium chloride has been found effective for preventive maintenance. It prevents formation of ice films and can be used for treatment or removal of small quantities or depths of snow and ice. A substantive use of liquid calcium chloride consists of treating rock salt in the rock salt stockpile or immediately prior to applying rock salt to highway surfaces. This mode of calcium chloride use is reported to substantially increase the effectiveness of rock salt at low temperatures and to reduce salt application requirements at low temperatures by 25 to 50 percent. Both rock salt and calcium chloride (in solid or liquid form) are employed admixed in relatively small proportions with sand or other abrasives. The added chemical provides some melting action and, in addition, serves to fix the abrasive in the snow or ice cover, thereby fixing it within the surface and preventing discharge of the abrasive to the roadside.

Salt brines (e.g., oil field brines) have been used effectively for highway deicers. Salt brines are apparently used significantly and routinely in Europe but less routinely in the United States. High-speed sodium chloride brine jet streams have been successfully field- tested. Snow and ice control on highways is effected by varying combinations of mechanical measures (plowing, blowing), chemical deicing, and abrasive application. The degree or level of control is usually varied to correspond with highway type and traffic volumes. Effective use of chemical deicers involves the timely use of the most effective combination of control measures, based as much as possible on accurate anticipation of meteorological conditions—temperatures, precipitation type and amount, wind velocities and directions. Equipment quality, including particularly metering capabilities and deicer-spreading parameters, is cited as being of great importance with respect to optimum cost and material-conserving application of deicers. Studies have, in fact, indicated that both the costs of ice and snow control and the quantities of deicer usage can be reduced substantially by improvements in equipment, operator training, better or greater use of mechanical measures in combination with chemical deicing, and accurate forecasting of weather conditions.

Ice and snow control on bridges is a special problem, due to some extent to the basic differences between highways and bridges with regard to ice and snow control, but in large measure due to the corrosion of steel reinforcement in concrete that is attributed to chloride-based deicers. Nonchemical approaches such as electric heating have been tested, and noncorrosive or less corrosive chemical deicers have been tested and are used in some parts of the United States. Options for controlling/minimizing reinforcement corrosion are under study.

The most substantial and potentially productive advanced deicer activity of the past several years involving calcium magnesium acetate (CMA) has had as a major objective the development of a noncorrosive and environmentally innocuous chemical deicer. The CMA activities encompassed essentially the complete scope of product conceptualization, research and development (including theoretical analyses of optional materials and the selection of potential candidates), formulation and laboratory characterization and testing, process development and cost reduction, hazard and environmental testing, studies of corrosivity and impacts on materials, and limited scale production followed by field testing. Finally, commercial producers or suppliers of deicers are active in the development of corrosion inhibitors for inclusion in relatively minor quantities in chloride-based deicers.

1.2.2 Aircraft and Runway Deicing

Compatibility with aircraft materials and runways is at a premium in these uses, and the demands for rapid achievement of safe runways are greater than on highways. Urea, formamide, the glycols, water, and inhibitors are the basic ingredients of deicing formulas employed to remove ice from aircraft and to deice runways. Potassium acetate is also approved for use in these applications. Additional information is available in the following SAE materials specifications: AMS 1425B, 1426B, 1427A, 1431A, and 1730A.

1.2.3 Residential/Commercial Deicing (Sidewalks, Parking Lots)

Rock salt (sodium chloride), potassium chloride, potassium chloride admixed with sodium chloride, calcium chloride, and urea may be variously admixed with small quantities of other materials purported to enhance deicer utility.

1.3 Selection and Use Criteria

Cost, operational reliability, and public safety have been and continue to be the prime considerations in the selection and use of a strategy for snow and ice control. For these reasons rock salt and sand or other available abrasives used in conjunction with the snow plow are generally preferred. A substitute or alternate chemical deicer can easily be 10 times as costly as rock salt, may require higher ratios of application to achieve comparable control of snow and ice, and may in addition be the source of operational difficulties. Nevertheless, in some sections of the United States, the threat to water quality and vegetation has been judged to be severe enough to limit or exclude the use of rock salt.

2

Deicer Test Methods

The deicer test methods are presented in six general categories as follows: physicochemical characterization, deicing performance, materials compatibility, engineering parameters, ecological effects, and health and safety aspects.

2.1 Physicochemical Characterization

2.1.1 Sampling

Representative samples of deicers are a requirements for use in tests such as chemical analysis, fundamental property measurement, materials compatibility tests, and others. The following ASTM procedures address sampling procedures for a variety of sources. The methods are suitable without modifications.

ASTM D 345-90	Drums and bags, hygroscopic solids
ASTM D 1568-87	Drums and bags, nonhygroscopic solids
ASTM D 75-87	Stockpiles, barges, railcars
ASTM D 3665-82	Trucks
ASTM D 345-90	Liquid containers
ASTM C 702-87	Field samples
ASTM D 2013-86	Nonhomogeneous solids: size reduction and mixing

2.1.2 Deicer Analysis

Accurate data on deicer chemical identities and compositions are necessary for characterization of chemical deicers. Minor as well as major constituents are potentially important. Methods applicable to chemical analysis range from colorimetric titrations through instrumental chromatographic and spectroscopic methods.

Selected methods are as follows:

- ASTM E 534-86 Moisture content
- ASTM E 534-86 Water-soluble/water-insoluble
- ASTM E 534-86 Inorganic constituents
- ASTM E 449-90 Inorganic constituents
- ASTM D 4327-88 Anions by ion chromatography
- ASTM D 3178-89 Carbon and hydrogen content
- ASTM D 3177-89 Sulfur content
- ASTM D 3179-89 Nitrogen content

These test methods are suitable for use in laboratories equipped with the necessary equipment and staffed with experienced analytical chemists.

2.1.3 Deicer Property Measurements

Several physical-chemical properties are related to chemical-deicer performance potential. The properties and associated methods are as follows.

2.1.3.1 *Aqueous Solubility*

The solubility of a deicing chemical in water at low temperatures is related to a theoretical ice-melting capacity. Temperatures of interest range from about 32°F (0°C) to the eutectic temperature of a deicer-water system. Solubility measurements basically involve the equilibration of an excess of solute with water at a constant temperature, followed by chemical analyses of supernatant liquids. The measurements must be accompanied by analysis of the deicer for purity, moisture content, and water insolubles.

2.1.3.2 *Freezing-Point*

Freezing-point determination involves the establishment of the concentration of the deicing chemical in water associated with various freezing points, from slightly less than 32°F (0°C) to near the eutectic temperature.

ASTM D 1177-88 is the selected method for determination of freezing points. Aqueous solutions at several selected concentrations are cooled slowly, with stirring, from a temperature above the freezing point to a temperature a few degrees below the onset of freezing. The intersection of cooling curves (time vs. temperature) represents the freezing point of a solution.

2.1.3.3 *Eutectic Temperature*

The equipment and general procedures employed for freezing-point determination in ASTM D 1177-88 are employed for eutectic temperature determination. A solution, initially at a concentration preferred to be either at, slightly lower, or slightly higher than the eutectic composition, is cooled slowly until a constant temperature is reached. The temperature remains constant until no liquid is present.

2.1.3.4 *Eutectic Composition*

The equipment and general procedures of ASTM D 1177-88 are employed for determination of eutectic compositions via one of the following modifications. Aliquots of the solution phase in equilibrium, with ice and solid deicer at the eutectic temperature identified by the method for eutectic temperature determination, are withdrawn and analyzed chemically. Alternatively, deicer concentrations are systematically varied about an approximate eutectic concentration, and the eutectic composition identified by freezing point measurement.

2.1.3.5 *Heat of Solution*

Experimental measurement of heats of solution of deicers in water should seldom be required. Compilation of data on heats of formation of solid or crystalline materials and of aqueous solutions usually will obviate the need for experimental determinations. Literature sources also will suffice for calculation of the heats of solution of mixed solutes. In the event that experimental determinations are judged to be necessary (e.g., for a material for which literature data are not available), solution calorimeters are available. These consist basically of a Dewar-type flask in which water and solute are mixed, and temperature changes measured via set procedures. The calorimetric equipment includes instrumentation needed for calibration so that temperature changes due to solute dissolution can be distinguished from the effects of calorimeters system heat capacities.

2.1.3.6 *Deicer Solution Viscosity*

Deicer solution viscosity, particularly at ice and snow temperatures, is of interest for two reasons—viscous or nonfree-flowing deicer solutions will likely be associated with low ice-deicer interaction rates and can be anticipated to be objectionable from the standpoint of deicer-pavement frictional characteristics. ASTM D 445-88 has been selected as the method for measurement of deicer solution viscosities.

2.1.3.7 *Brine pH*

Deicer brine pH, together with brine capacities to contribute to the alkalinity or acidity of the receiving environment, is potentially important relative to corrosion or materials degradation and relative to impacts on soils or receiving bodies of water. pH measurements are described in ASTM E 70-90. A method for measuring alkalinity is presented in ASTM E 449-90.

2.2 Deicing Performance

2.2.1 Ice Melting

The recommended test methods for evaluating ice-melting rate and capacity of chemical deicers are SHRP H-205.1 (for solid deicers) and SHRP H-205.2 (for liquid deicers). These tests involve the fabrication of ice with a specified thickness (1/8 in.) in a flat Plexiglas dish; equilibration to within 0.5°C (0°C) of a specified temperature; broadcast addition of deicer; and at specified time intervals, collection of brine via decantation, measurement of brine volume, and reintroduction of brine to the system for time periods of 1 hr or more. Both test methods are presented in detail in Appendix B.

2.2.2 Ice Penetration

The recommended test methods for evaluating ice-penetration rate of chemical deicers are SHRP H-205.3 (for solid deicers) and SHRP H-205.4 (for liquid deicers). In these tests, solid deicer particles of uniform weights or drops of liquid deicer are placed on the surface of ice, and depths of penetration measured over a period of 1 hr or more. A small quantity of water-soluble dye is placed on the ice surface to render the brine cavities highly visible. The ice specimen is frozen in small cavities drilled in Plexiglas with a countersink surface. Both test methods are described in detail in Appendix B.

2.2.3 Ice Undercutting

The recommended test methods for evaluating ice-undercutting rate of chemical deicers are SHRP H-205.5 (for solid deicers) and SHRP H-205.6 (for liquid deicers). In the H-205.5 test, a 1/8-in.-thick layer of ice is frozen on a concrete surface, a dye emplaced on isolated spots on the surface, and weighed deicer pellets are placed on the dye spots. Penetration through the ice to the concrete surface is followed by melting at the ice-substrate interface in approximately circular undercutting patterns. In the H-205.6 test for liquid deicers, measured liquid samples are placed in preformed cavities in the ice. Photographs taken at selected time intervals are employed to measure undercut areas, which are converted to undercut area per unit weight of deicer. Both test methods are presented in detail in Appendix B.

2.3 Compatibility With Bare and Coated Metals

2.3.1 Bare Metal Corrosion

The corrosive effects of deicing chemicals on bare metal substrates is an important consideration in the selection of materials for snow and ice control on highways and bridges. Addition of corrosion inhibitors to chloride deicing chemicals also has renewed interest.

SHRP H-205.7 test method is recommended to evaluate the corrosive effects of deicing chemicals and corrosion inhibited deicing chemicals on bare metals. Metal corrosion is measured by weight loss after specified exposure in aerated aqueous solutions of deicing chemicals. The recommended test method is described in detail in Appendix B.

Related procedures are:

- ASTM G 1-90, Cleaning procedure for corroded metal specimens.
- ASTM G 46-76, Pit depth analysis.

2.3.2 Corrosion of Coated Metal

- ASTM B 117-90, A salt spray test, is recommended.

Related or supporting methods are:

- ASTM D 1186-87, Coating thickness.
- ASTM D 1654-79a, Specimen evaluation.
- ASTM F 502-83, Effects on painted aircraft surfaces.

2.4 Compatibility With Metals in Concrete

The corrosive effects of deicing chemicals on reinforcing steel in concrete structures can be particularly destructive and costly to repair.

SHRP H-205.12 test method is an adaptation of ASTM G 109, and is recommended to evaluate the corrosive effects of deicing chemicals on reinforcing steel in a concrete environment. Test specimens are made by embedding short lengths of rebar inside standard concrete block.

Test specimens are ponded with deicer solutions and deionized water as controls. The macrocell current is determined by measuring the IR drop across a resistor connecting the rebar mats. The corrosion potential of the reinforcing steel in the test specimen can be measured using a saturated calomel electrode (SCE) as a reference.

The recommended test method is described in detail in Appendix B.

If the macrocell current is less than 1 μA, the specimens are considered to be noncorroding. If the macrocell current is more than 9 μA, the specimens are considered to be actively corroding. The observed rapid increase in the macrocell current when corrosion occurs can usefully serve to establish a "time-to-failure" criterion.

Related references are:

- ASTM STP 818, Corrosion of Metals in Association With Concrete (1984).
- ASTM STP 906, Techniques for Evaluating Corrosion of Rebars in Concrete (1986).
- ASTM STP 1065, Corrosion Rates of Steel in Concrete (1990).

2.5 Compatibility With Concrete and Nonmetals

2.5.1 Concrete Scaling Effects

Two test methods to evaluate the effects of deicing chemicals on concrete are recommended. SHRP H-205.8 is a rapid method to evaluate scaling/spalling effects of deicing chemicals on small concrete test specimens. SHRP H-205.9 is a method to evaluate scaling effects on larger concrete test specimens. Both test methods involve freeze/thaw cycling of concrete test specimens while in contact with deicing chemical brines. Both methods incorporate weighing the scaled/spalled concrete material to quantify the deicing chemical effects. Detailed descriptions of the two test methods are presented in Appendix B. Related or support methods are:

- ASTM C 672-91, Freeze/thaw cycling of concrete test specimens.
- ASTM C 39-86, Method for evaluating mechanical strength retention.
- ASTM C 192-90a, Procedures for preparing laboratory concrete test specimens.

2.5.2 Compatibility With Nonmetals

Procedures selected for evaluation of compatibility with nonmetals are:

- ASTM B 117-90, Salt spray test.
- ASTM G 31-72, Modified immersion corrosion test.
- ASTM D 638-90, Plastic tensile strength.
- ASTM D 412-87, Rubber tensile strength.

2.6 Engineering Parameters

2.6.1 Frictional Characteristics

The frictional characteristics of pavement surfaces exposed to deicer solutions are of concern from a safety standpoint. The recommended method for evaluating the frictional characteristics of chemical deicers is SHRP H-205.10. The method is based on the British pendulum slip resistance tester, in which a rubber slider is propelled across a sand-blasted glass surface wetted with a solution of deicer material. The length of the slider/glass contact path is accurately controlled. The loss of energy of the pendulum, due to friction between the slider and sand-blasted glass surface, is recorded by dial indicator on the upswing of the pendulum.

The result is a British pendulum number (BPN). Higher BPN values indicate less tendency for cars to skid on a road wetted with deicer melted snow or ice. The recommended test method is presented in detail in Appendix B.

2.6.2 Particle Size Distribution

Selected methods for determining the particle size distribution of solid deicers are:

- ASTM D 632-84, Test procedure for sodium chloride and nonhygroscopic deicers.
- ASTM D 345-90, Test procedure for hygroscopic materials.

2.6.3 Particle Shape

No test is available or recommended.

2.6.4 Flow Properties

The selected method for evaluating the flow properties of solid deicers is:

- ASTM D 1895-89, A pourability test.

2.6.5 Dusting Tendencies

The selected method for evaluating the dusting tendencies of solid deicers is:

- ASTM D 4331-84, Equipment and procedures for collecting and measuring air-entrained particulate matter.

2.6.6 Density

Selected methods for determining the density of solid and liquid deicers are:

- ASTM D 1895-89, Bulk or apparent density measurement of solids.
- ASTM D 1122-90a, Liquid density measurement.

2.6.7 Moisture Sensitivity

Applicable procedures for evaluating the moisture sensitivity of chemical deicers are:

- ASTM E 534-86, Moisture content of nonhygroscopic materials.
- ASTM E 104-85, Methods for achieving constant relative humidities and exposure of materials to constant moisture vapor tensions.

- ASTM D 3201-86, A reference method which details procedures for evaluating moisture uptake by wood products.

2.6.8 Moisture-Related Storage Characteristics

Standard procedures for evaluating the effects of moisture on deicer storage properties are not available. Of particular interest are low-temperature storage properties.

2.7 Ecological Effects

The environmental literature provides an authoritative, generalized protocol for evaluating the ecological effects of chemicals and numerous specific experimental procedures. No literature procedures are specific for determining the ecological effects of deicer chemicals. The recommended testing protocol in SHRP H-205.11 was developed, using EPA and ASTM test methods, to determine the effects of chemical deicers on plant and animal species that may be affected.

The set of ecological effects tests specified in SHRP H-205.11 includes five acute toxicity tests and one chronic toxicity test on specified aquatic animal species in water containing deicer chemicals. Two of the acute toxicity tests are conducted on species collected from the field environment. The ecological tests also includes one test on seed germination and plant growth. The ecological effects testing protocol and the component test procedures are described in detail in Appendix B.

Reference procedures and reference documents are as follows:

- ASTM E 943-88, Terms and Definitions
- ASTM E 729-88a, Acute Toxicity: Fish, Macroinvertebrates, and Amphibians
- ASTM E 1197-87, Soil Core: Terrestrial Environmental Effects
- ASTM STP 920, Community Toxicity Testing
- EPA/600/4-85/013, Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms

- EPA/600/4-85/014, Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms
- EPA/560/5-75/008, Test Methods for Assessing the Effects of Chemicals on Plants
- EPA 223-802352, Standardized Aquatic Microcosm Protocol

2.8 Health and Safety Aspects

The health and safety aspects of deicer manufacture, transport, and use are regulated via OSHA (Occupational Safety and Health Administration) protocols, as outlined in detail in the Hazard Communication Standard.

Appendix B

Primary Test Methods for Evaluating Chemical Deicers

SHRP H-205.1

Test Method for Ice Melting of Solid Deicing Chemicals

1.1 Scope

This test method covers the provision of data on the ice melting capacity of a solid chemical deicer as a function of time and temperature.

1.2 Applicable Documents

1.2.1 ASTM Standards

- C 702-87 Standard Practice for Reducing Field Samples of Aggregate to Testing Size

1.3 Summary of Method

This test utilizes a sheet of ice of uniform thickness (1/8 in., 3.175 mm), frozen in a flat, circular Plexiglas® dish. After equilibration to the desired temperature, deicer particles are broadcast-spread over the ice. At specified time intervals, generated brines are decanted by tilting the specimen to the perimeter of the dish, withdrawn via a syringe, measured for volume, and reintroduced to the test specimen so the melting process can continue.

1.4 Significance and Use

- This test method can be used to compare generic classes of solid deicers with respect to ice melting capacity.
- This test method can be used to evaluate modifications of generic solid deicers with respect to ice melting capacity.
- This test method can be used to assess new solid chemical deicers with respect to ice melting capacity.
- This test method provides a means to describe and compare ice melting capacities of solid deicers over a limited, defined time interval.
- This test method does not quantitatively measure the theoretical or extended time ice melting capacity of solid deicers.

1.5 Equipment

1.5.1 Plexiglas® Test Dish (See Figure B-1, page 99)

A flat, circular dish, 9 in. (22.86 cm) in diameter and 3/4 in. (1.905 cm) deep, is constructed from two pieces of Plexiglas®: one piece 1/4-in. (0.635-cm) x 11-in. (28-cm) square and the other 3/4-in. (1.905-cm) x 11-in.-square (28-cm). A 9-in. (22.86-cm) diameter hole is cut in a larger piece, and the two pieces are joined with cement or solvent designed for use with Plexiglas®. The apparatus may be constructed by shop personnel, but the services of a plastics fabricator are preferred.

1.5.2 Test Dish Accessories

The test dish may be used without attached accessories. However, attachment of a low-temperature thermometer and a digital timer to one edge of the dish is recommended. The thermometer serves as a means to observe temperatures and helps achieve constant test temperatures. The timer is essential for two purposes: monitoring elapsed time and standardizing and monitoring time elapsed during each brine collection operation.

The dimensions of the test dish may be adjusted to accommodate existing space limitations, however, any changes in the ice surface area may necessitate proportional changes in deicer test sample size and/or the volume of water used to grow the ice.

A suitable thermometer consists of a commercially available thermometer containing a red dye fluid and registering temperatures from about -50° to +80°F (-45.6° to 26.7°C). Such a thermometer is attached to the Plexiglas® dish with two screws. (A rubber piece, approximately 1/8 in. thick, should be cemented to the underside of the thermometer case to protect the bulb of the thermometer and hold in place the mounting screws in the thermometer case.)

The digital timer is attached to the thermometer with two small bolts. The calibration of the thermometer should be checked at 0°C by immersion in ice water and by comparison with a reference thermometer at one or two lower temperatures.

1.5.3 Auxiliary Equipment

1.5.3.1 *Syringes*

Plastic syringes with capacities ranging from 5 to 60 mL should be available. A B&D 18-G x 1 1/2-in. needle is suitable.

1.5.3.2 *Sample Bottles*

Twenty-milliliter glass vials equipped with a plastic cap and a plastic seal are preferred. These vials can be weighed on standard analytical balances and can be tightly closed to protect hygroscopic deicers.

1.5.3.3 *Analytical Balance*

A balance capable of weighing up to 20 g to the nearest milligram is preferred. The standard sample weight is 4.17 g; sample weights of 4.170 g ± 0.005 g are preferred.

1.5.3.4 *Dry Glove Box*

The preferred procedure for isolating test samples of hygroscopic deicers involves storage and transfer in a dry box. A sufficiently dry atmosphere may be maintained with a standard solid desiccant, such as anhydrous calcium chloride in pellet or powder form.

1.5.4 Regulated Temperature Enclosures

Ice melting capacities vary by as much as 10 percent per degree Fahrenheit. An enclosure which can be regulated to within ±0.5°F (±0.3°C) of the designated test temperature (5° to 25°F [-4° to −15°C]) is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The enclosure should have a temperature capability ranging from 25°F to at least 5°F (–4°C to at least −15°C).

Options for a controlled temperature system include: (1) a box equipped with hand ports and a window, and placed inside a walk-in cold room, and (2) a similar enclosure placed in an upright freezer, and equipped with hand ports and viewing windows fabricated and installed in the freezer door.

1.5.4.1 *Enclosure for Testing Deicers Inside a Cold Room*

A cold room test enclosure which has been extensively utilized is described in Annex 1. Dimension specifications may be adjusted to meet existing space limitations or to incorporate available equipment already on hand.

1.5.4.2 *Enclosure for Testing Deicers Inside an Upright Freezer*

A similar test enclosure designed for use in an upright freezer is presented in Annex 2. Dimension specifications may be adjusted to meet existing space limitations or to incorporate the use of available equipment already on hand.

1.6 Test Procedures

1.6.1 Syringe Calibration

Syringes are usually designed to measure volumes when the liquid is contained between the barrel or plunger and the tip of the needle. Brine volumes may be measured in this manner. However, it is difficult to avoid some loss of liquid when the liquid is forced into the needle. For this reason the preferred technique consists of drawing all the liquid into the syringe barrel, adjusting the barrel to a mark (e.g., the 10- or 20-mL mark), and recording the difference between the liquid meniscus and the mark. This apparent volume is greater than the actual volume, by an amount equal to the volume of the conically shaped top of the plunger.

If the preferred volume measurement technique is employed, the difference between apparent and actual volume must be determined. The following calibration technique is recommended.

Draw 15 to 20 mL (or volumes appropriate to syringe capacity) into the syringe. Depress the plunger to expel air and air bubbles, and fill the needle with liquid; adjust to a volume mark, and record the volume; retract the plunger to draw the liquid completely into the barrel; record the apparent volume indicated by the position of the liquid meniscus relative to the position of the plunger; repeat as necessary. The difference between apparent volume and actual volume is a volume correction factor to be subtracted from all apparent volumes recorded during the ice melting tests.

1.6.2 Ice Preparation

Test dishes should initially be flushed with an organic solvent, such as ethanol, to remove oils or greases. Thereafter, the dish should be thoroughly flushed between tests with deionized or distilled water.

One-hundred-thirty milliliters of water corresponds to an ice thickness of 1/8 in. (3.175 mm) in the test dish. This quantity of distilled or deionized water is placed in this dish and swirled or stirred to distribute water over the surface. The dish is then placed in a freezer or cold room on a level surface. When the ice is completely frozen, surface ice is melted with a circular (8.5 in. diameter) piece of 1/2 in. thick aluminum. The quantity of melted ice should be sufficient to form a free-flowing film of water over the entire surface of the ice. After swirling/tilting the dish to distribute melted ice uniformly over the surface, the dish is again placed in a cold enclosure and surface water refrozen.

The dish containing the ice is then placed in the constant temperature enclosure and equilibrated to the operating temperature. Overnight temperature equilibration is recommended, with the dish placed on edge to minimize contact with the floor of the enclosure.

1.6.3 Deicer Sample Size and Isolation

1.6.3.1 *Sample Size*

The recommended sample size is 4.170 ± 0.005 g. This sample size results in a larger application rate than that normally used in the field,but is necessary to ensure statistically measurable quantities of brine melt from ice specimens of the specified size.

1.6.3.2 *Sample Isolation*

The deicer samples should be as representative as possible with respect to particle size distribution and the inclusion (or exclusion) of particles that may appear to differ in appearance or purity from the bulk of the material. Rock salt, in this regard, tends to vary from supplier to supplier and may contain both large particles and small particles. Isolation of representative samples may in such cases require sieving and reconstruction of test samples by recombining the various fractions on a weight basis. In any event, procedures employed to isolate test samples should conform to standard procedures for isolating representative samples (ASTM C 702-87).

Special procedures are required for isolation of rock salt samples wetted with "liquid calcium chloride," that is, rock salt wetted with concentrated aqueous solutions of calcium chloride. The recommended procedure consists of sieving bulk rock salt into four or five particle size fractions, recombining the materials in proportion to weights of the fractions, and wetting individual samples with the required weight or volume of calcium chloride solution. Since fine particles wetted with calcium chloride solutions tend to adhere to sample vessel walls, a sample tube consisting of a 20-mL syringe with the needle end sawed off can be employed to discharge the test sample onto the surface of the ice test specimens.

It is recommended that comparative tests involving relatively small differences in ice melting capacity be conducted as a unit, with appropriate control or reference materials, and with particular attention given to representativeness of deicer samples. For example, it is inappropriate to compare results obtained with wetted rock salt to results obtained previously with nonwetted rock salt. Real differences may be obscured by slight temperature differences or by unknown differences in the purity or particle size in the rock salts employed in the tests.

1.6.4 Deicer Addition and Brine Collection (See Figure B-1, page 99)

One or more hours before the test, the chemical deicer test samples are placed in the constant temperature enclosure to equilibrate to test temperature.

Two tests are ordinarily conducted in one test operation. The second test is initiated 2 1/2 min after the first.

The first test is initiated by broadcast-spreading the deicer over the ice surface as uniformly as possible and immediately activating a timer. The second test sample is broadcast on the second ice specimen and a second timer activated at 2 1/2 min into the first test.

At 10 min, the dish for the first test is tilted, allowing brine to flow to the perimeter. Brine is withdrawn into the syringe as it accumulates. The brine decantation process is terminated uniformly at 1 1/2 min. The syringe is then positioned with the needle pointing upward and the plunger is withdrawn slightly to draw brine into the syringe. The plunger is depressed, if necessary, to collect brine in one volume, the barrel adjusted to a mark, and brine volume read and recorded. The brine is then returned in small increments to the ice specimen, with injection as much as possible into cavities in the ice. The test specimen is then shaken to redistribute brine in the ice specimen, set aside, and the second specimen subjected to brine collection. The collection, measurement, and return of the brine should be completed within 2 1/2 min.

Brine can be recovered more rapidly and completely by tilting the dish as much as possible and jarring the bottom edge of the dish on the floor of the enclosure.

With the two-test system, brine can be recovered and measured at 5-min intervals. The recommended time intervals are, however, 10, 20, 30, 45, and 60 min.

1.6.5 Test Replication

Three tests per deicer per temperature are recommended.

1.7 Data Treatment

Data treatment consists of tabulation of individual data points, together with the averages of three replicates and calculated standard errors. Standard errors are calculated by squaring the differences between the average value and observed values, for n replicates; summing the squares; dividing the sum by n(n-1); and taking the square root of the resulting number.

<u>Example standard error calculation</u>

Measured brine volumes in three replicates: 5, 6, and 7
Average brine volume: 6

$$\text{S.E} = \left[\frac{(6-5)^2 + (6-6)^2 + (6-7)^2}{3 \times 2}\right]^{1/2} = 0.577$$

Data treatment can be conveniently conducted by computer, and data points, including averages, entered on computer-generated graphs of brine volume vs. time. Smoothed curve plots, constructed by hand through the average results, may be employed to graphically display the results.

1.8 Data Conversions

1.8.1 Conversion to Brine Volumes Per Unit Weight of Deicer

Since the recommended test uniformly employed a specified deicer weight (4.17 g), comparative ice melting capacities are indicated by total brine volumes. Ice melting capacities will be more clearly indicated if volumes are converted to volumes per unit weight of deicer.

This data conversion, accomplished by dividing brine volumes by deicer weight (4.17 g), is recommended.

1.8.2 Conversion to Quantities (Weights) of Ice Melted Per Unit Weight of Deicer

The weight of ice melted per unit weight of deicer is a preferred data output. It would appear that such data could be generated by weighing brines to obtain values for ice melted. However, solid deicers vary in rate of dissolution. At short time intervals, and at lower temperatures in particular, brine weights will not yield reliable measures of melted ice quantities.

However, a method is available for calculating melted ice quantities with satisfactory accuracy. The method is based on the relationships between densities, concentrations, and temperatures. Thus, the concentration of calcium chloride in brines ranges from about 25 percent by weight to 8 to 10 percent by weight. From density data, the weight of water in 1 mL of brine ranges from 0.956 g/mL for an 18 percent solution at 5°F (−15°C), to 0.9805 g/mL for a 10 percent solution at 23°F (−5°C). The use of 0.97 g H_2O/mL brine accordingly will yield calculated values for melted ice well within the reproducibility of the brine collection and measurement process. The procedure should not be sensitive to insoluble matter or to small percentages of impurities in deicers.

Water in sodium chloride brines, in the temperature range 5°F (−15°C) to 23°F (−5°C), ranges from 0.93 g/mL in 20 percent solutions to 0.98 g/mL in 8 percent solutions. Appropriate values for calculating quantities of melted ice are as follows:

5°F (−15°C):	0.93 g H_2O/mL brine
15°F (−9.44°C):	0.95 g H_2O/mL brine
25°F (−3.888°C):	0.97 g H_2O/mL brine

Density data for aqueous calcium acetate and magnesium acetate at 64°F (+18°C) indicate that concentrated brines (20-22 percent) of mixtures (e.g., CMA brines) will contain 0.88 to 0.89 g H_2O/mL. Dilute brines associated with melting at temperatures of the order of 25°F (−4°C) will contain 0.92 to 0.93 g H_2O/mL.

Data for magnesium chloride are very similar to data for calcium chloride, and the factor (0.97 g H_2O/mL) suggested for calcium chloride is also appropriate for magnesium chloride.

For unknown or uncharacterized deicers, experimental determination of the densities of aqueous solutions of known composition will be required to calculate quantities of melted ice. Density measurements of 10, 15, and 20 percent solutions will suffice. Procedures and equipment for density measurement are specified in ASTM D 3505-85 and D 1122-84. The lowest temperature specified for density determinations is 50°F (+10°C). Measurement at the lowest specified temperature is recommended, plus a somewhat approximate determination at 0°C (ice water) to establish the effect of temperature on density. The weight of water contained in 1 mL of brine is calculated as follows:

$$\frac{\text{Percent } H_2O}{100} \times \text{density (g/mL)} = \text{g } H_2O/\text{mL}$$

It should be pointed out, however, that this approach is not universally suitable. For example, urea/water solutions are very nearly additive with respect to volumes (e.g., 10 cm^3 solid urea + 90 mL H_2O yields very close to 100 mL solution). For such systems the water content of brines varies substantially with solute concentrations (0.757 g H_2O/mL for 30 percent urea; 0.922 g H_2O/mL for 10 percent urea). With volume-additive systems, the water content of brines can be determined by subtracting the specific volume (mL/g) of the deicer from brine volume. However, this method is appropriate only when visual or other evidence indicates that the deicer is completely dissolved in brines.

1.9 Test Reproducibility

Standard errors for tests in triplicate range from about 2 to 10 percent of the average values.

1.10 Example Test Results

Table B-1 presents example ice melting results for pellet calcium chloride and sodium chloride (rock salt or halite) at –3.89°C (25°F), –9.44°C (15°F), and –15°C (5°F). Data in Table B-11 include the average milliliters of brine at 10, 20, 30, 45, and 60 min, and standard errors.

In Table B-2, data of Table B-1 are converted first to milliliters of brine per gram of deicer, and second to grams of ice melted per gram of deicer.

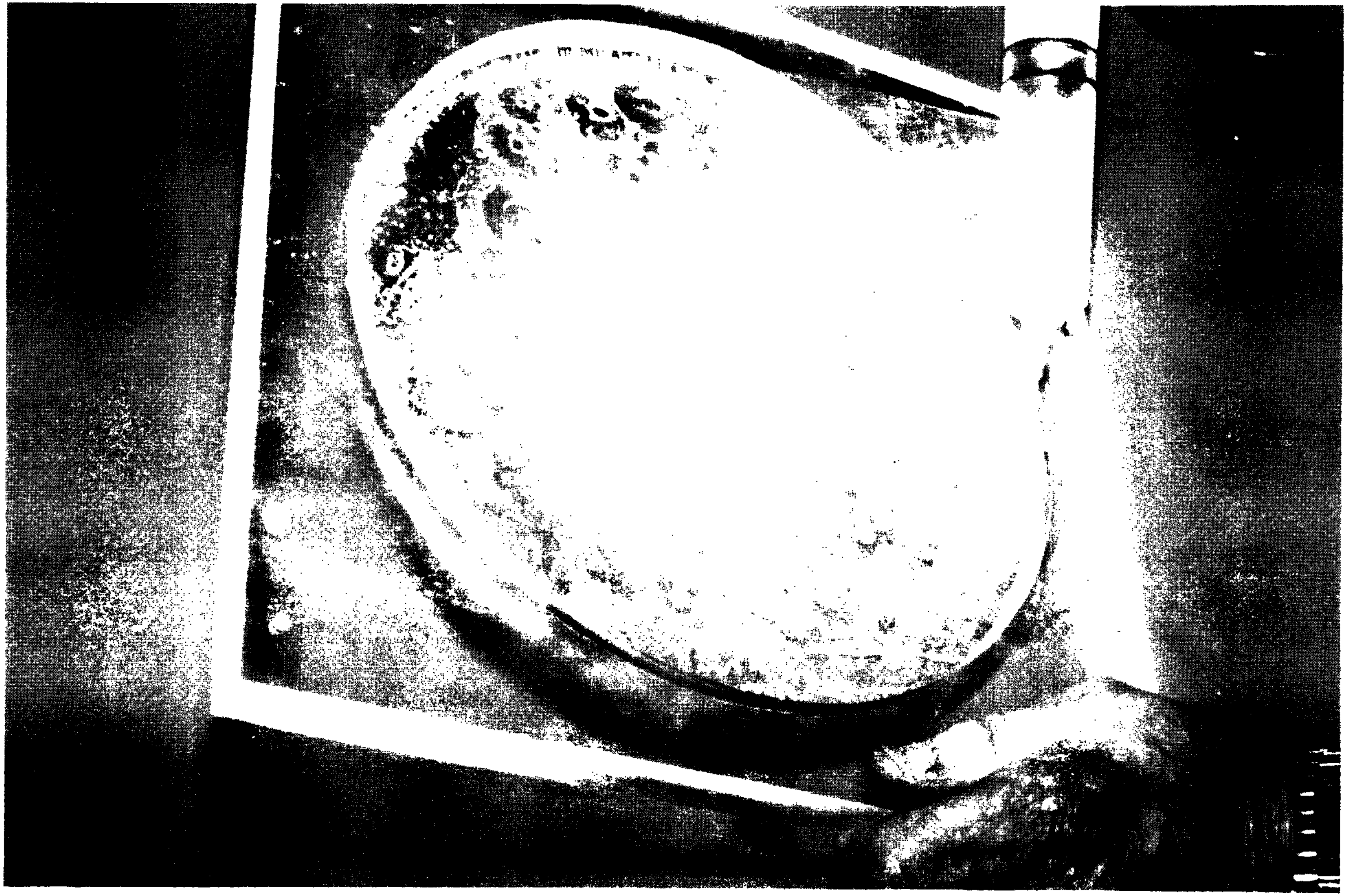

Figure B-1. Ice melting test dish with ice specimen during brine collection procedure.

Table B-1. Example of ice melting test results

Test parameters
Deicers:
$CaCl_2$ Pellets (90.1% $CaCl_2$)
NaCl, Morton-Thiokol Safe-T-Salt™ (97.8% NaCl)
Deicer Weight: 41.7 g
Temperatures: −3.89°C (25°F)
−9.44°C (15°F)
−15°C (5°F)
Replicates: 3

	NaCl		$CaCl_2$	
Time (mn)	Brine[a] volume (mL)	Standard error (%)	Brine[a] volume (mL)	Standard error (%)
	−3.89°C (25°F)			
10	8.7	5.2	11.8	12
20	15.3	5.4	19.7	1.4
30	21.3	4.7	23.5	1.5
45	26.2	3.3	27.0	1.2
60	29.7	3.8	29.2	1.4
	−9.44°C (15°F)			
10	3.1	4.0	7.5	8.3
20	6.0	2.8	12.3	3.9
30	9.3	0.4	14.4	3.3
45	12.7	0.5	15.7	1.9
60	15.7	2.1	16.7	2.0
	−15°C (5°F)			
10	1.0	11.0	4.9	6.0
20	1.5	5.8	10.6	3.0
30	2.6	6.2	12.0	1.2
45	3.8	5.3	12.8	1.0
60	5.2	5.0	13.1	0.3

[a] Average of 3 replicates.

Table B-2. Example of ice melting test results

Milliliters of brine per gram of deicer
Grams of ice melted per gram of deicer
Test parameters: Specified in Table B-1

	−3.89°C (25°F)		−9.44°C (15°F)		−15°C (5°F)	
Time (min)	mL/g	g ice melted/g deicer[a]	mL/g	g ice melted/g deicer[a]	mL/g	g ice melted/g deicer[a]
			$CaCl_2$ Pellets			
10	2.83	2.75	1.80	1.75	1.18	1.14
20	4.72	4.58	2.95	2.86	2.54	2.46
30	5.64	5.47	3.45	3.35	2.88	2.79
45	6.47	6.28	3.76	3.65	3.07	2.98
60	7.00	6.79	4.01	3.89	3.14	3.05
			Sodium Chloride			
10	2.09	2.03[b]	0.74	0.70[c]	0.24	0.22[d]
20	3.67	3.56	1.44	1.37	0.36	0.33
30	5.11	4.96	2.23	2.12	0.62	0.58
45	6.28	6.09	3.04	2.89	0.91	0.85
60	7.12	6.91	3.76	3.57	1.25	1.16

[a] mL/g • 0.97
[b] mL/g • 0.97
[c] mL/g • 0.95
[d] mL/g • 0.93

Annex 1

Enclosure for Testing Deicers Inside A Cold Room

Introduction

Deicer evaluation involves several operations or procedures which require temperatures ranging from -18°C (0°F) to a few degrees below the freezing point of water. Temperature control on the order of ±0.5°F or better is desired for three deicer tests—ice melting rate and capacity, ice penetration, and ice undercutting. This control is recommended because output data from the tests vary 5 to 10 percent per degree Fahrenheit; the reliability of absolute or comparative data is to a considerable extent determined by temperature control.

Several approaches to cold temperature test facilities are possible. The preferred facility consists of a walk-in cold room outfitted with a temperature-regulated box or enclosure. The walk-in cold room is set to operate at a temperature a few degrees lower than the temperature desired in the enclosure and serves three purposes: (1) allows the withdrawal of heat energy from the enclosure by passive (conduction and convection) or active (air circulation) methods; (2) provides a staging area for the final stages of preparing test specimens; and (3) provides space for auxiliary facilities for operations such as the controlled freezing of ice on concrete substrates.

A refrigerator-freezer combination is a necessary and convenient adjunct to the cold room facility; it will be used for purposes such as precooling water and concrete specimens to temperatures a few degrees above the freezing point of water and for initial freezing of ice in certain test specimens.

The minimum recommended size for the cold room is 8 x 10 x 8 to 9 ft tall, with added room for an air lock. The cooling system should be designed for routine achievement of about -5°F (-21°C) to ensure that tests can be conducted at 0°F (-18°C). An automated defrost system is required. The need for a good defrost system is greatest at low temperatures and in periods of high relative humidity. The defrost system should be designed and programmed so that the defrost cycle does not compromise temperatures in the regulated temperature enclosure. The recommended defrost system involves commercially available dual evaporators or chillers. Hot gases from one evaporator operating in a cooling mode are circulated through the coils of the second evaporator, on an alternating basis, to provide rapid defrost without compromising cold room temperatures.

Design of a Test Enclosure

A regulated temperature test enclosure which operates satisfactorily is as follows:

- Test enclosure box, inside dimensions: 30 in. high, 30 in. deep, 40 in. long, constructed from 3/4-in. plywood.

- Insulation: 2-in.-thick Styrofoam on bottom, top, ends, and rear side, placed on the outside of walls, ceiling, and floor.

- Plexiglas® window, hinged: 30 in. long x 18 in. wide, hinged at top approximately 2 in. from ceiling.

- Two hand ports, with 8-in. inside diameter x 4-in. plastic pipe sleeves, and the outer opening loosely closed with canvas flaps to permit hand access and to restrict airflow when not in use.

- One 15-W fluorescent light, ceiling-mounted.

- One fan, 115 V, 0.2 amp, placed in a convenient location with the air output impinging on a baffle to prevent direct flow of warmed air over test specimens.

- A thermistor temperature sensor attached to an adjustable regulator. The power outlet from the regulator is connected to the fan. The sensitivity of the sensor should be ±0.1°F (±0.05°C) or better.

- A calibrated thermometer with sensing element placed in the approximate location of test specimens. This thermometer may be used as the sole temperature indicator or alternatively to calibrate other thermometers or temperature-measuring systems affixed directly to test specimens. (Thermometers equipped with highly visible red fluids and with a temperature range of -50° to +70°F (-45° to +20°C) are suitable. Such thermometers also photograph well, if photographic records of tests are necessary or desired.)

- Auxiliary lighting, as required for photography. Lights for this purpose should be turned on only for a few seconds while pictures are taken. Auxiliary lights may be placed inside or outside the box.

Air temperatures in an enclosure of the type specified above can be changed in about 1 hr from one temperature setting to an adjacent temperature (5° to 10°F change) by lowering or raising cold room temperature, and either opening the window or turning on auxiliary lights. However, equilibration of the box (e.g., walls and floors, and test specimens) to a new temperature is basically an overnight process. The same consideration applies to test specimens with a substantial mass, such as ice frozen on a concrete block. If such a specimen is prepared at a temperature differing from a test temperature, several hours (preferably overnight) should be allowed for the test specimen to equilibrate to the test temperature.

At the discretion of laboratory personnel, the relatively simple temperature control system may be upgraded to provide a more rapid response to temperature changes or to ensure more uniform temperatures in the enclosure. Potential modifications include continuous air circulation, ducted and controlled circulation of air into and out of the enclosure, and staged inputs of thermal energy. In this regard, temperature control is more difficult for operations which require hand manipulation of specimens (e.g., ice melting capacity tests) than for tests such as ice penetration or ice undercutting.

Annex 2

Enclosure for Testing Deicers Inside an Upright Freezer

Introduction

When a walk-in cold room is not available, an upright freezer can be modified to accommodate ice melting, ice penetration, and ice undercutting tests. It is also possible to modify the same freezer to accommodate an ice freezing chamber designed for controlled freezing of ice in the bottom-up mode; however, it may be more convenient to conduct the ice freezing operation in a second freezer, which can be a conventional chest freezer.

Dimension specifications may be adjusted to permit use of equipment already on hand or for purposes of space limitations.

Design of Test Enclosure and Modification of Upright Freezer

Important design features for a regulated temperature test enclosure in an upright freezer are presented in this section (see Figure B-2, page 114).

The elements of the test enclosure and upright freezer are as follows:

- The body of the test enclosure is a plywood box sized to slide into the upper part of the freezer. The box is equipped with a Plexiglas® door hinged on one side of the front of the box.
- Two hand openings or ports in the lower corners of the Plexiglas® door. The openings are covered with slit canvas or cloth. These ports allow hand access into the box and insertion of materials or equipment.
- A double-walled Plexiglas® window, fabricated in the freezer door at a location which coincides with the upper part of the Plexiglas® door in the interior test enclosure box.

- A hinged door, designed for tight sealing when closed, located just below the double-walled Plexiglas® window in the freezer door. The door is positioned so that hands or equipment can be inserted through the outer freezer door and through the openings in the bottom of the Plexiglas® door in the test enclosure box. This door also is equipped with a slit canvas or cloth flap on the inside or freezer interior side.

- An air temperature sensor, such as a thermistor, and an accompanying temperature regulator, for temperature control. The thermistor is placed inside the test enclosure box, the regulator outside, and the power outlet of the regulator to a heat source placed inside the box.

- A small fan, placed in the interior of the test enclosure box, and operating continuously.

- Interior lighting, achieved by a ceiling-mounted, 15-W fluorescent light, with on/off switching.

- A camera port, located in the ceiling of the box.

- A calibrated thermometer or an optional temperature measurement device, placed in the interior of the test enclosure box.

- A plywood shelf approximately 10 in. wide, located in the rear of the test enclosure box.

Temperature Control

Temperature regulation in the interior of the enclosure is accomplished by setting the freezer temperature a few degrees lower than the selected test temperature and warming the interior of the enclosure via the thermostatically activated temperature regulator. The air circulating fan and the fluorescent light (when on) provide a continuous input of heat energy. A light bulb driven by the temperature regulator supplies energy as required.

Dimensions

Dimensions of the test enclosure and of modifications to the freezer door will depend on dimensions of the freezer. The width of the box should be 1 to 1.5 in. less than the distance between shelving brackets, since the box ordinarily will rest on a metal grid shelf supplied with the freezer. Box depth will similarly be a few inches less than the depth of the freezer.

Headspace above the box should be sufficient to accommodate a camera with the lens placed in a camera port. (Camera focusing, shutter speeds, and lens openings will necessarily be accomplished with the box partially withdrawn from the freezer.)

Representative System Specifications

Upright Freezer

An upright freezer (Puffer Hubbard Model IUF-3023) designed for nonresidential use is an acceptable freezer unit. The freezer has the following specifications:

Compressor: 3/4 hp
Forced air cooling
Automatic defrost: 3 programmable cycles per day
Capacity: 22.3 ft^3 (0.63 m^3)
Top-mounted cooling system
Operating temperature range, controlled by a simple dial: -30° to 0°C
Dimensions—exterior, excluding compressor compartment: 28-in. wide
87-in. high
33 1/4-in. deep

Regulated Temperature Enclosure

- Construction materials
  1/2-in. plywood sides, top, bottom, rear wall
  1/2-in. plywood shelf
  1/4-in. Plexiglas® door/window front

- Enclosure dimensions, interior
  23 1/2-in. height
  19 3/8-in. width
  24-in. depth

- Window/door, front of box
  19 1/2-in. width
  15 7/8-in. depth
  Cutouts
    Bottom/left: 7 1/2-in. width, 6 1/4-in. height
    Bottom/right: 7 1/2-in. width, 6 1/4-in. height
    Hinged to open from left

Freezer Door Modifications

- Door cutout
  21 1/2-in. height
  18 1/2-in. width

- Double-walled window
  Interior: 18 1/2-in. width, 14 7/8-in. height
  Exterior: 19 1/2-in. width, 16-in. height; two 1/4-in. Plexiglas® sheets
  Attached via screws to interior and exterior of upper part of freezer door frame, after application of a bead of caulking
  Bag of desiccant placed in airspace between windows

- Window/door support
  1 1/4-in.-thick wood, 18 3/4-in. long by width of door frame
  (~ 1 3/4 in.)
  Installed as crosspiece via screws between the bottom edges of the double window, with 1/2 in. of the 1 1/4-in. thickness protruding below the window. (This piece supports and separates the bottoms of the windows and serves as a frame for the hinged door.)

- Hinged door
  Installed beneath double-walled window
  19 1/2-in.-wide, 6 1/2-in.-high 1/4-in. Plexiglas® plate
  Hinged to open from top
  Weather-stripped with 1/4-in.-thick by 1/2-in.-wide black sponge weather stripping
  Three buttons to close door

- Cloth or canvas flaps (three)
  One sized to fit on the inside/bottom of the Plexiglas® door of the box
  One sized to fit on the outside/bottom of the Plexiglas® door of the box
  One sized to fit over the interior of the opening for the hinged door on the freezer door

These flaps are attached via screws. Each is slit vertically, with slits staggered from exterior to interior to provide for hand access with minimal transfer of heat.

- Hinged door insulation

To minimize transfer of heat through the hinged door, a 2-in.-thick piece of Styrofoam is cut to fit in the opening and removed only when the door is opened for hand access or transfer of materials.

Test Enclosure Accessories

- One small cage-mounted fan attached to the edge of the shelf in the box. A 6- to 7-W fan, rated at 30 scfm, is suitable.

- One ceiling-mounted, 15-W fluorescent fixture, with on/off switch.

- One light bulb (100 W) mounted in the right side/rear under the shelf. The power cord is connected to the temperature regulator.

- A radiant energy shield to prevent direct exposure of test specimens to radiation from the light bulb heater, and additionally to prevent radiant warming of walls and floors. The shield is a cardboard box, 8 in. square and 6 in. tall, open at the top, and with a 4- by 1/2-in. opening in the bottom, mounted in the corner so that the bulb is inside but with about a 1-in. space between the top of the cardboard box and the bottom of the shelf. Other shield materials are acceptable if light energy is largely contained and heat energy transmission to the enclosure occurs primarily via convective air.

- Temperature regulator mounted outside the freezer; probe inserted through a small opening in upper right rear of the box. Numerous regulators are commercially available. A Dyna-Sense electronic temperature controller, Cole-Parmer Model 2158, and a YSI Series 400 thermistor bead air temperature probe are suitable.

- An auxiliary incandescent light (60 W), with on/off switch, placed near the ceiling on the right wall of the box. This light provides lighting for photographs taken of undercutting tests. It is switched on momentarily as each picture is taken.

Procedures for Conducting Deicer Tests in Regulated Temperature Enclosure

Ice Melting Test

Procedures for conducting ice melting tests are essentially identical with procedures utilized with a regulated temperature enclosure located in a cold room. The process of collecting and measuring brines is accomplished by insertion of gloved operator hands through the opened hinged door on the freezer door and through the cloth flaps into the box. The hinged door should be closed between brine measurement periods.

Ice Penetration Test

General procedures for this test are the same as with an enclosure located in a walk-in cold room. Operations such as conditioning the surface of the ice, dye placement, and particle addition are carried out by inserting gloved hands into the box. After the deicers are in place, access to the box is not necessary for the remainder of the test.

Ice Undercutting Test

Placement of dye materials and of deicer liquids and solids is accomplished by inserting gloved hands into the box. If a camera equipped with remote control for the shutter and automatic film advance is available, photos can be taken remotely. With a manually operated camera, it will be necessary to momentarily open the freezer door when a picture is taken. The windows fog quickly when the door is opened, so an auxiliary timer located outside the freezer will be required.

Ice Freezing Accessory

Ice on undercutting specimens must be frozen carefully in the bottom-up mode. The necessary equipment may be fabricated and placed in the lower section of the upright freezer adapted for deicer performance tests. Alternatively, the equipment may be placed in the freezer compartment of a refrigerator or in a chest freezer.

The bottom-up ice freezer consists essentially of a box mounted on a steel or aluminum plate, 1/2 in. or more thick. The box is fitted with a removable lid and with shelf or plank supports located approximately midway between top and bottom. Two to three planks are placed loosely on the supports, with approximately 1 in. between the planks to permit air circulation. One to two (depending on box size) incandescent lamps (100 W each) are placed above the planks, located so that light energy does not directly enter the lower compartment. A thermocouple is inserted through one of the planks into the lower compartment airspace. A small fan is placed in the upper compartment to circulate air.

Ice is frozen by placing the substrate specimen, precooled to the specified temperature (+1 to +2°C) on the metal plate which has been cooled to -10°C (14°F) or lower; adding water precooled to the specified temperature (+1° to 2°C); placing the planks in position; and activating the fan and light bulbs. The bulbs are turned on full power initially and reduced in power via a variable power supply as required to achieve and maintain an air temperature slightly above 0°C (32°F).

Dimensions of the box are not critical and can be adjusted in accordance with space available in a freezer and in accordance with the number and size of specimens to be frozen in a single operation.

Freezing effectiveness will be increased if areas of the metal plate not in direct contact with test specimens are covered with insulation to prevent exposure to warm air.

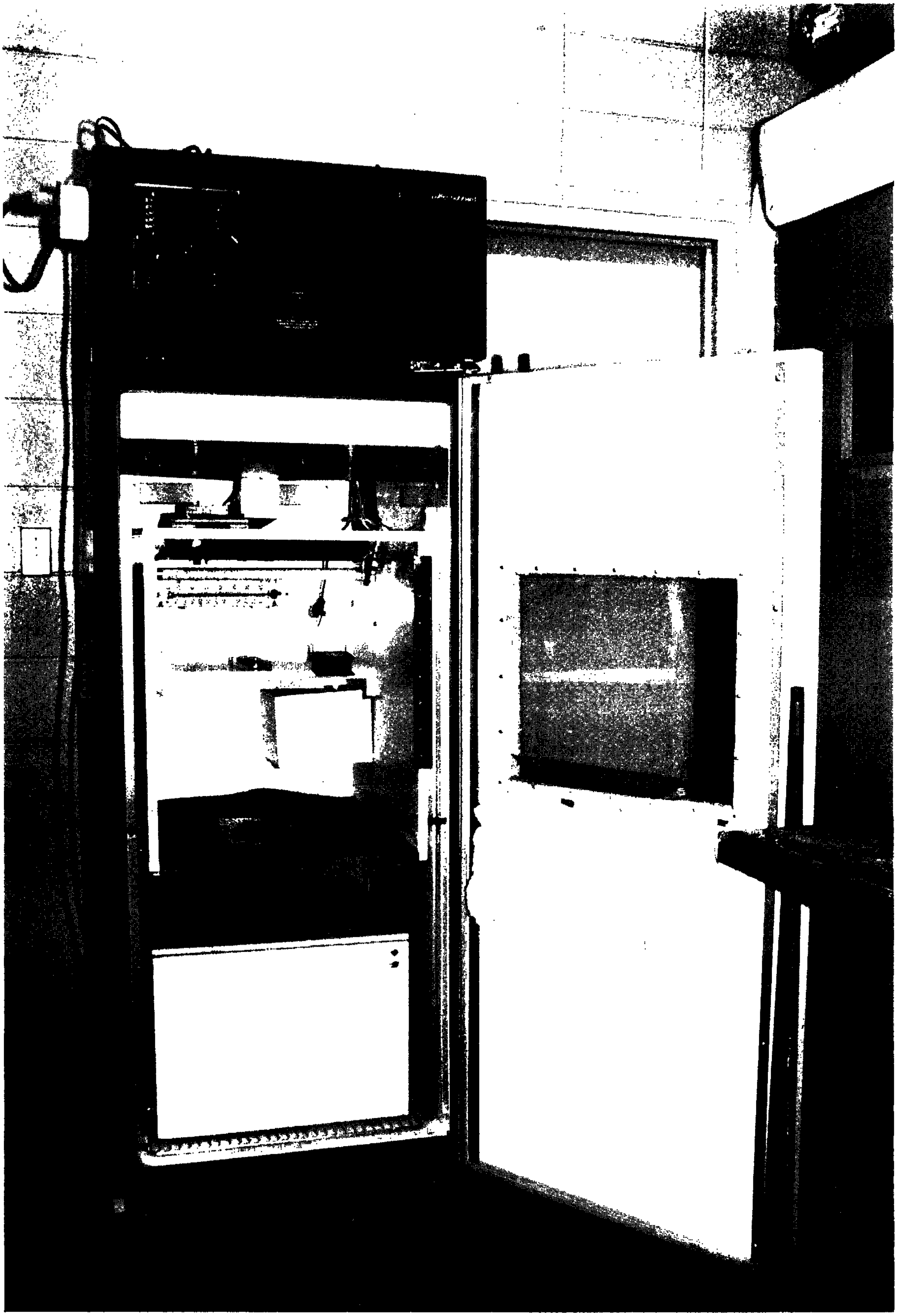

Figure B-2. Enclosure for testing deicers inside an upright freezer.

SHRP H-205.2

Test Method for Ice Melting of Liquid Deicing Chemicals

2.1 Scope

This test method covers the provision of data on ice melting capacities of liquid deicing materials as a function of time and temperature.

2.2 Applicable Documents

2.2.1 ASTM Standards

- E 440-90 Method for Analysis of Calcium Chloride

2.2.2 Other Documents

- SHRP H-205.1 Ice Melting of Solid Deicing Chemicals

2.3 Summary of Method

The test utilizes a sheet of ice of uniform thickness (1/8 in., 3.175 mm) frozen in a flat circular Plexiglas® dish. After equilibration to the desired temperature, a weighed quantity of liquid deicer is distributed over the surface of the ice via a syringe. At specified time intervals, generated brines are decanted to the perimeter of the dish by tilting the dish, withdrawn via a syringe, measured for volume, and reintroduced to the specimen so that the melting process can continue.

2.4 Significance and Use

- This test method is similar to the test for solid deicers (SHRP H-205.1).
- This test method can be used to compare generic classes of liquid deicers, to evaluate modifications to generic liquid deicers, or to assess new liquid deicers for ice melting capacity.
- This test method provides a means to describe and compare the ice melting capacities of liquid deicers over a limited, defined time interval.
- This test method does not quantitatively measure the theoretical or extended time ice melting capacity of liquid deicers.

2.5 Equipment

2.5.1 Plexiglas® Test Dish

A flat, circular dish, 9 in. (22.86 cm) in diameter and 3/4 in. (1.905 cm) deep, is constructed from two pieces of Plexiglas®: one piece 1/4-in. (0.635-cm) x 11-in. (28-cm) square and the other 3/4-in. (1.905-cm) x 11-in.-square (28-cm). A 9-in. (22.86-cm) diameter hole is cut in the center of the 3/4-in.-thick piece of Plexiglas®, and the two pieces are joined with cement or solvent designed for use with Plexiglas®. The apparatus may be constructed by shop personnel, but the services of a plastics fabricator are preferred. The dimensions of the test dish may be adjusted to accommodate space limitations, however, any changes in the ice surface area may necessitate proportional changes in deicer test sample size and/or volume of water used to grow the ice.

2.5.2 Test Dish Accessories

The test dish may be used without attached accessories. However, attachment of a low-temperature thermometer and a digital timer to one edge of the dish is recommended. The thermometer serves as a mean to observe temperatures and helps achieve constant test temperatures. The timer is essential for two purposes: monitoring elapsed time and standardizing and monitoring time elapsed during each brine collection operation.

A suitable thermometer consists of a commercially available thermometer containing a red dye fluid and registering temperatures from about -50° to +80°F (-45.6° to +26.7°C). Such a thermometer is attached to the Plexiglas® dish with two screws. (A rubber piece, approximately 1/8 in. thick, should be cemented to the underside of the thermometer case to protect the bulb of the thermometer and hold the mounting screws in place in the thermometer case.)

The digital timer is attached to the thermometer with two small bolts. The calibration of the thermometer should be checked at 0°C by immersion in ice water and by comparison with a reference thermometer at one or two lower temperatures.

2.5.3 Auxiliary Equipment

2.5.3.1 *Brine Collection Syringes*

Plastic syringes with capacities ranging from 5 to 60 mL should be available. A B&D 18 G x 1 1/2-in needle is suitable.

2.5.3.2 *Syringes for Handling Liquid Deicer Samples*

Plastic syringes with a capacity of 5 mL are employed for isolation of test samples of liquid deicers, and for addition of deicers to ice test specimens.

The plastic tube supplied with syringe needles is employed as a cap placed over the needle to limit exposure of deicer in the syringe to atmospheric moisture and to prevent loss of liquid by evaporation during the handling process, which includes weighing the filled syringe and then the empty syringe after the liquid has been discharged to the ice specimen.

2.5.3.3 *Analytical Balance*

A balance capable of weighing 10 to 20 g to the nearest milligram (preferably to 0.1 mg) is required.

2.5.4 Regulated Temperature Test Enclosures

Collected quantities of brine vary by as much as 10 percent per degree Fahrenheit. An enclosure which can be regulated to within ±0.5°F of the designated test temperature (5° to 25°F [–4° to –15°C]) is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The selected test temperature should not be lower than the crystallization/precipitation temperature of the liquid deicer.

The enclosure should have a temperature capability ranging from 25°F (–4°C) to at least 5°F (–15°C).

Options for a controlled temperature include: (1) a box equipped with hand ports and a window, and placed inside a walk-in cold room, and (2) a similar enclosure placed in an upright freezer, and equipped with hand ports and viewing windows fabricated and installed in the freezer door.

2.5.4.1 *Enclosure for Testing Deicers Inside a Cold Room*

A cold-room testing enclosure, which has been extensively utilized, is described in Annex 1 of Test Method SHRP H-205.1. Dimension specifications may be adjusted to existing space limitations or to incorporate available equipment.

2.5.4.2 *Enclosure for Testing Deicers Inside an Upright Freezer*

A similar testing enclosure designed for use in an upright freezer is presented in Annex 2 of Test Method SHRP H-205.1. Dimension specifications may be adjusted to existing space limitations or to incorporate available equipment already on hand.

2.6 Test Procedure

2.6.1 Ice Preparation

Test dishes should initially be flushed with an organic solvent, such as ethanol, to remove oils or greases. Thereafter, the dish should be thoroughly flushed between tests with deionized or distilled water.

One-hundred-thirty milliliters of water corresponds to an ice thickness of 1/8 in. (3.175 mm) in the test dish. This quantity of distilled or deionized water is placed in the dish and swirled or stirred to distribute water over the surface. The dish is then placed on a level surface in a freezer or cold room. When the ice is completely frozen, the surface ice is melted with a circular (8.5-in. diameter) piece of 1/2-in.-thick aluminum. The quantity of melted ice should be sufficient to form a free-flowing film of water over the entire surface of the ice. After swirling/tilting the dish to distribute melted ice uniformly over the surface, the dish is again placed in a cold enclosure and surface water refrozen.

The dish is then placed in the constant temperature enclosure and equilibrated to the operating temperature. Overnight temperature equilibration is recommended, with the dish placed on edge (vertical) to minimize contact with the floor of the enclosure.

2.6.2 Liquid Deicer Test Sample: Isolation, Measurement, and Addition

Liquid deicers may vary from pure liquids to mixtures or solutions. Procedures for isolating and handling test samples will vary in accordance with properties of the liquids. Example procedures are presented below.

2.6.2.1 *Pure or Neat Liquids*

Liquids which do not solidify or precipitate dissolved constituents are isolated in sample syringes and syringe-plus-sample weight is determined at ambient laboratory conditions. The deicers in the syringes are then placed in the test enclosure and equilibrated to test temperatures, discharged to the ice specimen, and then the empty syringes reweighed after equilibration to the ambient laboratory environment. An example of this procedure using ethylene glycol is presented in the next section.

2.6.2.2 *Anhydrous Ethylene Glycol*

This chemical deicer is commercially available in 800 mL glass bottles equipped with a "Sure-Seal" cap (Supplier: Aldrich). The needle of a plastic syringe (option: B&D 5 mL, equipped with a B&D 18 G x 1 1/2-in. needle, plus the plastic needle cap provided with the needle) is inserted through the Sure-Seal cap, with the bottle inverted, 4.5 to 5 mL liquid drawn into the syringe, and the needle removed from the bottle. With the needle pointing upward, excess liquid and air bubbles are discharged into a tissue, to the 3.8 mL mark. The plunger is then drawn slightly into the barrel, any liquid on exterior surfaces removed by a tissue, and the needle cap placed over the syringe needle. (The cap fits snugly on the end of the syringe.)

The filled syringe is weighed, and placed in the test enclosure to equilibrate to the test temperature (conveniently overnight).

The ice melting test is initiated by removing the needle cap, and discharging the liquid onto the ice surface in circular bands.

The needle cap is placed over the needle, the syringe returned to the ambient laboratory environment, and empty weight determined after allowing about 1 hr for temperature and outer surface moisture equilibration.

2.6.2.3 *Calcium Chloride Solutions*

A wide range of options exist for testing of calcium chloride solutions. These tests fall into two basic classes: (a) tests involving reagent grade calcium chloride, that is, aqueous solutions essentially free of impurities such as alkali halide, and (b) tests involving commercially available calcium chloride which may contain alkali chlorides at levels of 2 to 5 percent and magnesium chloride at levels from 0.2 to 0.5 percent. The commercially available forms usually are slightly alkaline in aqueous solutions.

The highest concentration, in water, of pure or reagent grade calcium chloride which can be precooled to a test temperature of the order 0° to 5°F (-18° to -15°C) without precipitating calcium chloride is about 35 weight percent $CaCl_2$. The crystallization points of calcium chloride solutions derived from the less pure sources of calcium chloride tend to be a few degrees higher than crystallization points of solutions containing the same percentages of reagent-grade calcium chloride. A reasonable guideline for testing commercial source solutions, without danger of precipitating dissolved constituents is as follows: 32 percent solutions may be precooled to 0°F to +5°F (-18° to -15°C); and 35 percent solutions to 32°F (0°C). These guidelines apply to Dowflake™ or Peladow™ (Dow Chemical Company products) derived calcium chloride solutions.* Calcium chloride solutions from other suppliers may differ, and appropriate product bulletins should be consulted.

Test procedures will vary according to the purity and concentration of calcium chloride in test liquids. The most important constraint is the temperature to which a solution may be cooled before addition to ice specimens without precipitating dissolved solids. To eliminate the possible effects of sensible heat on ice melting, deicer concentrations should be adjusted to allow for precooling to test temperatures.

Background ice melting data on calcium chloride solutions has been obtained with reagent grade calcium chloride solution saturated at 32°F (0°C). This solution is prepared by mixing a calculated excess of the dihydrate of calcium chloride ($CaCl_2 \cdot 2H_2O$) with water, placing it in a stoppered flask in an ice bath in a refrigerator, and withdrawing saturated supernatant liquid after equilibration for a minimum of 12 hr. Early in the equilibration process, a few crystals of calcium chloride dihydrate must be added to initiate crystallization/precipitation of excess calcium chloride.

Details of the procedure are as follows:

- Weigh 75 g of calcium chloride dihydrate in a small beaker.
- Transfer to a 250-mL Erlenmeyer flask.
- Measure 70 mL deionized or distilled water.
- Add water to the Erlenmeyer flask using the water to complete transfer of calcium chloride dihydrate.
- Swirl/mix the solution to dissolve calcium chloride completely, while cooling the flask in tap water.
- Stopper the flask with a rubber stopper, and place in an ice/water bath in a refrigerator.

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* *Calcium Chloride Properties and Form Handbook*, Dow Chemical USA, Form No. 173-1188-88 B&L; and Supplements "Properties of Dowflake™ Calcium Chloride," and "Properties of Peladow™ Calcium Chloride."

- After about 1 hr, add a few crystals of calcium chloride dihydrate to initiate crystallization.

- After 12 or more hours in the ice bath, remove a majority of the clear supernatant liquid with a pipette or syringe. Transfer to a tightly stoppered flask.

- The solution saturated at 32°F (0°C) contains 37.3 percent $CaCl_2$, and is set aside for use in ice melting tests.

- The day before ice melting tests, 3.8 mL test samples are drawn into a 5-mL syringe, the needle capped, and weight of filled syringe measured. The filled syringe is placed in a refrigerator overnight at 35°F (+2° to +3°C).

- Immediately before the test, the syringe is removed from the refrigerator, and the contents discharged to the ice specimen.

- After equilibration to ambient laboratory conditions, the empty syringe is weighed.

If tests are to be made with calcium chloride derived from various sources of flake, pellet, or solutions of calcium chloride, test solutions saturated at 32°F (0°C) may be prepared with appropriately adjusted quantities of water and the source of calcium chloride. In such cases, however, the concentration of calcium chloride should be determined by analysis using the methods specified in ASTM E449-90. Alternatively, solutions with other concentrations of calcium chloride may be prepared from either reagent grade calcium chloride or commercial grade calcium chloride. A calcium chloride solution containing 32 percent $CaCl_2$ is commercially available. Verification of concentration (by ASTM E449-90) is advised, however.

2.6.2.4 *Sodium Chloride Solutions*

As with calcium chloride solutions, sodium chloride solutions may be prepared from reagent grade sodium chloride or from one or more of the numerous sources of commercial grade sodium chloride; the latter are generally referred to as halite or rock salt. Additionally, salt brines may be available for use or for testing relative to highway ice and snow control.

The solubility of reagent grade sodium chloride in water at 32°F (0°C) is 26.3 percent by weight; at 68°F (20°C) the solubility is slightly higher (26.41 percent by weight). Commercial sodium chloride supplied for uses such as highway ice and snow control has a specified minimum sodium chloride content of 95 percent; impurities include other chlorides, calcium sulfate and insoluble matter. Salt brines from other sources can be expected to vary substantially in concentration and in composition.

Ice melting data obtained with concentrated solutions of reagent grade sodium chloride constitute a point of reference for salt solutions consisting primarily of sodium chloride. Further, the ice melting capacity of a 26.3 percent solution of reagent grade sodium chloride should differ little from the ice melting capacity of a 26.3 percent solution of the water soluble material derived from a product containing 95 to 97 percent NaCl.

The following general guidelines are applicable to ice melting tests conducted with aqueous solutions of sodium chloride. They are applicable as well to any liquid deicer containing two or more materials.

- Precautionary measures must be taken to ensure that test conditions do not cause changes in composition. For example, a solution at or near a saturated condition should not be cooled, or be allowed to cool, below a saturation temperature prior to addition to ice.

- While ice melting data may be obtained for a liquid with an essentially unknown composition, more meaningful data can be obtained if composition is known. Thus compositions should be established either via chemical analysis or by preparing liquids from measured quantities of materials of known compositions and purities.

The procedure for preparing a test solution of sodium chloride from reagent grade material is as follows:

- Place 45 g reagent grade sodium chloride in a 250-mL Erlenmeyer flask, and add 110 mL deionized or distilled water.

- With occasional swirling/mixing, let stand at ambient laboratory temperature for 3 to 4 hr.

- Place the stoppered flask in a water/ice bath in a refrigerator for 12 hr or more.

- Remove clear supernatant liquid with a pipette or syringe. Place in a suitable stoppered flask for storage. The concentration of NaCl is 26.3 percent by weight.

- Draw 3.8 mL of liquid into a 5-mL syringe; weigh; cool overnight in a refrigerator; discharge contents on the ice specimen; weigh empty syringe to obtain weight of liquid discharged to the ice specimen.

2.6.3 Brine Collection and Measurement

A digital timer is activated when liquid samples are discharged to ice specimens. At intervals of 10, 20, 30, 45, and 60 min, test dishes are tilted; liquid flowing to the perimeter of the dish is drawn into a syringe over a 1 1/2 min period; the collected volume measured and recorded; and the liquid returned to the ice specimen as uniformly as possible over the ice surface. The collection, measurement, and return of the brine should be completed within 2 1/2 min.

Measurement of liquid volumes is accomplished as follows: with the syringe in a vertical position (needle up) the barrel is withdrawn slightly to draw liquid into the syringe; if air bubbles are present, tap the syringe to remove; depress the barrel until liquid level nears the needle, and note and record volume. Alternatively, volumes between marks on the syringe barrel may be predetermined, and brine volumes measured between marks with the fluid entirely in the barrel of the syringe. The apparent volumes between mark volumes are about 0.3 to 0.8 mL (depending on syringe capacity) greater than true volumes. This alternate procedure is less susceptible to accidental loss of liquid from the syringe needle.

2.6.4 Test Replication

Three tests per deicer per temperature are recommended.

2.7 Data Treatment

2.7.1 Raw Data

Raw data accumulated in the ice melting test for liquids consists of the following:

a. Brine volumes collected at specified time intervals. The volumes are measured at the operating test temperature, and thus reflect densities at these temperatures.

b. The mass of the liquid in the form that it is charged to the ice. With liquid deicers consisting of chemicals dissolved in water, the mass includes the weight of water.

c. Deicer compositions, which may range from pure liquid deicing chemicals to liquids containing deicing chemicals in specified concentrations.

In addition, certain supporting information may be available for use in analyzing and interpreting results. These include densities of deicer-water systems, and particularly should include the compositions of deicer-water systems at equilibrium with ice at test temperatures. With liquid deicers, these types of data are essential for determining how much of the collected liquid is melted ice (i.e., for distinguishing between melted ice and the deicing liquid contained in collected brines).

2.7.2 Two-Step Treatment of Raw Data

A two-step treatment of raw data obtained in the ice melting tests is recommended.

The first step consists of conversion of brine volumes to volumes per unit weight of the deicer as charged (milliliters brine/gram deicer).

The second step consists of calculation of the quantity of ice melted, expressed as grams of ice melted per gram of deicer liquid. This result can be converted, if desired, to the quantity of ice melted per gram of deicing chemical (i.e., per gram of dry weight calcium chloride or sodium chloride in aqueous solution deicers).

2.7.2.1 *Background and Basis of Two-Step Data Treatment*

2.7.2.1.1 Step 1: Brine Volume Per Unit Weight of Deicer. These values are calculated by dividing collected volumes by weights of deicers as initially charged, as indicated in Table B-3. Data for all replicates (normally three) are averaged and subjected to analyses for reproducibility.

2.7.2.1.2 Step 2: Calculation of the Quantities of Melted Ice Contained in Collected Brines. In principle, these calculations require accurate data on brine densities together with accurate information on the concentrations of deicing chemical in the brines. With aqueous deicers, such as calcium chloride and sodium chloride solutions in water, the water charged together with the active deicing ingredient must be distinguished from melted ice.

Table B-3. Ice melting results
25°F; 37.3% $CaCl_2$

	No. 1 5.2534 g		No. 2 5.1833 g		No. 3 5.2212 g			
Time (min)	Total mL	mL/g	Total mL	mL/g	Total mL	mL/g	Average (mL/g)	Standard error[a](%)
10	12.2	2.32	11.7	2.26	13.1	2.51	2.37	3.2
20	14.5	2.76	13.9	2.69	14.6	2.80	2.75	1.2
30	16.2	3.09	15.2	2.94	16.6	3.18	3.07	2.2
45	17.2	3.28	16.5	3.19	17.6	3.37	3.28	1.6
60	18.2	3.47	17.2	3.32	18.5	3.55	3.45	2.0

[a]Standard error = $\left[\dfrac{(\bar{n}-n_1)^2 + (\bar{n}-n_2)^2 + (\bar{n}-n_3)^2}{n\ (n-1)}\right]^{1/2}$

Example

$\bar{n} = 3.45,\ n_1 = 3.47,\ n_2 = 3.32,\ n_3 = 3.55$

Standard error = $\left[\dfrac{(3.45-3.47)^2 + (3.45-3.32)^2 + (3.45-3.55)^2}{3 \times 2}\right]^{1/2} = \mathbf{0.06745}$

Standard error, percent = $\dfrac{\mathbf{0.06745}}{\mathbf{3.45}} = \mathbf{2.0}$ **percent**

Analyses of density changes accompanying the dilution, with water, of ethylene glycol, concentrated calcium chloride solutions, and sodium chloride solutions indicate that volume changes accompanying dilution are very close to being additive, as shown by the following examples.

Example 1: Dilution of 20 to 10 percent $CaCl_2$ at 23°F (–5°C)

Densities: 20 percent $CaCl_2$, 1.1874
10 percent $CaCl_2$, 1.0894

100 g (20 percent) + 100 g H_2O → 200 g (10 percent)

$$\frac{100}{1.1874}\text{ mL} + 100\text{ g } H_2O \rightarrow \frac{200}{1.0894}\text{ mL}$$

84.2176 mL + 100 g H_2O → 183.5873 mL
$\Delta V = 99.37$ mL

1 mL ΔV equivalent to 1.006 g H_2O

Example 2: Dilution of 20 to 10 percent NaCl at 32°F (0°C)

Densities: 10 percent NaCl, 1.07677
20 percent NaCl, 1.15663

100 g (20 percent) + 100 g H_2O → 200 g (10 percent)

86.458 mL → 185.7407 mL

$\Delta V = 99.283$ mL

1 mL ΔV equivalent to 1.007 g H_2O

Example 3: Dilution of 35 to 10 percent $CaCl_2$ at 68°F (20°C)

Densities: 35 percent, 1.3373
10 percent, 1.0863

100 g (35 percent) + 250 g H_2O → 350 g (10 percent)

74.7775 mL → 322.1946 mL

ΔV = 247.417 mL

1 mL ΔV equivalent to 1.01 g H_2O

Example 4: Dilution of 100 to 10 percent ethylene glycol (EG) at 68°F (20°C)

Densities: 100 percent EG, 1.1155
10 percent EG, 1.0108

100 g EG + 900 g H_2O → 1,000 g (10 percent EG)

89.6459 mL → 989.3154 mL

ΔV = 899.67 mL

1 mL ΔV equivalent to 1.00037 g H_2O

Example 5: Dilution of 100 to 30 percent EG at 68°F (20°C)

Densities: 100 percent EG, 1.1155
30 percent EG, 1.03775

100 g EG + 233.33 g H_2O → 333.33 g (30 percent)

89.646 mL → 321.207 mL

ΔV = 231.56 mL

1 mL ΔV equivalent to 1.0077 g H_2O

2.7.2.2 *A Simple, Direct Approach to Calculating the Quantities of Melted Ice contained in Brines*

This approach consists of developing specific volumes (mL/g) of the deicer liquids, and subtracting the specific volumes of 1 g deicer from collected brine volumes per gram of deicer. The difference between specific volume and collected column represents within 1 percent the weight of ice melted; the melted ice dilutes the liquid deicer to a concentration which does not have to be established.

2.7.2.2.1 Step 1: Determination of Specific Volumes of Liquid Deicer. Specific volumes employed in calculations of quantities of ice melted should be specific volumes at test temperatures. Densities of deicer liquids at temperatures below 32°F (0°C) are not available in the literature. Sufficient data is, however, available at 32°F (0°C) and at higher temperatures to permit extrapolation to low temperatures. Table B-4 lists specific volumes developed in this manner for ethylene glycol, 37.3 percent $CaCl_2$, and 26.3 percent $CaCl_2$.

It is apparent from the data in the tables that the following specific volumes are appropriate for use at the temperature range 25° to 5°F (–4° to –15°C).

Ethylene glycol	0.886 mL/g
37.3 percent $CaCl_2$	0.725 mL/g
26.3 percent NaCl	0.824 mL/g

Table B-4. Specific volumes of liquid deicers

	Temperature	mL/g
Ethylene glycol	68°F	0.896
	25°F	0.888
	15°F	0.886
	5°F	0.884
37.3 percent $CaCl_2$	68°F	0.733
	25°F	0.726
	15°F	0.725
	5°F	0.723
26.3 percent NaCl	50°F	0.830
	32°F	0.827
	25°F	0.825
	15°F	0.824
	5°F	0.822

2.7.2.2.2 Step 2: Procedure for Calculating Quantities of Melted Ice. The procedure for conversion of Step 1 data (milliliter brine per gram deicer) to quantities of ice melted (gram ice melted per gram deicer) is as follows.

Step 2-1: Selection of the appropriate value for deicer specific volume

a. Literature data for deicers of known composition. Literature sources include:

- *International Critical Tables*, Vol. III. Densities are reported for numerous aqueous solutions at several temperatures and concentrations.

- *CRC Handbook of Chemistry and Physics*, 69th Edition, Section D.

Freezing temperatures, densities, and other properties are tabulated for aqueous solutions. Densities are given for one temperature, normally 68°F (20°C). Derivation of densities from literature data will usually require interpolation between adjacent concentrations; and extrapolation to temperatures below 32°F (0°C).

b. Densities determined experimentally as specified in ASTM D 3505-85 or D 1122-84.

Calculations of specific volumes are accomplished by dividing 1 by the selected or derived density.

$$\text{Specific volume, } \frac{\text{mL}}{\text{g}} = \frac{1}{\text{g/mL}}$$

Step 2-2: Calculation of Quantities of Ice Melted

Procedure: Subtract the specific volume of the liquid deicer from tabulated values of milliliter brine per gram deicer.

Example data: Step 2, Ice melted per unit weight deicer.

Example test data obtained for ethylene glycol, 37.3 percent $CaCl_2$, and 26.3 percent NaCl are presented in Tables B-5, B-6, and B-7.

If desired, quantities of ice melted per gram of calcium chloride solution and sodium chloride solution can be converted to grams of ice melted per gram of dry weight calcium chloride and sodium chloride by multiplying by 1/0.373 and 1/0.263, respectively.

Results of these conversions for 60-min data, and equilibrium/ calculated data, are shown in Table B-8.

Table B-5. Example ice melting test data for ethylene glycol (EG)[a] (based on averages for all replicates)

Time (min)	25°F		15°F		5°F	
	mL Brine per g EG	Ice melted, g/g EG	mL Brine per g EG	Ice melted, g/g EG	mL Brine per g EG	Ice melted, g/g EG
10	3.85	2.97[b]	2.87	1.99[b]	2.54	1.66[b]
20	4.77	3.89	3.22	2.34	2.55	1.67
30	5.31	4.43	3.44	2.56	2.64	1.76
45	5.81	4.93	3.60	2.72	2.72	1.84
60	6.12	5.24	3.71	2.83	2.77	1.89
Equilibrium,[c] calculated	8.72	7.83	4.32	3.43	3.16	2.28

[a] Test information

Average deicer wt.,	25°F, 4.1704 g (four replicates) 15°F, 4.2193 g (four replicates) 5°F, 4.137 g (three replicates)

Standard error, percent

25°F:	range 0.3 to 2.3 average, 1.0
15°F:	range 0.5 to 0.9 average, 0.7
5°F:	range 0.3 to 2.2 average 1.0

[b] Gram ice melted/g EG = mL brine/g EG -0.885.

[c] Equilibrium g ice melted/g EG calculated from literature values for EG concentrations in solutions at equilibrium with ice:

25°F: $\frac{1\ \text{g EG}}{0.1132} - 1 = 7.83$

15°F: $\frac{1\ \text{g ED}}{0.226} - 1 = 3.43$

5°F: $\frac{1\ \text{g EG}}{0.3046} - 1 = 2.28$

Table B-6. Example ice melting test data for 37.3 percent $CaCl_2$[a]

	25°F		15°F		5°F	
Time (min)	ml Brine/g solution	g Ice melted/g solution	mL Brine/g solution	g Ice melted/g solution	mL Brine/g solution	g Ice melted/g solution
10	2.37	1.65[b]	1.82	1.10[b]	1.50	0.78[b]
20	2.75	2.03	2.01	1.29	1.58	0.86
30	3.07	2.35	2.10	1.38	1.61	0.89
45	3.28	2.56	2.17	1.45	1.62	0.90
60	3.45	2.73	2.20	1.48	1.63	0.91
Equilibrium,[c] calculated	4.74	4.01	2.46	1.73	1.812	1.08

[a] Test information
Average of three replicates
Average deicer wt., 25°F, 5.2193 g
15F, 5.1843 g
5°F, 5.2649 g

Standard error, percent
25°F: range, 1.6 to 3.6
average, 2.1

15°F: range, 0.3 to 0.7
average 0.5

5°F: range, 0.3 to 1.6
average, 0.9

[b] Grams ice melted per g deicer solution = mL brine/g -0.725.

[c] Equilibrium quantities of ice melted per g deicer solution calculated from literature equilibrium brine concentrations.

$$25°\text{F}: \frac{0.373}{0.0745} - 1 = 4.007$$

$$15°\text{F}: \frac{0.373}{0.1365} - 1 = 1.73$$

$$5°\text{F}: \frac{0.373}{0.17925} - 1 = 1.08$$

Table B-7. Example ice melting test data for 26.3 percent NaCl[a]

	25°F		15°F		5°F	
Time (min)	mL Brine/g solution	g Ice melted/g solution	mL Brine/g solution	g Ice melted/g solution	mL Brine/g solution	g Ice melted/g solution
10	1.97	1.15[b]	1.46	0.64[b]	1.06	0.24[b]
20	2.33	1.51	1.51	0.69	1.09	0.27
30	2.51	1.69	1.57	0.75	1.09	0.27
45	2.63	1.81	1.62	0.80	1.10	0.28
60	2.81	1.99	1.62	0.80	1.11	0.29
Equilibrium,[c] calculated	4.01	3.19	1.79	0.96	1.23	0.40

[a] Test information

Average of three replicates

Average deicer wt., 25°F, 4.6273 g
15°F, 4.6633 g
5°F, 4.5916 g

Standard error, percent
25°F: range, 0.6 to 1.7
average, 1.1

15°F: range, 1.0 to 1.8
average 1.2

5°F: range, 0.3 to 1.5
average, 1.1

[b] Grams ice melted per g deicer solution = mL brine/g -0.825.

[c] Equilibrium quantities of ice melted per g deicer solution calculated from literature equilibrium brine concentrations.

$$25°F\text{: } \frac{0.263}{0.0628} - 1 = 3.188$$

$$15°F\text{: } \frac{0.263}{0.1345} - 1 = 0.955$$

$$5°F\text{: } \frac{0.263}{0.1882} - 1 = 0.3975$$

Table B-8. Ice melted (g) per g dry weight calcium chloride and sodium chloride

	60 Min	Equilibrium (calculated)
$CaCl_2$ (37.3%)		
25°F	7.32	10.74
15°F	3.96	4.64
5°F	2.44	2.90
NaCl (26.3%)		
25°F	7.57	12.12
15°F	3.04	3.63
5°F	1.10	1.51

2.8 Calculation of Theoretical or Equilibrium Quantities of Ice Melted

Theoretical quantities of ice which may be melted by a deicer may be calculated accurately when reliable data are available on freezing points and associated deicer concentrations. Literature data are usually provided for pure chemicals, however, and it is conceivable that little or no data may be available for a candidate deicer. Theoretical quantities of ice melted for mixed nonhomogeneous deicers cannot be determined with these methods.

2.8.1 Calculations for Pure Chemicals

The *CRC Handbook of Chemistry and Physics*, 69th Edition contains extensive tables which list the freezing temperatures, concentrations, and other data for aqueous solutions of chemicals. Interpolation is usually needed to identify the concentration associated with a freezing temperature of interest. When the concentration, in weight percent, associated with the desired temperature has been identified, the weight of brine expected at equilibrium is calculated by dividing deicer dry or pure weight by (equilibrium concentration ÷ 100), as follows:

$CaCl_2$ at 25°F: brine concentration at equilibrium—7.45 percent by weight

Therefore:

1 g $CaCl_2$ = 0.0745•equilibrium brine weight

$$\text{Equilibrium brine wt./g } CaCl_2 = \frac{1}{0.0745} = 13.42 \text{ g}$$

$$\begin{aligned} \text{Water in brine} &= \text{brine wt.-wt. } CaCl_2 \\ &= 13.42\text{-}1 \text{ g } CaCl_2 \\ &= 12.42 \end{aligned}$$

Calculations for a calcium chloride solution (37.3 percent solution) are as follows, at 25°F:

Wt. $CaCl_2$ per gram deicer solution = 0.373

0.373 = 0.0745•equilibrium brine wt.

$$\text{Equilibrium brine wt.} = \frac{0.373}{0.0745} = 5.007 \text{ g}$$

Ice melted = (brine wt.) - (deicer solution wt.) = 5.007-1 = 4.007

2.8.2 Calculations for Commercial Quality Deicing Chemicals

Deicing chemicals typically contain insoluble matter, 2 to 3 percent by weight of soluble chemicals other than the principal ingredient, and may contain water.

Water and insoluble matter should be excluded from equilibrium calculations. The 2 to 3 weight percent of soluble impurities should be included.

For example, rock salt (sodium chloride) containing 95 percent NaCl, 2 percent other alkali chlorides, and 3 percent water insolubles plus calcium sulfate should be considered for practical purposes to be 97 percent sodium chloride.

Pellet calcium chloride composed (for example) of 91 percent $CaCl_2$, 5 percent H_2O, 2 percent other halides, and 2 percent water insolubles should be considered for practical purposes to be 93 percent calcium chloride.

2.8.3 Calculations for a Known Chemical, Freezing Point/Composition Data Lacking

The freezing point depression constant for water can be used to estimate equilibrium ice melting capacities of a chemical of known molecular weight, and knowledge of the number of ions per molecular weight in aqueous solutions.

A freezing point depression constant is defined as the difference between 32°F (0°C) and the freezing point temperature of an aqueous solution containing one equivalent weight of the chemical in 1,000 g H_2O. For water, the freezing point depression constant is 1.86°C. The freezing point of a solution is calculated as shown in the following example.

Chemical: molecular wt. 50 g
equivalent wt. 25 g (2 ions/molecule)

Concentration: 10 percent in water, therefore 10 g chemical plus 90 g H_2O

a. g chemical/g H_2O = $\frac{10}{90}$

b. g chemical/ g H_2O x 1,000 = g chemical/1,000 g H_2O

$$\left(\frac{10}{90} \text{ x } 1{,}000 = 1{,}009/9 \text{ g per } 1{,}000 \; H_2O\right)$$

c. g chemical/1,000 g H_2O ÷ g chemical/eq. wt. = no. of equivalents per 1,000 g H_2O

$$\left(\frac{1{,}000}{9} \div 25\right) = \frac{40}{9} \text{ equivalents per } 1{,}000 \text{ g } H_2O$$

d. Number of equivalents/1,000 g H_2O•(-1.86) = freezing temperature, °C

$$\frac{40}{9} \text{ x } -1.86 = -8.27°\text{C}$$

e. $\dfrac{\text{g } H_2O}{\text{g chemical}}$ = ice melting capacity per gchemical

$$\frac{90 \text{ g } H_2O}{10 \text{ g}} = 9 \text{ g ice melted per g chemical at } -8.27°\text{C}$$

Calculated freezing temperatures and melting capacities are usually close to measured values in dilute solutions (i.e., for freezing temperatures of the order of -5° to -8°C). At lower temperatures, substantial differences may exist between measured and calculated data.

SHRP H-205.3

Test Method for Ice Penetration of Solid Deicing Chemicals

3.1 Scope

This test method provides data on the times required at various temperatures, for the penetration of solid deicers through ice layers likely to be encountered in highway deicing.

3.2 Applicable Documents

- SHRP H-205.1 Ice Melting of Solid Deicing Chemicals

3.3 Summary of Method

Pellets of solid deicers are placed on the surface of an ice specimen amended with an appropriate dye material, at regulated constant ambient temperatures, and depths of penetration are measured at several time intervals over a 60-min period.

3.4 Significance and Use

- This test can be used to compare generic solid chemical deicers, modifications to solid deicers, or new solid chemical deicers with respect to their ability to penetrate ice layers at specified temperatures during specified time intervals.

- This test can provide data which may indicate whether significant ice undercutting and ice disbondment can be expected when using a given solid chemical deicer.

- Nonhomogeneous (mixed) deicers may be dissolved in a selected amount of water and tested as liquid deicer via SHRP H-205.4.

3.5 Test Equipment

3.5.1 Test Apparatus (See Figure B-3, page 146)

The recommended ice penetration apparatus consists of a 1/2- x 2- x 8-in (1.25- x 5.08- x 20.32 cm) Plexiglas® plate cemented to a base 1 1/2 in. (3.81 cm) wide x 1/4 in. (0.635 cm) thick x 8 in. (20.32 cm) long with the 2-in. (5.08 cm) dimension in a vertical position. Ten 35-mm deep cavities are drilled in the center of the plate at 17-mm spacings with a 5/32-in. drill. These cavities are then enlarged in a countersink fashion to yield a surface cone 10 mm in diameter and 5 mm long at the point of intersection of the cone with the 5/32-in. cavity.

A conventional drill press and drill bit are employed. The drilling operations must be carried out slowly and with frequent withdrawal of the bits to remove Plexiglas® strands. This mode of drilling prevents overheating and favors the formation of clear rather than whitened or crazed surfaces. The countersink bit employed was a center reamer, four-flute construction, 60-degree angle, 1/2-in. diameter body (Cleveland Cutting Tools, Catalog No. 46210).

3.5.2 Equipment Accessories

3.5.2.1 *Penetration Depth Measurement*

To facilitate measurement of penetration, a measuring device with marks spaced 1 mm apart is placed behind the test apparatus. The device can conveniently be fabricated by converting millimeter-scale graph paper to overhead projector transparencies and cutting out an appropriately sized section. This piece may either be affixed to the test apparatus or mounted on a stand so that the test apparatus can be placed in front of the measuring device. A stand must be constructed to hold the test apparatus so that it can be positioned for observation through a window/door in the regulated temperature test enclosure.

3.5.2.2 *Deicer Handling Equipment*

Individual deicer pellets in the range of 22 to 27 mg are required. An analytical balance is accordingly required. Weighed pellets may be either placed in individual vials or several pellets of a given material placed in one vial.

Hygroscopic deicers (e.g., calcium chloride pellets) should be handled in a dry glove box and stored in tightly stoppered vials.

Pellets are transferred to the ice surface with forceps. Before testing, pellets are most conveniently handled by placement in pellet caddies constructed from Plexiglas® plates by drilling shallow cups at intervals. Pellets in the cups can be more readily observed if the caddies are painted with black enamel.

A timer, preferably a digital timer that can be activated at test start, is required.

The test chamber must be equipped with a suitable temperature indicating device. Temperature monitoring will be more convenient if a thermometer is attached to the stand holding the test apparatus.

3.5.3 Regulated Temperature Test Enclosures

Ice penetration test results vary by as much as 10 percent per degree Fahrenheit. An enclosure which can be regulated to within ±0.5°F (±0.28°C) of the designated test temperature is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The enclosure should have a temperature capability ranging from 25°F to at least 5°F (-4°C to at least -15°C).

Options for a controlled temperature include: (1) a box equipped with hand ports and a window, and placed inside a walk-in cold room, and (2) a similar enclosure placed in an upright freezer, and equipped with hand ports and viewing windows fabricated and installed in the freezer door.

3.5.3.1 *Cold Room Test Enclosure*

A cold room testing enclosure, which has been extensively utilized, is described in Annex 1 of Test Method SHRP H-205.1.

3.5.3.2 *Upright Freezer Test Enclosure*

A similar testing enclosure designed for use in an upright freezer is presented in Annex 2 of Test Method SHRP H-205.1.

3.6 Test Procedure

3.6.1 Water Supply

Deionized or distilled water is required. Approximately 100 mL of water should be deaerated by brief boiling, then cooled to near ambient room temperature.

3.6.2 Dye Solution and Dye Applicator

A dye solution is prepared by weighing 1 g of commercial Bulls-Eye™ solution (50 weight percent in water) into a 4-oz wide-mouth jar containing 20 mL of deionized water. A plastic disposable pipette tip (e.g., 1 to 100 μL yellow flex tips for use with Eppendorf pipettes) attached to a 4- to 5-cm length of glass tubing is employed to transfer dye to ice surfaces.

3.6.3 Ice Preparation

Deaerated water is drawn into a syringe, the needle inserted into the cavities, and the cavities filled while withdrawing the needle. Bubbles in the water column (usually none) should be forced out if necessary.

The ice penetration apparatus is placed in a freezer until the water is completely frozen. A globule of ice will protrude from the surface. The globules are melted with an aluminum iron; excess water is ordinarily left on the surface. The apparatus is placed in the constant temperature chamber and equilibrated overnight to the operating temperature.

Final preparation of the ice specimen occurs 2 to 3 hr before the tests by remelting surface ice and wiping the surface with a tissue to remove excess water. If necessary, small droplets of water may be added via a glass stirring rod dipped in water, perhaps followed by a superficial ironing to relevel the surface.

After a smooth, flush ice surface is achieved, 2 to 3 dye droplets are transferred to the surface of the ice in each cavity, as follows. Dip the pipette tip in the dye solution, and transfer the adhering droplet to an off-center position. Repeat once or twice, placing droplets adjacent to each other in a semicircle. Liquid on the tip tends to freeze, so hold tip in solution 3 to 4 sec between transfers. Dye droplets are approximately pinhead in size and weigh slightly less than 1 mg. Three droplets accordingly should weigh about 2.5 mg and contain about 0.2 mg of dye ingredient which should have the melting capacity of about 0.03 mg NaCl.

The system is then allowed to equilibrate to temperature for 1 to 2 hr.

3.6.4 Test Initiation

Deicer pellets (22 to 27 mg) are transferred to the cold room 30 to 60 min before test initiation and transferred to the pellet caddy immediately before the test.

Pellets are then transferred as quickly as possible from left to right on the test apparatus. The timer is activated when the last pellet is in place. Placement of 10 pellets requires 30 to 40 sec.

NOTE: Nonhomogeneous (mixed) deicers may be dissolved in a selected amount of water and tested as liquid deicers using SHRP Method H-205.4.

3.6.5 Data Recording

Penetration depths are observed and recorded at preselected time intervals. Recommended time intervals are 3, 5, 10, 15, 20, 30, 45, and 60 min. Readings should be taken from left to right (i.e., in the order of pellet placement) and started approximately 30 sec before the designated time.

Recorded data consist only of the maximum depth of the ice melted in the cavities. Data accounting is best conducted via ruled data sheets that contain reminders to enter all test parameters (e.g., date, deicer, temperature, and test replicate numbers).

3.6.6 Test Replication

Five replicates are recommended. If several deicers are to be tested, it is recommended that deicers be alternated on the test apparatus and tested on more than one apparatus.

3.7 Data Outputs and Reproducibility

3.7.1 Data Outputs

Data displays consist of tables which contain penetration depths for each replicate and time interval, calculated average penetration depths, and calculated standard errors. Data may additionally be displayed graphically, usually with smoothed curves, hand-drawn through the average data points.

Standard errors are calculated by summing the squares of differences between observed and average penetration depths, dividing the sum of the squares by the product of the number of replicates (n) times (n - 1), and calculating the square root of the resultant number:

Example: n = 5
Observed penetration depths: 6, 5, 7, 5.5, 6.5

Average = (6 + 5 + 7 + 5.5 + 6.5) ÷ 5 = 6

$$\text{S.E.} = \left(\frac{(6-6)^2 + (6-5)^2 + (6-7)^2 + (6-5.5)^2 + (6-6.5)^2}{5 \times 4}\right)^{1/2} = 0.354$$

Data interpretations may include development of times required for penetration through selected thicknesses of ice. Appropriate thicknesses are 1/8 in. (~ 3.2 mm) and 1/4 in. (~ 6.4 mm). These may be approximately derived by inspection of data or taken from plots of penetration versus time. In this regard, times for penetration to a depth of 1/8 in. (~ 3.2 mm) are required for interpretation and display of results of ice undercutting data.

3.7.2 Data Reproducibility

Penetration depths can be observed and recorded to the nearest 0.1 mm, e.g., 2.0 ± 0.1 mm. Depths less than 1 mm are difficult to pinpoint and are recorded chiefly to define the initiation of penetration. Maximum depths of penetration at 25°F (-4°C) are about 25 mm. Potential reading errors are thus small at high penetration depths and of the order of 10 percent at 1 to 2 mm. Standard errors of the order of 10 percent of the average of five replicates may be anticipated. The range found for standard errors extends from about 2 to 20 percent, with the higher standard errors usually occurring at small (1 to 2 mm) penetration depths.

3.8 Example Test Results

Example ice penetration test results for solid chemical deicers are presented in Table B-9.

Table B-9. Example of ice penetration test results

	Rock salt pellets (22-26 mg)				Calcium chloride pellets	
	20°F		15°F		5°F	
Time (min)	Penetration depth avg. (mm)	Standard error (%)	Penetration depth avg. (mm)	Standard error (%)	Penetration depth avg. (mm)	Standard error (%)
3	1.0	6	0.5	30	1.4	12
5	1.9	11	1.3	22	2.3	7
10	3.6	13	2.1	13	4.2	12
15	5.1	9	3.2	12	6.2	11
20	6.7	13	4.0	13	7.6	11
30	10.0	18	5.7	8	9.3	8
45	13.1	10	8.9	11	9.4	9
60	13.4	9	10.4	12	9.5	8

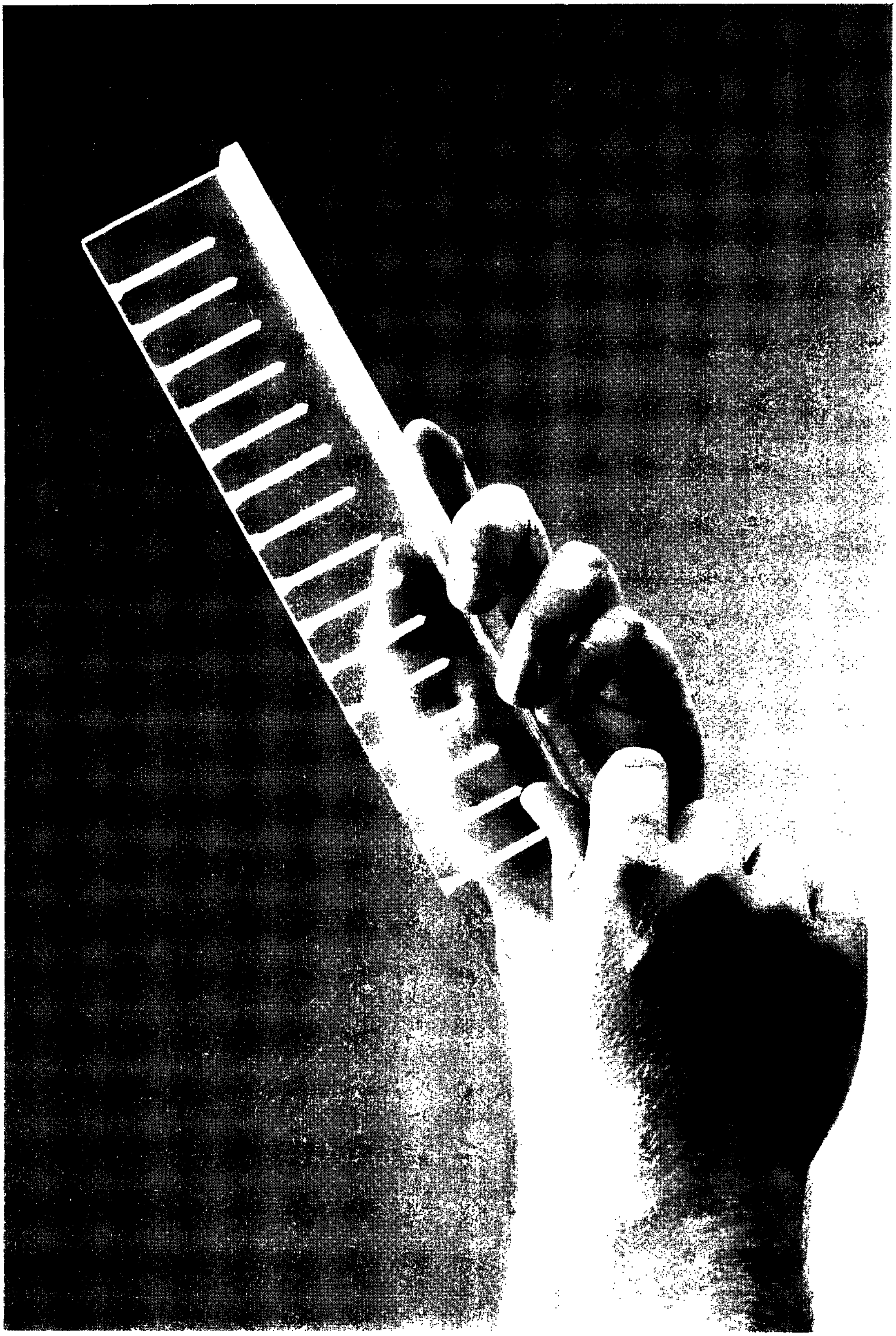


Figure B-3. Ice penetrating test apparatus.

SHRP H-205.4

Test Method for Ice Penetration of Liquid Deicing Chemicals

4.1 Scope

This test method provides for determination of the capability of a liquid deicer to melt through ice and penetrate to a pavement substrate/ice interface.

4.2 Applicable Documents

- SHRP H-205.1 Test Method for Ice Melting of Solid Deicing Chemicals
- SHRP H-205.3 Test Method for Ice Penetration of Solid Deicing Chemicals

4.3 Test Method Summary

The method and procedures presented for measuring or, more accurately, observing the penetration behavior of liquid deicers essentially provides for containment/localization of a quantity of liquid deicer on a relatively small ice surface, and for observation of melting which occurs chiefly by propagation of a uniform melt front downward through the ice. The results of the liquid deicer penetration test should probably be viewed as measures of ice melting capacity, with ice melting capacity being evidenced by the formation of a pool of brine contained in a volume and at a depth defined by the surface area of ice exposed to the liquid deicer.

4.4 Significance and use

- This test method can be used to compare generic liquid chemical deicers,modifications to liquid deicers, or new liquid chemical deicers with respect to their ability to penetrate ice layers at specified temperatures during specified time intervals.

4.5 Equipment and Procedures

The equipment and procedures employed for tests of ice penetration are essentially the same as those employed for tests with solid particle deicers. These are presented in SHRP H-205.3, Test Method for Ice Penetration by Solid Deicers.

Liquid deicers differ from solid particle deicers in two respects relative to a penetration test. First, a liquid normally will spread over an ice surface in thin films. Film thickness will result in barely observable depth of melting into the ice surface. Significant penetration will occur only when deicer liquids are localized in depressions in the ice. Second, liquids exhibit little tendency for the directed penetration exhibited by solid particles, but instead tend strongly to melt ice uniformly downward from the liquid/ice interface.

The procedural modifications for testing liquid deicers are as follows.

4.5.1 Preparation of Ice Surfaces

In the test for solid deicers, the surface of the ice in the penetration cavities is made level and flush with the Plexiglas® surface by melting excess or protruding ice with an aluminum plate, striking off and wiping off excess water, and refreezing surface water so that the ice surface is smooth and flush with the upper edge of the conically shaped cavity which is 10 mm in diameter.

With liquids, the ice ironing process is again utilized. However, melted water at the surface is wiped off with a tissue so that the resulting ice surface is a fraction of a millimeter below the rim of the cavity.

4.5.2 Liquid Test Sample Handling and Application

4.5.2.1 *Incorporation of Dye*

Approximately 5 mL of liquid deicer is placed in a screw-capped vial with a capacity of about 20 mL. Fifteen to 20 mg of Bulls-Eye™ dye is added to the liquid.

4.5.2.2 Test Sample Application to Ice Surface

The liquid deicer is cooled either to test temperature or to a temperature which will not result in precipitation of dissolved deicer. A 30-µL sample is drawn into a disposable plastic pipetting tip of a pipetting system designed for isolation of known liquid volumes, and the 30-µL test sample discharged onto the surface of the ice. Test sample weights will be in the range of about 34 to 42 mg, depending on deicer density.

Five replicates are recommended; these can be applied in a single test array in 30 to 45 sec.

Several suitable pipetting systems are commercially available. An Eppendorf pipette, Model 4710, 10 to 100 µL, equipped with yellow disposable tips was employed in developmental tests.

4.5.3 Temperature Regulated Test Enclosures

Ice penetration test results vary by as much as 10 percent per degree Fahrenheit. An enclosure which can be regulated to within ±0.5°F (±0.28°C) of the designated test temperature is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The enclosure should have a temperature capability ranging from 25°F to at least 5°F (-4°C to at least -15°C).

Options for a controlled temperature include: (1) a box equipped with hand ports and a window, and placed inside a walk-in cold room, and (2) a similar enclosure placed in an upright freezer, and equipped with hand ports and viewing windows fabricated and installed in the freezer door.

4.5.3.1 *Cold Room Test Enclosure*

A cold room enclosure, which has been extensively utilized for deicer testing, is described in Annex 1 of Test Method SHRP H-205.1.

4.5.3.2 *Upright Freezer Test Enclosure*

A similar test enclosure designed for use in an upright freezer is presented in Annex 2 of Test Method SHRP H-205.1.

4.6 Data Recording and Analysis

Penetration depths, in millimeters, are recorded at several time intervals (5, 10, 15, 20, 30, 45, and 60 min) over a 1-hr period, as with tests with solid particle deicers. Data evaluation, such as error analysis, is conducted as specified in SHRP H-205.3.

4.7 Example Ice Penetration Test Results

Reference penetration results were obtained with ethylene glycol, 37.3 percent aqueous calcium chloride, and 26.3 percent aqueous sodium chloride. The chloride solutions were prepared by saturating water with reagent grade chlorides at 32°F (0°C). These solutions were precooled to 34° to 36°F (+1° to +2°C) prior to addition to the ice to prevent precipitation of solutes. The ethylene glycol was precooled to the test temperatures of 5°F (-15°C), 15°F (-9.44°C), and 25°F (-3.89°C).

Test conditions and test results (averages of five replicates) are presented in Table B-10.

Table B-10. Example ice penetration test results with liquid deicers

Test parameters

Deicers:	Test temperatures:
Ethylene glycol (EG), 31-34 mg	−15°F
37.3 percent $CaCl_2$, 41-43 mg	−9.44°F
26.3 percent NaCl, 35-38 mg	−3.89°F

Number of Replicates: 5

Test Results mm penetration, average of 5 tests

	EG		37.3% $CaCl_2$		26.3% NaCl	
Time (min)	mm	Standard error (%)	mm	Standard error (%)	mm	Standard error (%)
			−15°C (5°F)			
5	0.42	4.8	0.62	6.0	Barely detectable penetration	
10	0.78	4.8	0.73	4.1		
15	0.97	2.1	0.86	4.7		
20	1.12	3.3	0.99	2.5		
30	1.12	3.3	1.01	2.4		
45	1.18	5.4	1.07	3.2		
60	1.21	3.3	1.08	3.1		
			−9.44°C (15°F)			
5	0.76	5.3	0.76	3.2	Maximum penetration 0.3-0.4 mm	
10	1.06	3.8	1.10	2.9		
15	1.26	3.2	1.16	2.1		
20	1.82	4.4	1.50	3.0		
30	2.10	1.5	1.58	3.7		
45	2.23	1.3	1.78	2.1		
60	2.88	2.8	1.95	1.6		

Table B-10. (Continued)

Time (min)	EG mm	EG Standard error (%)	37.3% $CaCl_2$ mm	37.3% $CaCl_2$ Standard error (%)	26.3% NaCl mm	26.3% NaCl Standard error (%)
			−3.89°C (25°F)			
5	1.32	7.3	1.2	11	0.2	–
10	1.96	3.7	1.6	6.0	0.3	–
15	2.28	3.2	2.1	7.3	0.4	–
20	2.60	4.7	2.3	4.7	0.6	–
30	3.50	3.1	3.1	5.5	1.0	6
45	4.72	4.6	3.3	5.1	1.1	3
60	5.48	3.8	3.5	4.3	1.6	7

SHRP H-205.5

Test Method for Ice Undercutting by Solid Deicing Chemicals

5.1 Scope

This test method covers measurement and comparison of the ice undercutting capabilities of solid deicers on ice firmly and uniformly bonded to a standard mortar specimen under specified conditions.

5.2 Applicable Documents

5.2.1 ASTM Standards

- C 109-90 Standard Test Method for Compressive Strength of Hydraulic Cement
- C 778-90 Standard Specifications for Standard Sand

5.2.2 Other Documents

- SHRP H-205.1 Ice Melting of Solid Deicing Chemicals

5.3 Test Method Summary

The ice undercutting test involves the placement of individually weighed pellets of solid deicer on the surface of a uniform thickness of ice frozen on a substrate representing a pavement material, and measuring the area of ice melted between the ice and the substrate over a selected time interval.

The overall physical and chemical processes involved in the test consist of initial melting on the surface of the ice, followed by penetration through the ice via a small diameter cavity, and succeeded by melting a thin layer of ice between the substrate and ice. The resultant undercut area is rendered visible via a small quantity of dye material, namely bromcresol green. Photographs taken at selected time intervals furnish records of the undercutting process and serve as a means for measurement of undercut areas.

A mortar mix substrate is mixed and cured in accordance with ASTM specifications for a mortar mix employed for strength measurement (ASTM C 109-90, Standard Test Method for Comprehensive Strength of Hydraulic Cement). The mold consists of a plastic Rubbermaid™ tray, 6 in. x 9 in. x 2 in. deep, with the bottom surface sand-blasted to yield a textured surface with indentations approximately 1.7 mil, peak to valley. The surface of the mortar exposed to the sand-blasted area exhibits a texture corresponding to peak to valley indentations of about 2 mils.

Duct tape and latex caulk are employed to construct a barrier to contain water on the textured surface of the mortar specimen. The mortar specimen and an inventory of water are chilled to about 35°F (+2°C). The specimen is placed on a level, cold metal plate placed in the bottom of an insulated chamber and precooled water added to yield ice 1/8 in (3.175 mm) thick. The air temperature above the specimen is maintained at about 33° to 35°F (+1° to +2°C), and the plate temperature at about +14°F (-10°C) so that the water freezes slowly and from the substrate up.

The mortar/ice specimen is then placed in a temperature-regulated system or enclosure and equilibrated to the selected test temperature. Small quantities of an aqueous dye solution are placed on selected spots on the ice surface, and the dye solution is allowed to freeze. Weighed deicer pellets are placed on the dye spots, and the dye/undercutting patterns are photographed at selected intervals over a 60-min time period.

The preferred and recommended procedure for measuring undercut areas consists of measurement of the dimensions of dye spots exhibited in projected images of photographic slides. Areas calculated from dye spot dimensions are converted to undercut areas per unit weight of deicer (cm^2 undercut per gram deicer).

5.4 Significance and Use

- This test method is used to evaluate the ice undercutting capabilities of homogeneous pellets of solid deicers on ice uniformly bonded to a standard mortar substrate.
- This test method includes adaptations for the evaluation of undercutting capabilities of homogeneous pellets of solid deicers on ice bonded to other substrates such as concrete or asphalt.
- This test can be used to compare deicers and to determine the time and temperature relationships to ice undercutting under idealized conditions.
- This test method is designed to predict a lower limit for the extent of undercutting by solid deicers on ice uniformly bonded to a standard substrate.
- Nonhomogeneous (mixed) deicers may be dissolved in a selected amount of water and tested by liquid deicers using SHRP Method H-205.6.

5.5 Equipment and Materials Preparation

5.5.1 Mortar Molds

The recommended mold for casting mortar specimens consists of a Rubbermaid™ tray, 6 in. x 9 in. x 2 in. deep. The four small "legs" on the tray are removed so that the bottom is flat. The inside bottom of the tray is sandblasted with natural silica sand to yield a textured surface with 1.5 to 2.0 mil peak to valley indentations. Specifications for the sand are given in Section 4.1, 20-30 Sand, ASTM C 778-90a, Standard Specifications for Standard Sand.

5.5.2 Mortar Specimen Preparation

The recommended mortar mix contains sand, water, and portland cement in the following proportions by weight:

1.0 part cement
0.485 part water
2.60 parts sand

The sand is a fine sand, with particle sizes corresponding approximately to the following:

Mesh size	Percent passing
16	99-100
30	45
40	30
50	15
60	6
100	< 1

The recommended procedure for fabrication of the mortar specimens is as follows:

1. Eighty- to ninety-pound batches of mortar are mixed, preferably in a cement mixer.
2. Mortar sufficient to fill a 9 in. x 6 in. x 2 in. Rubbermaid™ tray is placed in individual trays.
3. The filled trays are vibrated to remove air bubbles, and the surface of the mortar is struck and leveled with a suitable striker.
4. The specimens are cured overnight at 100 percent relative humidity. Specimens are then removed from the trays, placed in saturated lime water, and cured for 28 days.

The mortar specimen surface that was in contact with the inside bottom of the tray mold will serve as the ice growing substrate.

5.5.3 Water/Ice Retention System

A water retention dam or edge is constructed around the outside of the cured mortar specimen by placing a bead of latex caulk around the outside edge of the mortar specimen, near the textured surface, followed by placing a 1-in. wide strip of duct tape around the edge so that a 1/4-in. to 3/8-in. lip protrudes above the surface. Small strips of tape should be placed as reinforcements at each of the four corners. The tape is finger pressed to the mortar so that a small bead of caulk is visible around the edge. The caulk is allowed to cure for several hours.

5.5.4 Special Considerations

The cleanliness or purity of substrates is known to affect the nature of bonding between ice and substrates. Care should be taken to protect mortar specimens from exposure to dusts and laboratory chemicals, and finger contact with mortar surfaces should be avoided.

The latex caulk should not be siliconized. Standard handling procedures for cured mortar specimens consists of thorough flushing with tap water followed by a thorough deionized water rinse.

The advisability of reusing mortar specimens is questionable. However, it has been found that if used test specimens are brought into the laboratory and immediately flushed with tap water no visual traces of dye remain. Such specimens have been reused three to four times with no evidence of poorly bonded ice, and results have corresponded closely with results obtained with fresh specimens.

5.5.5 Ice Preparation

5.5.5.1 *Equipment*

Ice preparation equipment requires the following:

1. A refrigerator for precooling mortar specimens and water to approximately 35°F (+2°C).

2. A freezing chamber constructed so that ice can be frozen in a bottom-up mode. A suitable chamber consists of a 1/2-in.-thick metal plate approximately 2 1/2 ft x 2 1/2 ft on which is placed a box constructed of Styrofoam® insulation and covered with a

> Styrofoam® lid. The box is approximately 15 in. deep. Inside this box is placed a square, 6-in. tall, 1/2-in.-thick plywood box with no bottom or top. The box is equipped with two removable plywood pieces that serve as a loosely fitted lid which permits air circulation and does not permit direct exposure of the mortar/water specimens to radiant energy emitted by light bulbs employed to maintain air temperature slightly above the freezing point of water.

The metal plate is exposed to an ambient temperature of the order of 15°F (-10°C). Air temperatures are maintained, via a rheostated power supply and a light bulb, at 33° to 35°F (1° to 2°C).

This freezing chamber is most conveniently employed in a walk-in cold room set to operate at about 15°F (-10°C). Optionally, the freezing chamber may be placed in a freezer. In this connection, the recommended plate temperature of 15°F (-10°C) is not critical. Satisfactory ice specimens have been obtained with nominal plate temperatures ranging from about 20° to 0°F (-7° to -18°C). With a plate temperature of 15°F (-10°C), ice specimens are frozen in approximately 2 hr.

5.5.5.2 *Ice Preparation*

The most important consideration relating to reproducible results is the quality of the ice as determined by the freezing method. Rapid freezing, or more specifically, flash-freezing of a surface film of ice should particularly be avoided.

Mortar specimens precooled to approximately 35°F (2°C) are placed in the freezing chamber. Precooled deionized water (96 to 98 mL) is added to the mortar specimen. The auxiliary plywood lids are placed over the specimens, the insulated top is placed over the enclosure, and the heating lamp (100 W incandescent bulb) is activated to near full voltage until air temperature reaches 33° to 35°F (+1° to +2°C). The power to the lamp is then lowered to maintain an air temperature of +33° to +35°F (+1° to +2°C) until the ice is frozen.

Ice frozen in this manner tends to vary from very smooth and level to quite granular with an irregular surface. The smooth and level surfaces are suitable for use in the undercutting tests. The granular and irregular surfaces must be "ironed" (partially melted) with an aluminum plate, and the surface refrozen.

Care must be taken in all steps of mortar specimen preparation and ice freezing to ensure uniform ice thickness [1/8 in. (3.175 mm)]. Thus, mortar specimens should be of uniform thickness, and should be placed on level surfaces during the freezing operation.

5.5.5.3 *Dye Addition*

The recommended dye material is bromcresol green. This material is sparingly soluble in water at ambient temperatures. A saturated dye solution is prepared by adding 75 mg of dye to 50-mL deionized water.

Dye is applied to the ice surface with a standard felt tip marker pen. The pen is prepared for use by punching a small hole in the upper end of the pen barrel, and flushing out residual ink with deionized water using a syringe with the needle inserted into the barrel. The felt tip is squared off by cutting with a razor blade. Dye is applied, as spots, to the surface of the ice by dipping the felt tip in the dye, and briefly pressing the wetted felt tip on the ice. Two applications per spot are required to transfer a sufficient amount of dye. The first dye spot is allowed to freeze before the second application. The dye spots should be separated by about 1 in. (2.5 to 3.0 cm), so that no overlapping of undercut patterns occurs.

The dye spots normally are placed on the ice 1 to 2 hr before initiation of undercutting tests.

5.5.5.4 *Temperature Equilibrium*

The preferred and recommended temperature condition for the undercutting test involves equilibration to the selected temperatures, with temperatures controlled to within ±0.5°F (ca. ±0.3°C). Overnight equilibration of the mortar/ice specimen in a temperature-regulated enclosure is accordingly preferred.

5.5.6 Deicer Materials

Each test involves deicer particles weighed to the nearest 0.1 mg on an appropriate analytical balance. Recommended particle weights correspond approximately to weights of particles in the +6 - 8 mesh size range. Rock salt pellets in this size range exhibit average weights of 30 to 35 mg. Pellet calcium chloride particles generally weigh 20 to 25 mg. Test particle weight uniformity to within ±5 mg of a selected average weight is satisfactory. However, individual particle weights must be known accurately.

Moisture-sensitive deicers (e.g., pellet calcium chloride) should be stored and handled in a dry atmosphere (preferably a dry glove box).

Weighed deicer particles are stored in capped vials and transferred to the cold test facility 1 to 2 hr before the test.

Handling and transfer of deicer particles is conveniently accomplished by a plexiglas plate, 3/8 in. thick, containing small cups fabricated with a 5/8-in drill. The individual pellets are transferred from vials to the caddy, and then transferred to the center of dye spots with forceps.

Nonhomogeneous (mixed) solid deicers may be dissolved in a selected amount of water and tested in liquid deicers using SHRP Method H-205.6

5.5.7 Accessory Equipment

5.5.7.1 *Undercutting Test Apparatus*

The undercutting test apparatus includes a camera loaded with ASA 400 slide film mounted approximately 30 in. above the test specimen; a digital timer positioned so the elapsed time is recorded on photographic records, and length markings (e.g., 5 cm and 10 cm) which also will be recorded on photographs. An optional accessory is a temperature-indicating device (e.g., a low temperature thermometer equipped with a red dye liquid) to display test temperatures on photographs.

Lighting requirements will be dictated by the test enclosure. With a white-walled enclosure, a ceiling mounted 15 W fluorescent bulb augmented momentarily with light from a 60 W incandescent bulb is satisfactory. The incandescent light is activated only for a few seconds during each photograph to minimize radiant heating of test ice.

5.5.7.2 *Regulated Temperature Test Enclosures*

Ice undercutting test results vary by as much as 10 percent per degree Fahrenheit. An enclosure which can be regulated to within ±0.5°F (±0.28°C) of the designated test temperature is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The enclosure should have a temperature capability ranging from 25°F to at least 5°F (-4°C to at least -15°C).

Options for a controlled temperature include: (1) a box equipped with hand ports and a window, and placed inside a walk-in cold room, and (2) a similar enclosure placed in an upright freezer, and equipped with hand ports and viewing windows fabricated and installed in the freezer door.

5.5.7.2.1 Cold Room Test Enclosure. A cold room test enclosure, which has been extensively utilized, is described in Annex 1 of Test Method SHRP H-205.1.

5.5.7.2.2 Upright Freezer Test Enclosure. A similar test enclosure designed for use in an upright freezer is presented in Annex 2 of Test Method SHRP H-205.1.

5.6 Test Procedure (See Figure B-4, page 166)

The test specimen will accommodate 20 deicer pellets. Five replicates per deicer material are recommended; one test specimen can accommodate as many as four materials.

Ten pellets can be loaded on the ice in 1 min or less. Therefore, it is recommended that a 20-pellet set of tests be conducted in two subsets with the second subset initiated at the 5-min mark. The first subset is accordingly loaded and the timer activated; and the second subset loaded commencing at 4 min.

Photographs are taken at the following time intervals for each subset: 5, 10, 15, 20, 25, 30, 45, and 60 min.

5.7 Procedures Using Other Test Substrates

The preferred and recommended method involves using the specified portland cement mortar as the test substrate. However, the undercutting test can be conducted with concrete and asphaltic substrates.

5.7.1 Testing With Concrete Substrates

Portland cement concrete substrates differ from the mortar substrates in two respects: (a) differences in the mix, that is, the presence of aggregates in the mix, differences in size gradations of sand in the mix, and differences in proportions of sand, aggregate, and cement; and (b) physical characteristics of surfaces, which may range from relatively smooth to deliberately roughened or grooved surfaces.

Concrete surfaces may also include core samples taken from existing highways or from laboratory-prepared specimens. Core samples will have surface characteristics which are a combined result of the procedures employed to pour the concrete and prepare its surface and of exposure to traffic and environmental effects. The surfaces of core samples and subsurface materials will also have been chemically influenced by weathering, exposure to ambient chemicals, and by exposure to deicing chemicals, oils and greases, and tire residues.

Undercutting tests may be conducted with substrate specimens which represent a wide range of surface physical and chemical characteristics. Test substrate options range from relatively smooth laboratory-prepared specimens, through surface-roughened laboratory specimens, to the several conditions which may exist with core samples. Laboratory specimens will be essentially virgin, with respect to external contamination and the effects of traffic. Core samples, on the other hand, can be expected to be chemically contaminated and to have surfaces modified physically by mechanical and physical/ chemical processes.

The characteristics of the materials and surfaces employed for undercutting tests will depend on the objectives of the undercutting test. Potential test objectives are described in the following subsections.

5.7.1.1 *Deicer Evaluation or Comparison With a General or Generic Representation of Portland Cement Concrete*

A normally smooth or troweled surface obtained with small laboratory portland cement concrete specimens is recommended. Recommended specimen preparation procedures should be followed, with reasonable care being exercised to prevent surface chemical contamination. Lime-cured specimens should be brushed lightly to remove surface deposits due to the lime, followed by tap water and deionized water flushes.

The undercutting test procedure, for solid deicer particles with those specimens, is that specified for use with smooth, lightly textured mortar specimens. In the event that undercutting patterns are not symmetrical circles, undercut areas should be measured with a planimeter or a computerized stylus.

5.7.1.2 *Undercutting Tests Involving a Representation of a Generic Roughened Surface Portland Cement Surface*

A laboratory-prepared specimen with a broomed surface is recommended. With such a surface, undercut areas must be measured with a planimeter or computerized stylus.

5.7.1.3 *Undercutting With Highway Core Specimens*

The objective of such tests is determination of undercutting to be expected with specimens which retain the physical and chemical surface conditions of in-service highway substrates.

Tests with this objective should utilize core specimens which have been brushed and vacuumed to remove surface dusts. The undercutting test procedure with such specimens is the same as that for mortar specimens, but with undercut areas measured with a planimeter or computerized stylus. The specimen should be presoaked with deionized water prior to preparing the ice specimen, but not flushed with water to remove soluble chemical contaminants.

5.7.1.4 *Research-Oriented Tests With Core Specimens*

The tests conducted as indicated in Sections 10.1.1, 10.1.2, and 10.1.3 will provide measures of undercutting expected with generic virgin portland cement concrete and with one or more representative field condition core specimens. Undercutting tests conducted in SHRP H-203 indicate that with small specimens subjected to rigorous pretreatment procedures designed to eliminate surface chemical contamination, a substantial difference exists between smoothed/clean and as-received/clean substrates. The physical characteristics of surfaces (which in some cases vary markedly from the smoothed condition to the as-received condition) have been shown to affect the extent of undercutting; rough surfaces were undercut less extensively than smooth surfaces. The possible effects of chemical contamination have not been addressed.

Core specimens present the opportunity to determine the effects of chemical contamination. Thus, core samples may be subjected to thorough water washes to remove soluble contaminants, and to solvent-based treatments to remove water-insoluble organic contaminants to determine whether one or both types of contaminants affect ice undercutting.

The undercutting tests suggested in Sections 10.1.1, 10.1.2, and 10.1.3 will provide data for representing virgin or laboratory portland cement concrete and for field condition specimens. From a research standpoint, of interest is whether or not environmental contaminants (including residual deicing chemicals) significantly affect undercutting. Evidence of effects may, in principle, be obtained by "cleaning" core specimens utilizing techniques developed in the SHRP H-203 program, or by systematically contaminating laboratory specimens with materials of interest.

5.7.2 Testing with Asphaltic Substrates

5.7.2.1 *Types of Asphaltic Substrates*

Asphaltic substrates range potentially from asphalt per se, through laboratory-prepared and field core aggregate-containing asphalts. As with portland cement concrete substrates, it is appropriate to test laboratory specimens to develop baseline undercutting results for asphaltic substrates; to extend tests to field cores in the as-received condition to determine undercutting expected with field representative materials; and optionally, to consider research-oriented tests designed to elucidate possible effects of field conditions.

5.7.2.2 *Test Procedure With Asphaltic Substrates*

The dark to black asphaltic substrates cannot be tested with the indicating dye (bromcresol green) specified for light portland cement-based substrates. For the dark substrate, a dye (disodium salt of fluorescein) which fluoresces a brilliant yellow color under ultraviolet radiation must be employed. With this dye, placement of dye droplets on the ice surface is not satisfactory. Fluorescence in surface melts tends to obscure fluorescence in the undercut areas.

The technique utilized is as follows:

- A warmed rod, 5/32 in (0.4 cm) in diameter, is utilized to melt a small cavity in the ice, down to the substrate. Melted water is removed from the cavity with a syringe.
- An aqueous solution of fluorescein dye (150 mg dye in 5 mL H_2O) is prepared and 5 µL deposited in the bottom of the cavity and permitted to freeze.
- Forty microliters of deionized water is deposited over the dye and allowed to freeze.
- Solid deicer particles are placed directly over the dye spot in the small crater formed by melted water refrozen around the cavity.
- Photographs are taken of the undercut patterns, which are rendered visible by exposure to UV radiation. A UV lamp capable of generating a strong signal is required (example: Spectroline Model B-100 black light lamp, long wave ultraviolet [365 nm], 115 V, 60 Hz, 3.3 A). Note: The UV lamp requires a several-minute warmup and cannot be tolerated in a test enclosure. In tests conducted in a cold room, the lamp can be inserted momentarily into the test enclosure to fluoresce the undercut patterns. With a regulated temperature enclosure placed in an upright freezer, photos can be taken by withdrawing the test specimen into the laboratory, positioning it briefly under the camera and lamp, taking a picture, and returning the specimen to the enclosure. The operation can be completed in 20 sec or less. The number of photos, including the 60-min photo, should be limited to three or four, and the time intervals selected to best represent the rate of propagation of the undercut pattern.

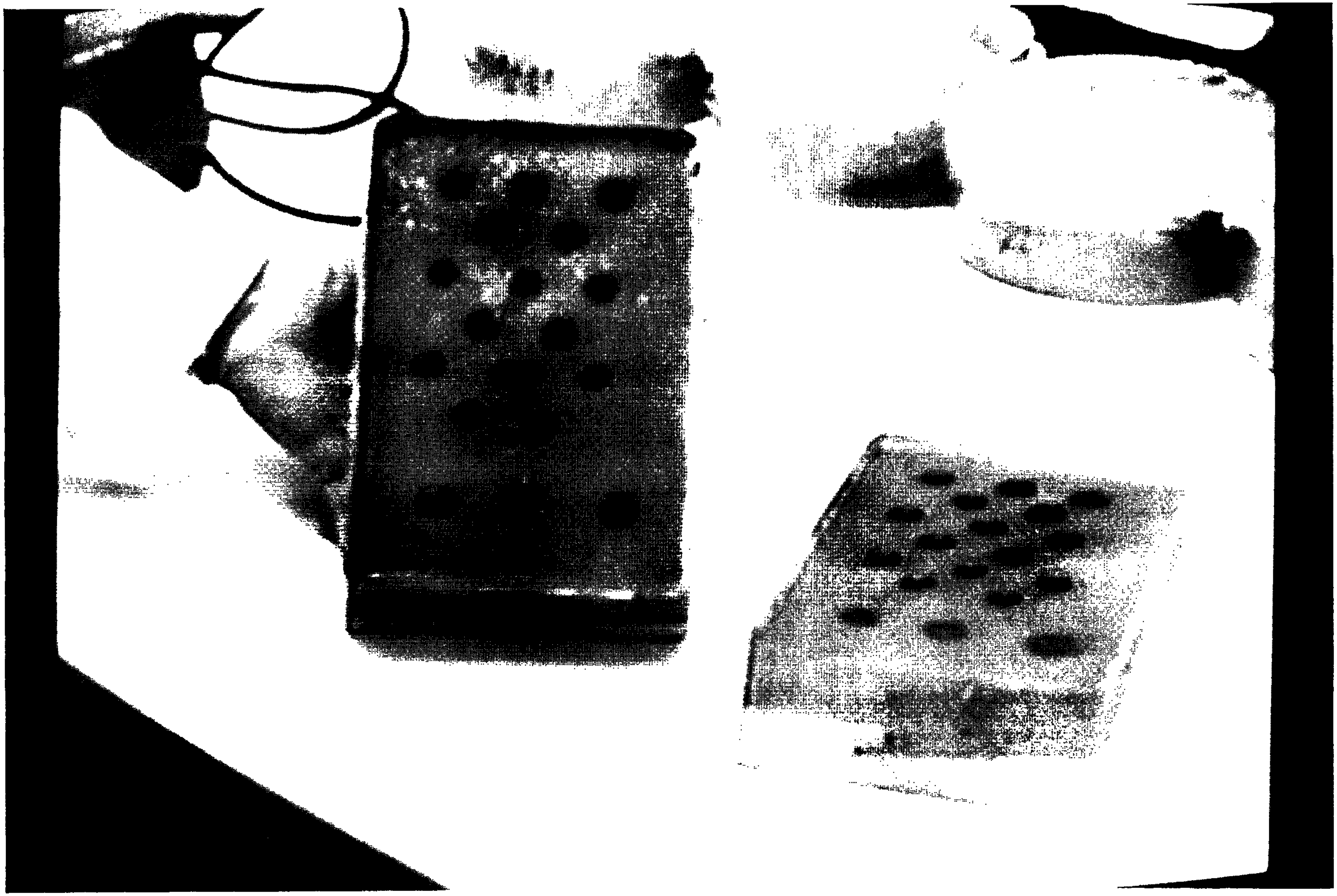


Figure B-4. Undercutting test specimen.

5.8 Undercutting Data Calculations and Interpretation

5.8.1 Data Collection

The required set of raw data consists of measured undercut areas associated with known deicer particle weights, at a given temperature and at a succession of time intervals.

The recommended procedure for obtaining undercut areas consists of projecting photographic slides with the image reversed onto a glass screen covered with onionskin paper, so that the image can be viewed looking toward the projector lens. The dimensions of the patterns are either measured directly or the patterns traced on the paper so that areas can subsequently be measured with a planimeter or a computerized stylus. (The image should be adjusted in size so that it is true size, that is, 1 cm = 1 cm, or a multiple of true size.)

With the recommended mortar test specimens, a majority of the undercut patterns are close to being perfect circles. Direct measurement of diameters is accordingly acceptable; 2 to 3 diameter numbers may be recorded and averaged for the less perfect circles.

5.8.2 Calculations

Areas or diameter measurements are converted to area undercut per unit weight deicer (cm^2 undercut/g deicer) by the following procedures:

1. For measured diameters of undercut patterns

    d = diameter, in millimeters

    m = pellet weight, in milligrams

    A = undercut area, cm^2/g deicer $= \frac{d^2}{m} \times 7.854$

2. For measured undercut areas

    a = undercut area, cm^2

    m = deicer weight, milligrams

    A = undercut area per gram deicer, in cm^2/g

$$A = \frac{a}{m} \bullet 1{,}000 \text{ cm}^2/\text{g}$$

3. Standard error calculation

Formula

$$\text{S.E.} = \left(\frac{\Sigma(\overline{m}_n - d_n^2)}{N \bullet (n-1)} \right)^{1/2}$$

$\overline{m}_n$ = average of n replicates

d_n = value for replicate n

n = number of replicates

Example

$d_1 = 75$

$d_2 = 80$

$d_3 = 85$

$d_4 = 82$

$d_5 = 77$

$$\overline{m}_n = \frac{75 + 80 + 85 + 82 + 77}{5} = 79.8$$

$\overline{n}_n = D_{1\text{-}5} = 4.8, -0.2, -5.2, -2.2, 2.8$

$$\text{S.E.} = \left(\frac{4.8^2 + (-0.2)^2 + (-5.2)^2 + (-2.2)^2 + 2.8^2}{5 \bullet 4} \right)^{1/2} = 1.772$$

5.8.3 Data Presentation

Tabular representations of results consist of tables containing calculated undercut areas per unit weight of deicer (cm^2/g), for each replicate at each temperature and at each time interval, the averages of five replicates, and standard errors. The results may also be displayed graphically, with smoothed curves drawn through the average values.

5.8.4 Interpretive Problems or Uncertainties

The following processes occur in the undercutting tests. Initial melting occurs at the surface and yields a surface film of dye-colored brine. The first photographs (e.g., at 5 min) exhibit the surface brines, which must not be interpreted as undercutting. With most deicers, the color of the surface brine fades within a few minutes, and an intensely colored small cavity of melted brine develops as the deicer penetrates through the ice to the substrate/ice interface. The undercutting process is judged to be occurring when the ice/substrate interface is reached. Normally, the succeeding photographs will clearly indicate the approximate time at which undercutting commences, that is, the diameter of the dye patterns is clearly becoming substantially greater than the diameter of the penetration cavity.

In graphical presentation of results, the adopted practice consists of utilizing penetration data to define the approximate time required to penetrate through 1/8 in. (3.175 mm) of ice and constructing the smoothed curves so that no undercutting occurs prior to that time.

For deicers that do not penetrate well but tend to melt surface ice, the surface brine is likely to be intensely colored, and photographic results may be mistakenly assumed to represent undercutting. In such cases, penetration data are essential for interpretation of results. This problem occurs with CMA at all temperatures and with deicers such as urea and potassium chloride when test temperatures approach eutectic temperatures.

5.8.5 Data Reproducibility

Undercut areas per unit weight at 60-min range from about 80 to 100 cm^2/g at 25°F (-4°C) to 10 to 12 cm^2/g for sodium chloride at 5°F (-15°C). Standard errors, on a percentage basis relative to the average of five replicates, are generally of the order of 3 to 5 percent with the larger undercut areas. Standard errors generally increase, on a percentage basis, as the undercut area per unit weight decreases as temperatures are lowered or as elapsed time decreases. For undercut areas of the order of 10 to 15 cm²/g, standard errors of the order of 10 to 20 percent may be expected.

5.9 Example Ice Cutting Test Results

Ice undercutting test results with pellet calcium chloride (90 percent $CaCl_2$) and sodium chloride (Morton-Thiokol Safe-T-Salt) at 25°F (-3.89°C) and 5°F (-15°C) are presented in Tables B-11 and B-12.

Table B-11. Example ice undercutting test results, cm²/g deicer

	$CaCl_2$ pellets (90% $CaCl_2$)			
	25°F		5°F	
Time (min)	Average of 5 replicates (cm²/g)	Standard error (%)	Average of 5 replicates (cm²/g)	Standard error (%)
5	16.8	2.0	–	–
10	46.5	4.4	7.6	–
15	61.0	5.0	11.6	11.0
20	66.2	4.0	15.6	3.8
30	71.7	4.1	25.4	4.8
45	76.9	3.1	28.1	5.6
60	77.3	3.0	28.7	5.5

Table B-12. Example ice undercutting test results, cm^2/g deicer

	Sodium chloride (rock salt)			
	25°F		5°F	
Time (min)	Average of 5 replicates (cm^2/g)	Standard error (%)	Average of 5 replicates (cm^2/g)	Standard error (%)
5	8.1	–	0	–
10	24.6	15.0	0	–
15	46.0	6.1	0	–
20	58.8	4.7	0	–
30	79.5	3.5	5.7	14
45	84.9	1.6	7.5	7.7
60	86.5	2.5	10.7	8.7

SHRP H-205.6

Test Method for Ice Undercutting By Liquid Deicing Chemicals

6.1 Scope

This test covers the determination of the extent of ice undercutting by liquid chemical deicers at the interface between a substrate and ice frozen on the substrate.

6.2 Applicable Documents

- SHRP H-205.1 Test Method for Ice Melting By Solid Deicers
- SHRP H-205.5 Test Method for Ice Undercutting By Solid Deicing Chemicals

6.3 Summary of Method

Liquid deicers must be physically contained or localized at an undercutting test site to prevent the formation of thin films of the deicer over an ice surface. Additionally, liquid deicers have little tendency for directed or downward penetration through ice, and thus will possess little capability to undercut if they are not placed either on a thin film of ice or in a cavity in ice which extends to a bare substrate.

The undercutting test method for liquids accordingly utilizes a small cylindrical cavity in ice formed on a substrate, with the bottom of the cavity consisting of substrate essentially free of ice. Weighed quantities of liquid deicers are placed in the cavities, and undercutting commences immediately. Undercutting is evidenced by the formation of essentially circular undercut patterns. The dimensions of the observed and measured undercut patterns reflect the net result of melting on the walls of the ice cavity and melting at the ice/substrate interface via a film which is relatively thick near the center and relatively thin at the extremities of the circular undercut areas. The area undercut is defined as the total area of the circular undercut pattern minus the area of the original cavity.

6.4 Significance and Use

- This test method can be used to evaluate and compare the rate and extent of ice undercutting caused by liquid chemical deicers applied to a standard ice/mortar substrate interface at various temperatures.

- This test method includes adaptations for evaluation of ice undercutting caused by liquid chemical deicers on ice bonded to other substrates such as concrete or asphalt.

6.5 Equipment, Materials Preparation, and Procedures

Equipment and procedures utilized for the undercutting test parallels or is identical to that employed for the ice undercutting test for solid deicers, as specified in SHRP H-205.5, with differences as noted below.

6.5.1 Substrate

The substrate consists of a mortar specimen with a lightly textured surface, as specified in H-205.5.

6.5.2 Ice Specimen

A 1/8-in. (3.2-mm)-thick layer of ice is frozen on the mortar substrate, via the bottom-up freezing procedure specified in H-205.5.

6.5.3 Cavity Fabrication in Ice

Cavities in the ice (1/8-in. [3.2-mm] deep [down to the mortar surface]) are fabricated using a warm aluminum rod and a syringe, as follows.

A rod is fabricated by machining 0.5 cm of one end of a 1/4-in. aluminum road to a nominal diameter of 5/32 in. (The measured diameter of the rod used in tests was 0.152 in. (0.386 cm).

Approximately 100 mL water in a 250-mL beaker is warmed to 149° to 158°F (65° to 70°C). The aluminum rod is placed in the water and allowed to warm.

The needle is removed from a 5-mL plastic syringe so that the plastic tip with an outside diameter of 4.064 mm (0.160 in.) is available to extract melted ice from cylindrical cavities formed with the warmed aluminum rod.

The warm rod is taken out of the water, wiped with a tissue, placed vertically on the ice surface, and pressed downward with moderate pressure for 3 to 4 sec.

The rod is removed, the syringe tip placed in the cavity, and melted water drawn into the syringe. The syringe is held in position 2 to 3 sec, then withdrawn and water ejected. If visual inspection indicates a film of ice on the bottom of the cavity, the process with rod and syringe can be repeated, taking care not to enlarge the diameter of the cavity.

The cavity-forming process preferably is conducted in the cold (e.g., at test temperatures) so that melted ice forced onto the surface of the ice is refrozen and does flow back into the cavity or into adjacent cavities.

Cavities are fabricated in sets of five. The mortar specimen will accommodate three sets of five cavities, with cavities about 4 cm (1.5 in.) apart.

6.5.4 Temperature Equilibration

The mortar/ice specimen containing cavities is equilibrated overnight to the test temperature.

6.5.5 Deicer Handling Procedures

6.5.5.1 *Dye Addition*

Small droplets of neat Bulls-Eye™ dye are placed in a dry, empty, weighed vial via a glass stirring rod until a dye weight of 15 to 20 mg has been transferred.

Five milliliters of liquid deicer is then added to the vial, which is then capped. The vial is weighed in the ambient laboratory environment and cooled either to test temperature or to the lowest temperature at which the deicer remains as a liquid or does not precipitate dissolved solids.

6.5.5.2 *Deicer Addition to Cavities*

With a pipetting system (e.g., an Eppendorf Model 4710, 10- to 100-µL delivery range, yellow plastic, disposable pipette tips) designed for delivery of small quantities of liquid, a 30-µL quantity of deicer is placed in each of five cavities.

The deicer vial is returned to ambient laboratory and reweighed after 1 hr or more to determine the weight of the five test samples.

6.5.5.3 *Slide Photographs of the Test Specimen*

Photographs are taken of the following time intervals: 5, 10, 15, 20, 30, 45, and 60 min.

The developed slides are projected on onion skin paper, with the image reversed and adjusted to actual size, and diameters measured with a millimeter scale ruler.

6.5.6 Regulated Temperature Test Enclosures

Ice undercutting test results vary by as much as 10 percent per degree Fahrenheit. An enclosure which can be regulated to within ±0.5°F (±0.28°C) of the designated test temperature is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The enclosure should have a temperature capability ranging from 25°F to at least 5°F (-4°C to at least -15°C).

Options for a controlled temperature include: (1) a box equipped with hand ports and a window, and placed inside a walk-in cold room, and (2) a similar enclosure placed in an upright freezer, and equipped with hand ports and viewing windows fabricated and installed in the freezer door.

6.5.6.1 *Cold Room Test Enclosure*

A cold room test enclosure, which has been extensively utilized, is described in Annex 1 of Test Method SHRP H-205.1.

6.5.6.2 *Upright Freezer Test Enclosure*

A similar test enclosure designed for use in an upright freezer is presented in Annex 2 of Test Method SHRP H-205.1.

6.6 Optional Procedures Using Other Test Substrates

A general rationale and procedures for conducting undercutting tests with solid deicers, with substrates other than the portland cement mortar substrate, have been presented under H-205.5.

The rationale for selection of additional substrates presented in H-205.5 remains the same for tests involving liquid deicers. Test procedures for the additional substrates are presented in Sections 1.6.1 and 1.6.2.

6.6.1 Portland Cement Concrete (PCC) Substrates

The experimental procedures utilized for PCC substrates are identical to those specified for the portland cement mortar specimen.

6.6.2 Asphaltic Substrates

The fluorescent dye (disodium salt of fluorescein) must be utilized. Incorporation of the dye in a stockpile inventory of liquid deicer has yielded poor and erratic results. Consequently, the procedure utilized is as follows:

- Prepare a dye solution consisting of 150 mg fluorescein in 5 mL H_2O.
- Prepare a cavity in ice using the warmed rod and syringe technique.
- Place 5 μL of the dye solution in the cavity; allow it to freeze.
- Add 30 μL of liquid deicer to the cavity and proceed as indicated for tests with liquid deicers containing Bulls-Eye™ dye, but with photographs taken with dye spots fluoresced with an ultraviolet lamp.

6.7 Data Treatment and Calculations

6.7.1 Data Treatment

One array of five tests yields five sets of undercut areas for a deicer which pertain to the average deicer weight rather than to individually measured test weights. The five sets of areas may be treated as a five-replicated test and analyzed for reproducibility (error analysis) based on total undercut areas minus the area of the cavity at test start. Random error analyses of undercut areas in a five-test array indicate standard errors ranging from about 1 to 6 percent of the average of five areas.

In method development tests, three sets of five were conducted, and error analyses were conducted for a triply-replicated test.

Both approaches are deemed acceptable.

6.7.2 Calculations

The following data are provided by the tests:

- Undercut pattern diameters in millimeters for a specified deicer at given temperatures and elapsed times.
- The weight of deicer for five tests in grams and the average weight for individual tests.
- The diameter of the cavity at test start, taken to be 4.2 mm.

6.7.2.1 *Undercut Area Calculation*

$$A = \text{(total area - cavity area) in } cm_2$$

$$= \frac{\pi d^2 \bullet 10^{-2}}{4} - \frac{\pi \bullet (4.2)^2 \bullet 10^{-2}}{4}$$

$$= 0.007854 \bullet d^2 - 0.13854$$

where d = diameter in millimeters

Example:

d = 20 mm

$A = [0.007854 \bullet (20^2 - 0.13854)] \text{ in } cm^2$

$= 3.00306\ cm^2$

6.7.2.2 *Calculation of Undercut Area Per Gram of Deicer*

Undercut area (UA) expressed as cm^2/g deicer

$$\text{UA in } cm^2/g = \frac{A,\ cm^2}{\text{deicer weight}}$$

Example: $A\ cm^2 = 3.00306\ cm^2$

Average deicer weight = 35 mg = 0.035 g

$$\text{UA in cm}^2\text{/g} = \frac{3.00306}{0.035 \text{ g}} = 85.8 \text{ cm}^2\text{/g deicer}$$

6.7.2.3 *Secondary Calculations*

With deicers consisting of aqueous solutions, conversion to undercut areas per unit weight of the deicing chemical (e.g., dissolved calcium chloride or sodium chloride) may be appropriate. Such conversions are accomplished by dividing undercut areas per gram of deicer by the weight of deicing chemical in 1 g of deicer.

Example:

Deicer: 26.3 percent NaCl by weight

UA, cm^2/g 26.3 percent NaCl = 26.3 cm^2/g

$$\text{UA, cm}^2\text{/g NaCl} = \frac{26.3}{0.263} = 100 \text{ cm}^2\text{/g NaCl}$$

Example results reported in Tables 1, 2, and 3 have been expressed as undercut areas per gram of ethylene glycol, and as undercut areas per gram of $CaCl_2$ (dry weight) and NaCl (dry weight) contained in the aqueous test solutions.

6.8 Example Undercutting Test Results

Example undercutting results are presented in Tables B-13, B-14, and B-15 for ethylene glycol, 37.3 percent for calcium chloride solutions, and 26.3 percent sodium chloride solutions. Set Nos. 1, 2, and 3 in the tables are five test sets. Average deicer weights (i.e., five sample weight ÷ 5) fell in the following ranges:

Ethylene glycol: 31 to 34 mg

37.3 percent aqueous $CaCl_2$: 41 to 43 mg

26.3 percent aqueous NaCl: 35 to 38 mg

Table B-13. Liquid deicer undercutting results with ethylene glycol

Time (min)	cm^2/g ethylene glycol Set No. 1	Set No. 2	Set No. 3	Average	Standard error (%)
			25°F		
5	22.7	24.9	20.2	22.6	7.4
10	39.6	40.7	34.7	38.3	4.8
15	48.3	51.4	43.2	47.6	5.0
20	54.2	55.6	48.0	52.6	4.4
30	58.0	61.1	51.9	57.0	4.7
45	60.2	63.3	56.4	60.0	3.3
60	60.5	64.1	57.3	60.6	3.2
			20°F		
5	17.8	17.0		17.4	2.3
10	29.8	28.1		28.9	2.9
15	36.3	34.5		35.9	4.0
20	40.0	38.3		39.2	2.1
30	45.6	-		45.6	-
45	49.5	44.7		47.1	5.1
60	49.6	44.7		47.2	5.2
			15°F		
5	6.8	8.0	5.3	6.7	1.2
10	16.9	15.7	14.6	15.7	4.2
15	22.4	20.5	21.9	21.6	2.6
20	26.8	23.7	24.0	24.8	3.9
30	29.5	27.1	26.8	27.8	3.0
45	31.5	28.6	29.6	29.9	2.8
60	32.0	30.5	29.7	30.7	2.2
			5°F		
5	3.6	2.1		2.9	2.6
10	5.4	3.3		4.4	2.4
15	8.0	7.4		7.7	5.2
20	9.8	8.5		9.2	7.1
30	12.6	12.4		12.5	0.8
45	14.6	14.9		14.8	1.0
60	15.2	15.3		15.3	0.3

Table B-14. Liquid deicer undercutting results with 3.75 percent $CaCl_2$ solution

Time (min)	cm²/g $CaCl_2$ Set No. 1	Set No. 2	Set No. 3	Average	Standard error (%)
		25°F			
5	–	66.5	64.7	65.5	1.4
10	89.4	91.8	84.2	88.5	2.5
15	94.0	96.8	90.9	93.9	1.8
20	97.8	96.8	91.4	95.3	2.1
30	101.7	98.3	93.4	97.8	2.5
45	102.9	98.3	93.6	98.3	2.7
60	103.8	102.2	95.36	100.4	2.6
		20°F			
5	50.6	52.2		51.4	1.5
10	63.1	63.9		63.5	0.6
15	65.2	74.7		64.9	0.4
20	69.6	67.4		768.5	1.6
30	70.3	–		70.3	–
45	71.1	70.4		70.7	0.5
60	71.6	72.1		71.9	0.4
		15°F			
5	37.4	35.3	33.4	35.4	3.3
10	46.4	44.8	43.8	45.0	1.7
15	52.2	49.3	46.7	49.4	3.2
20	53.3	50.4	48.0	50.6	3.0
30	53.7	51.1	50.1	51.6	2.1
45	53.9	52.0	50.1	52.0	2.1
60	54.6	52.0	50.1	52.2	2.5
		5°F			
5	22.6	11.0		16.8	3.5
10	31.1	18.2		24.7	2.5
15	31.9	21.7		26.8	1.9
20	32.2	22.7		27.5	1.7
30	32.9	24.0		28.5	1.6
45	33.0	25.4		29.2	1.3
60	33.9	25.8		29.5	1.2

Table B-15. Liquid deicer undercutting results with 26.3 percent NaCl solution

	cm^2/g NaCl				
Time (min)	Set No. 1	Set No. 2	Set No. 3	Average	Standard error (%)
		25°F			
5	73.2	72.9	66.6	70.9	3.0
10	89.0	92.3	87.5	89.6	1.6
15	99.3	104.6	94.0	99.3	3.1
20	101.0	107.8	97.0	101.9	3.1
30	103.3	108.2	98.6	103.4	2.7
45	104.0	108.2	100.1	104.1	2.3
60	104.0	108.2	100.9	104.4	2.0
		20°F			
5	39.6	43.4		41.5	4.6
10	55.7	57.0	64.5	56.4	1.1
15	59.2	61.6	64.5	60.4	2.0
20	60.7	-	64.5	60.7	-
30	64.4	64.5	64.5	64.5	0.1
45	68.5	64.5	64.5	66.5	3.0
60	69.4	64.5	64.5	67.0	3.7
		15°F			
5	23.7	19.6	20.9	21.4	5.8
10	28.0	29.0	27.6	28.2	1.5
15	32.9	31.6	29.6	31.4	3.1
20	34.2	32.8	31.0	32.7	2.8
30	34.9	33.0	34.8	34.2	1.8
45	37.3	35.6	35.2	36.0	1.8
60	37.6	35.6	35.2	36.1	2.1
		5°F			
5	3.7	4.9		4.3	1.4
10	5.2	6.0	7.1	5.6	7.1
15	6.0	6.3	7.1	6.2	2.4
20	6.5	6.8	7.1	6.7	2.2
30	6.8	7.1	7.1	7.0	2.1
45	6.8	7.1	7.1	7.0	2.1
60	6.8	7.1	7.1	7.0	2.1

SHRP H-205.7

Test Method for Evaluation of Corrosive Effects of Deicing Chemicals on Metals

7.1 Scope

This test method covers evaluation of the corrosive effects of deicing chemicals on metal substrates. This test method is also intended to evaluate the effectiveness of corrosion inhibiting additives to deicing chemicals.

7.2 Applicable Documents

7.2.1 ASTM Standards

- G1-91 Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
- G31-72 Standard Practice for Laboratory Immersion Corrosion Testing of Metals
- G46-76 Standard Practice for Examination and Evaluation of Pitting Corrosion
- C876-91 Standard Test Method for Half-Cell Potentials of Uncoated Reinforcing Steel in Concrete
- E70-90 Standard Test Method for pH of Aqueous Solutions With the Glass Electrode

7.3 Significance and Use

- This test method can be used to evaluate the corrosive effects of deicing chemicals on metal substrates.
- This test method can be used to evaluate the effectiveness of corrosion inhibiting additives to deicing chemicals.
- This test method can be used to evaluate the corrosive effects of other aqueous, near neutral pH solutions.

7.4 Apparatus and Materials

7.4.1 Apparatus

Nylon toothbrush, adult.

Fishing line: Nylon.

Sample ID tags.

Fiberglass brushes: Abrasive pencil (see Note 1).

NOTE 1: Suitable pencils and brushes are available from Eraser Co., Syracuse, NY 13221. Super Fybrglass Eraser Model AB0001 pencil and brush refills AB0009, Vigor No. 703, local.

Electrodes:

1. For mV measurements: Voltmeter and saturated calomel electrode (SCE).
2. For pH measurements: pH meter and pH electrode.

Electrically insulating liquid tape.

Aeration system:

Air system filter capable of removing 99% oil plus water.

Air system pressure regulator.

Electrode attachment wire: 22-gauge, tin coated, single strand, insulated.

Test cell beakers: 1,000-mL disposable polyethylene plastic (~ 11.5-cm inside diameter top, 10-cm inside diameter bottom, 14.5-cm height).

Plexiglas® spacers: 1-cm length rings cut from Plexiglas® pipe, 4 in. O.D. x 1/4 in. W.T.

Test cell lids: Polystyrene (1-cm hole in center for air inlet).

Metal test specimens: ~ 1/8 in. x 1 in. x 2 in. Other test specimen geometries may be used in the test, although comparisons of deicers should be made using test specimens of identical metal and geometry. Also, use of test specimens with different geometries may require other modifications in such specifications as test all size and design, test specimen spacing, test solution volume, test specimen cleaning procedures, etc. (see Note 2).

NOTE 2: Suitable test coupons are available from Metal Samples, P.O. Box 8, Munford, Alabama 36268.

Analytical balance.

Ultrasonic bath.

Steam bath.

Brass screws, nuts as appropriate.

7.4.2 Materials

Deionized water.

Acetone, reagent grade.

Sodium chloride, NaCl, reagent grade.

Other chemical deicers as required.

Candidate corrosion inhibitors as required.

7.5 Test Preparation

7.5.1 Preparation of Metal Test Specimens

Clean (see Note 3) and weigh to the nearest 0.1 mg the corrosion test specimens.

NOTE 3: Rinse twice with reagent grade acetone and air dry.

Attach a nylon string with ID tag to each test specimen.

Label the tag with test specimen number and test cell position number and assign to a test cell.

Store the test specimens in a sealed plastic bag in a desiccator until placed in the test cell.

7.5.2 Preparation of "Electrode" Test Specimens

Attach a 20-cm insulated wire (22-gauge, single-strand, tin-coated) with a brass screw and nut.

Cover the connection area of the attached wire with liquid tape.

Store the electrode test specimens in a desiccator until placed in the test cell.

7.5.3 Preparation of Test Cells (See Figure B-5, page 197)

Notch the lip of each plastic test cell for the number of test specimens plus 1 electrode test specimen, evenly spaced around the circumference of the plastic test cell. The maximum number of 1/8-in. x 1-in. x 2-in. test specimens (including the electrode test specimen) that can be accommodated by this test cell configuration is 9.

Fit a Plexiglas® spacer inside the plastic test cell.

Rinse the test cells with ethanol and allow to dry in laboratory air.

Attach the electrode test specimen to the test cell.

Hang the test specimens in the test cells so that the top of each test specimen is just below the bottom of the Plexiglas® spacer ring. Secure the nylon string on the test specimen with tape to the outside of the test cell.

7.5.4 Preparation of Test Solutions

All solutions should be prepared in deionized water.

Prepare a master test solution containing a selected concentration (weight percent) of chemical deicer dissolved in deionized water. For example a 3 weight percent aqueous solution of sodium chloride would be made by dissolving 30.93 g of reagent grade NaCl in 1,000-g deionized water:

$$\frac{\text{grams NaCl}}{\text{grams solution}} = \frac{30.93}{1{,}000 + 30.93} = 3.0 \text{ weight percent}$$

Prepare inhibited solutions by adding selected amounts of corrosion inhibitor(s) and/or other additives to prepared master solution. Inhibitors are usually added as a weight percent of the total solids (deicer plus inhibitor) in the test solution:

$$\frac{\text{grams of inhibitor}}{\text{grams of deicer plus grams of inhibitor}} = \text{inhibitor concentration, weight percent of total solids}$$

Control solutions should include, as a minimum, deionized water, sodium chloride at the same test concentration chosen for the deicers, and master solution without any corrosion inhibitor or other additives.

Determine the pH of all freshly prepared master solutions, master solutions containing corrosion inhibitor(s), and control solutions.

It is important to specify exactly how test solutions are prepared and to note any deicer or inhibitor composition analysis data used to determine actual weights of materials used.

7.6 Test Procedures

7.6.1 Start-Up Procedures

Prepare test solutions according to the test schedule (Note 4, Note 5).

Place test specimens in the test cells, and set the test cells in position.

Fill test cells to the 750-mL mark with appropriate test solution.

Cover test cells with a lid through which the aerator tip is attached.

Turn on air supply and regulate the bubble rate.

NOTE 4: Appropriate test matrices:
Exposure time: 1, 2, 4 and 6 weeks or
1, 3, and 6 weeks or
1, 2, 4, and 8 weeks
Inhibitor concentration: Optimum
Lower than optimum
Higher than optimum
Replication: Duplicate test cells

Different types of exposure methods may be employed to simulate different types of structures and field conditions, but comparison of the relative effectiveness of deicer formulations should be made using identical exposure conditions.

NOTE 5: It is advised that duplicate test specimens, in each test cell, be exposed for the longest exposure time (i.e., 6 or 8 weeks).

7.6.2 Removing Corroded Test Specimens

After exposing the test specimens for the required time, remove lids and air source from all test cells to be tested.

Measure pH of test solutions to correspond to the test specimen exposure times (see Note 4.). The electrical potential of the electrode specimens in the test solutions is measured on the same schedule.

Remove all test specimens for the day and suspend in beakers of deionized water. Reattach lids and air supply to the test cells.

Clean the test specimens according to ASTM G1-90. (A cleaning procedure developed from ASTM G1-90 specifically for cold rolled steel and structural aluminum specimens exposed to various deicer solutions is detailed in the Annex.)

Clean unexposed test specimens (cleaning controls) along with corroded test specimens.

Weigh the clean and dry test specimens to the nearest 0.1 mg.

Visually inspect each test specimen for pitting.

Store the test specimens in plastic bags in a desiccator.

Adjust the test cell solution levels to the 750-mL mark with deionized water, and regulate the air bubble rate in the range of 90 to 100 cc air/min.

7.6.3 Test Specimens Weighing Procedure

Handle test specimens with Teflon forceps.

Weigh test specimens in random order to avoid bias.

Rezero balance before each weighing.

Store steel and aluminum test specimens in separate bags in desiccators after weighing.

Weigh test specimens to 0.1 mg.

7.7 Calculations

Evaluate all test specimens according to ASTM G1-90, "Evaluating Corrosion Test Specimens." A major point of ASTM G1-90 is cleaning an unexposed test specimen (cleaning control) along with and identically to the corroded test specimens. The cleaning control should adjust for weight changes due only to the cleaning procedure for each material.

7.7.1 Weight Lost (mg)

$$WL\ (\text{mg}) = (I - F)\ 1{,}000\ \text{mg/g}$$

where: WL = test specimen weight lost (mg)

I = test specimen initial weight (g)

f = test specimen final weight (g)

7.7.2 Corrected Weight Lost (mg)

$$CWL = WL - CC$$

where: CWL (mg) = corrected test specimen weight lost (mg)

WL = test specimen weight lost (mg)

CC = cleaning control weight lost (mg)

7.7.3 Corrosion Rate (mpy)

$$CR = (K \times W)/(A \times T \times D)$$

where:

CR (mpy) = corrosion rate (mils per year)

K = constant = 3.45×10^3 for corrected weight lost in mg

W = corrected weight lost (mg)

A = specimen area (cm^2)

T = time (hr)

D = specimen density (g/cm^3)
C1018 density = 7.86 g/cm^3
A7075 density = 2.81 g/cm^3

7.8 Report

The report should include the following:

- Composition of test specimens.
- Size of test specimens.
- Corrosive solution composition, and exact solution preparation procedure.
- Inhibitor concentration.
- Exposure time and mode.
- Corrosion rate.
- Pitting corrosion.

7.9 Sample Data and Test Matrix

A sample of typical data generated using this test procedure is presented in Table B-16. The CMA and calcium phosphate solutions were prepared as weight percent of total solids in 3 weight percent sodium chloride.

In this study, each test cell contained 4 corrosion test specimens and 1 electrode test specimen. Two test cells (duplicates) were set up for each different control or deicer concentration.

Twelve test cells and a total of 60 test specimens were used. Three additional unexposed test specimens were used as cleaning controls, one at each exposure interval.

One test specimen was removed from each test cell after 1 and 3 weeks of exposure, and two test specimens were removed from each test cell after 6 weeks of exposure.

Each entry in the data table represents an average of two test specimens (one from each duplicate cell) for each deicer or control tested.

Table B-16. Sample of data generated using test Method H-205.7 for the evaluation of corrosive effects of sodium chloride deicer solutions on cold rolled steel (C1018) coupons

Week	Avg. wt. lost (mg)	Avg. cor. rate (mpy)	Avg. pH	Avg. potential (−V)
3 wt % NaCl control				
1	182.1	16.82	6.7	0.708
3	382.2	11.77	7.4	0.671
6	709.9	10.93	7.4	0.671
6	559.1	8.61	7.4	0.671
		Mean corr rate = 12.0		
		S dev corr rate = 3.5		
DI water control				
1	37.4	3.46	6.3	0.293
3	174.7	5.38	7.7	0.229
6	411.6	6.34	7.4	0.260
6	365.4	5.63	7.4	0.260
		Mean corr rate = 5.2		
		S dev corr rate = 1.5		
CMA in 3 wt % NaCl[a]				
1	104.3	9.63	7.2	0.736
3	255.3	7.86	7.3	0.733
6	489.5	7.54	7.3	0.721
6	462.9	7.13	7.3	0.721
		Mean corr rate = 8.0		
		S dev corr rate = 1.1		
$Ca(H_2PO_4)_2$ in 3 wt % NaCl[b]				
1	19.7	1.82	5.3	0.641
3	30.4	0.94	5.5	0.652
6	43.1	0.66	5.7	0.660
6	44.7	0.69	5.7	0.660
		Mean corr rate = 1.0		
		S dev corr rate = 0.5		

Table B-16. (Continued)

Week	Avg. wt. lost (mg)	Avg. cor. rate (mpy)	Avg. pH	Avg. potential (−V)
$Ca(H_2PO_4)_2$ in 3 wt % NaCl[c]				
1	18.2	1.68	5.5	0.646
3	28.8	0.89	5.9	0.672
6	41.4	0.64	6.2	0.669
6	45.2	0.70	6.2	0.669
		Mean corr rate = 1.0		
		S dev corr rate = 0.5		
$Ca(H_2PO_4)_2$ in 3 wt % NaCl[d]				
1	17.5	1.61	6.0	0.670
3	29.3	0.90	6.9	0.644
6	130.9	2.02	7.3	0.726
6	146.5	2.26	7.3	0.726
		Mean corr rate = 1.7		
		S dev corr rate = 0.6		

[a] CMA (3.33 g) + NaCl (29.90 g) + H_2O (966.77 g).

[b] $Ca(H_2PO_4)_2$ (1.09 g) + NaCl (29.97 g) + H_2O (968.94 g).

[c] $Ca(H_2PO_4)_2$ (0.77 g) + NaCl (29.98 g) + H_2O (969.25 g).

[d] $Ca(H_2PO_4)_2$ (0.45 g) + NaCl (29.99 g) + H_2O (969.56 g).

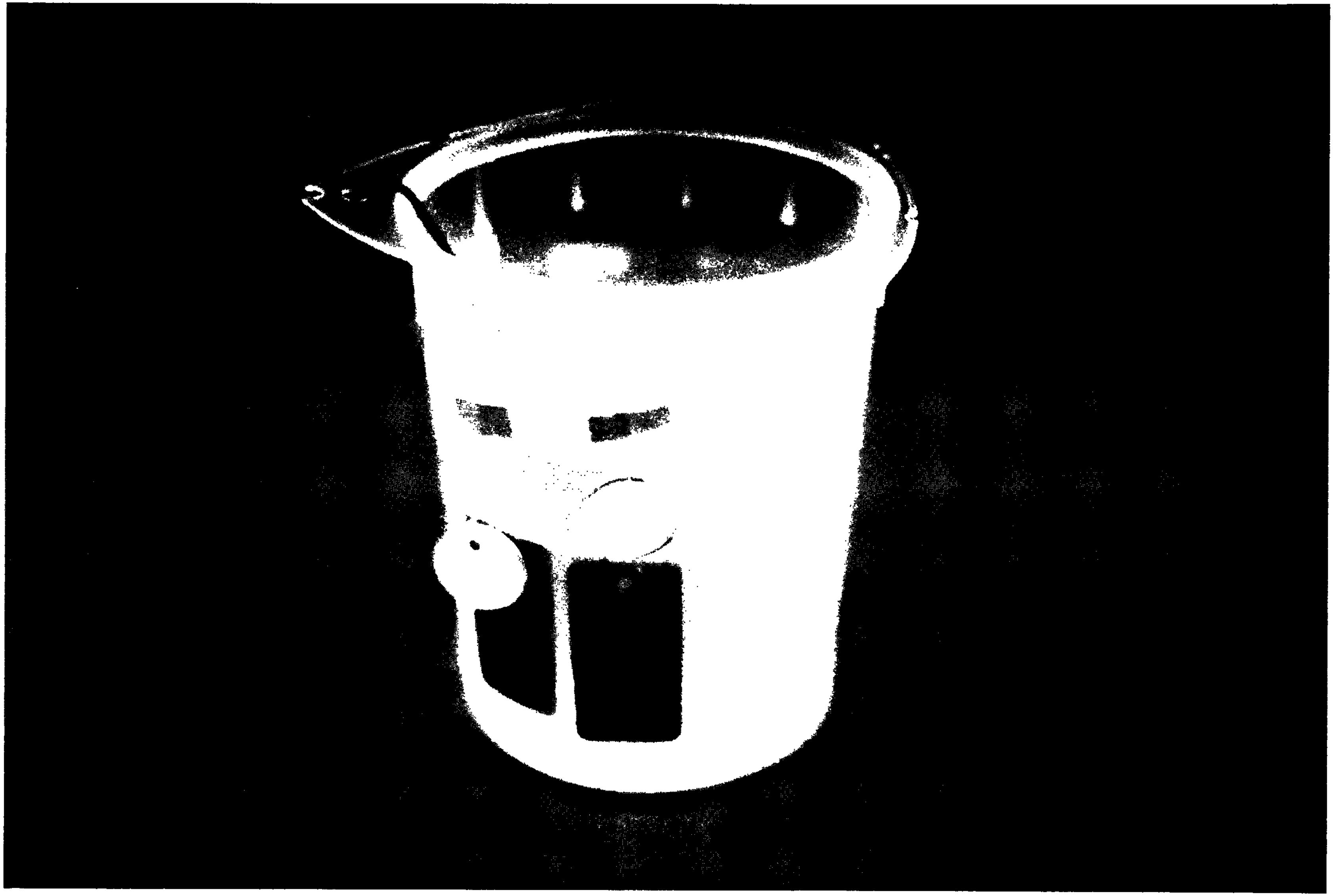


Figure B-5. Corrosion test cell

Annex

Cleaning Procedures for Cold Rolled Steel and Structural Aluminum Specimens Exposed to Deicer Solutions

The following procedure is included as an aid to the development of uniform methodology for cleaning corroded metal corrosion test specimens.

Clean one unexposed steel and one unexposed aluminum test specimen along with the corroded test specimens each day to assess weight loss due to the cleaning process.

1. Nylon brush each corroded test specimen under cold running tap water, using a separate nylon toothbrush for steel and for aluminum.

   - Place test specimens in a plastic holder and scrub-brush 20 to 30 times on each side. Give all test specimens on any given day the same number of strokes.

   - Then, for each test specimen (a) rinse with deionized water from a squeeze bottle; (b) rinse with acetone from a squeeze bottle; and (c) place on a paper towel to dry.

     Note: During brushing/rinsing operations, handle all test specimens by the attached nylon string.

2. Acid bath: Handle test specimens with vinyl gloves; use fresh acid each day.

   - Cut off nylon string and ID tag.

   - String the test specimens onto a Teflon band with Teflon spacers between to prevent contact. Place steel and aluminum specimens on separate bands. Close bands with tape.

   - Immersed the test specimens in the appropriate acid bath.

3. Steel test specimens:

   - Cleaning solution: 50 g stannous chloride + 20 g antimony trioxide + 1,000 mL concentrated hydrochloric acid.
   - 120 mL in a 150-mL beaker.
   - Stir with an ultrasonic bath.
   - 10-sec immersion time at 77°F (25°C).

4. Aluminum test specimens:

   - Cleaning solution: 20 g chromic oxide + 50 mL concentrated phosphoric acid, diluted to 1 L with deionized water.
   - 120 mL in 150-mL beaker.
   - Heat on a steam bath.
   - 2-min immersion time at 194°F to 212°F (90° to 100°C).

5. Rinsing and drying: Handle test specimens with vinyl gloves and Teflon forceps.

   - Deionized water rinse: Immerse the cleaned test specimens twice, clean water each time.
   - Acetone rinse: Dip twice in acetone, clean each time.
   - Hang the test specimens to dry.

6. Fiberglass brushing: Handle test specimens with vinyl gloves and Teflon forceps.

    - Remove test specimens from the Teflon band.
    - Brush 20 to 30 strokes on each side and once around edges.
        - Scrub-brush steel test specimens.
        - Brush aluminum test specimens by stroking in one direction.
        - All test specimens of the same metal on any given day are brushed the same number of strokes.
    - Wipe test specimens with paper towels.
    - Dip test specimens in acetone and put on a paper towel to dry.
    - NOTE: Some variance in the abrasiveness of the fiberglass brush refill may be noted from day to day.

SHRP H-205.8

Test Method for Rapid Evaluation of Effects of Deicing Chemicals on Concrete

8.1 Scope

This test method covers evaluation of the effects of chemical deicing formulations and freeze/thaw cycling on the structural integrity of small (~ 54 cm^3) test specimens of non-air-entrained concrete. The method is intended for use in quantitatively evaluating the degradative effects by measurement of test specimen weight loss. The method is most effective when sodium chloride is run as a control.

8.2 Applicable Documents

8.2.1 ASTM Standards

- C 109-90 Test Method for Compressive Strength of Hydraulic Cement Mortars (using 2-in. or 50-mm cube specimens)
- C 192-90a Practice for Making and Curing Concrete Test Specimens in the Laboratory
- C 672-91 Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals
- C 778-90 Standard Specifications for Standard Sand

8.3 Significance and Use

- This test method can potentially be used to evaluate the effect of various deicer solutions on the structural integrity of concrete during freeze/ thaw cycling.

- This test method is not intended to be used in determining the durability of aggregates or other ingredients of the concrete.

- No relationship has been established between specimens cut from hardened concrete and specimens prepared in the laboratory.

8.4 Apparatus

8.4.1 Freezing Equipment

A chest or upright freezer of sufficient size to hold the specimen containers and capable of lowering the temperature of the specimens from 70°F (21°C) to 0° ± 5°F (-17.8° ± 2.8°C) in less than 18 hr, and maintaining this temperature is required.

8.4.2 Thermometer (Optional)

Digital thermometer with thermocouple for use in monitoring temperature of the test specimens during freeze/thaw cycling. Sensitivity 1.8°F (1°C); range -4° to +77°F (-20° to +25°C), minimum.

8.4.3 Molds and Mold Supports

8.4.3.1 *Test Specimen Molds*

PVC pipe, 1 1/2-in. diameter x 1 7/8-in. length, or equivalent, having a volume of 3.31 in^3 (54 cm^3). To facilitate demolding, one saw cut should be made through each specimen mold along the 1 7/8-in. length axis. Cover the cut with duct tape or equivalent.

8.4.3.2 *Concrete Compressive Strength Control Cube Molds*

Cube molds (2 in.) conforming to the requirements of ASTM C 109-90.

8.4.4 Tamping Rods

8.4.4.1 *For Test Specimens*

Conforming to the requirements of Method C 192-90a.

8.4.4.2 *For Compressive Strength Control Cubes*

Tamper for 2-in. cube consolidation conforming to the requirements of ASTM C 109-90.

8.4.5 Containers

8.4.5.1 *Sampling and Mixing Pan*

The pan should be flat-bottom and of heavy-gauge metal, watertight, of convenient depth, and of sufficient capacity to allow easy mixing by shovel or trowel of the entire batch; or, if mixing is by machine, to receive the entire batch on discharge of the mixer and allow remixing in the pan by trowel or shovel.

8.4.5.2 *Concrete Mixer*

A power-driven revolving drum, tilting mixer, or suitable revolving pan or revolving-paddle mixer capable of thoroughly mixing batches of the prescribed sizes at the required slump.

Note: A pan mixer may be more suitable than a revolving drum mixer for mixing small batches of concrete. The rate of rotation, degree of tilt, and rated capacity of tilting mixers are not always suitable for laboratory-mixed concrete. It may be desirable to reduce the rate of rotation, decrease the angle of tilt from the horizontal, and use the mixer at somewhat less than the manufacturer's rated capacity.

8.4.5.3 *For Freeze/Thaw Test Specimens*

Shallow cylindrical 1 1/2-qt plastic storage dish fitted with a lid and measuring 6 in. inside diameter and ~ 3.0 in. deep (Rubbermaid No. HS 900 or equivalent).

8.4.6 Compressive Strength Testing Machine

For 2-in. control cubes conforming to the requirements of ASTM C 109-90.

8.4.7 Scales

8.4.7.1 *For Weighing Ingredients of Concrete*

Conforming to the requirements of ASTM C 109-90.

8.4.7.2 *For Weighing Freeze/Thaw Test Specimens*

Sensitivity, 0.1 g; range, 0 to 120 g.

8.4.8 Curing Chambers

- A tank for curing and storage of test specimens and control cubes prior to testing. The tank should be made of noncorroding materials and capable of being maintained at a temperature of 73° ± 3°F (23° ± 1.7°C). The tank should be fitted with a shelf above water level to permit curing of specimens near 100 percent relative humidity for extended periods after demolding.

- A chamber for curing and storage of test specimens and control cubes prior to testing. The chamber should be capable of being maintained at a temperature of 73° ± 3°F (23° ± 1.7°C) and 50 percent ± 5 percent relative humidity for extended periods.

8.4.9 Sponges

For use in freeze/thaw test containers. Cellulose sponges, 6 3/8 x 3 5/8 x 1 in., O-Cell-O® or equivalent.

8.4.10 Sieves

For use in preparing aggregate mixture--as specified in ASTM C 192-90a.

8.4.11 Miscellaneous Equipment

Steel trowel, wooden strike-off board, rubber gloves, and graduated cylinders.

8.5 Materials

- Sand should be clean, natural silica sand, saturated surface-dry, and suitable for concrete construction.

- Aggregate should be crushed limestone, 3/8-in. designation. Aggregate should be saturated surface-dry and suitable for concrete construction.

8.5.1 Cement

Portland Type I.

8.6 Proportioning and Mixing of Concrete

8.6.1 Proportioning

The following concrete mix design is specified, following the ASTM C 672-91:

Water (tap)	9.11 weight percent
Cement (Portland Type 1)	17.79 weight percent
Sand (50/50 coarse/fine)	31.13 weight percent
Aggregate (3/8-in. max. limestone, saturated surface dry)	41.97 weight percent
Water/cement ratio	0.51
Slump (ASTM C 143)	2.5 ± 1 in.
Density	$\simeq$ 145 lb/ft^3
Compressive strength	> 4,000 psi

Non-air-entrained.

8.6.2 Mixing of Concrete

- Hand-mixing should meet the requirements in ASTM C 192-90a. Hand-mixing should be followed for batches of 1/4 ft^3 volume or less (approximately 35 lb).

- Machine-mixing should meet the requirements in ASTM C 192-90a. Machine-mixing should be followed for batches exceeding 1/4 ft^3 volume. Machine-mix the batch 3 to 5 min to attain homogeneous mixture, followed by approximately 3 min rest to determine the concrete slump. Return the concrete to the mixer and remix for 2 min.

- It is important not to vary the mixing sequence or procedure from batch to batch. Add fine and coarse aggregate to the mixing container and mix until it appears that a homogeneous mixture has been attained. Then add the cement and again mix until a homogeneous mixture is attained. Next, add water from a graduated cylinder and mix the mass until the concrete is homogeneous and of the desired consistency. Remix the batch periodically during the casting of the test specimens, and keep the mix container covered to prevent evaporation.

8.7 Preparation of Test Specimens

8.7.1 Number of Specimens

At least four specimens should be made for each deicer solution, control solution, or condition to be tested.

Note: Twenty specimen molds held in a wooden box 7 3/4 x 9 3/4 x 1 7/8 in. has been found to facilitate specimen preparation.

8.7.2 Configuration of Specimens

Specimens should be in the configuration of the mold previously specified.

8.7.3 Place of Molding

The molding of specimens should be done as near as practicable to the place where the specimens are to be stored during the first 24 hr. It is suggested that they be placed on a rigid surface, free from vibration, and at a relative humidity of about 50 percent after they are made.

8.7.4 Placing and Consolidation of Concrete in the Molds

8.7.4.1 *Placing and Rodding*

Concrete is to be placed in the mold in three equal layers. Rod each layer a total of 25 strokes with the rounded end of the 3/8-in. diameter x 12-in. long tamping rod. Distribute the strokes uniformly over the cross section of the mold, and for each upper layer, allow the rod to penetrate into the underlying layer. After each layer is rodded, tap the outside of the mold lightly 10 to 15 times to close any holes left by rodding and to release any air bubbles that may have been trapped. The final layer should slightly overfill the mold.

8.7.4.2 *External Vibration*

Fill each mold about half full, and rod around the perimeter and in the center about 5 times with a 3/8-in. diameter x 12-in. long tamping rod.

Overfill each mold about 1/4 in., and place on a vibration table. Vibrate according to ASTM C 192, paragraph 6.4.3.

Note: It is helpful to hold the specimen molds down in the box, during vibration, using a board approximately 8 x 10 x 3/4 in.

8.7.5 Finishing

After consolidation, finish the top surface by striking it off with a wooden striking board or steel trowel to smooth and level the surface exactly even with the top edges of the mold.

8.7.6 Curing

8.7.6.1 *Initial Cure and Demolding of Freeze/Thaw Test Specimens*

Immediately upon completion of molding, cover the test specimens to prevent excessive evaporation of water. Keep all test specimens in the molds 20-24 hr, and then demold them by removing the tape from the mold's side surface. Flex the sides of the mold while exerting pressure to the concrete bottom surface with the thumbs.

8.7.7 Storage

After demolding, place the test specimens in a moist storage tank at 73° ± 3°F (22.8° ± 1.7°C) and near 100 percent relative humidity for 13 days.

Specimens should be cured an additional 14 days at 73° ± 3°F (22.8° ± 1.7°C) and 50 percent ± 5 percent relative humidity before testing.

8.8 Temperature and Humidity

8.8.1 Temperature

The temperature should conform to the requirements of ASTM Method C 109-90. Materials should be brought to a room temperature of 68° to 81.5°F (20° to 27.5°C) before mixing. Temperature of the mixing water, moist tank, and storage tank should be maintained at 73° ± 3°F (23° ± 1.7°C).

8.8.2 Humidity

The humidity should conform to the requirements of ASTM Method C 109-90. Lab relative humidity should be 50 percent ± 5 percent. The moist tank for curing and storage should not be less than 95 percent.

8.9 Test Specimen Age

After 28 days (1 day, covered, in ambient air while still molded and 13 additional days at near 100 percent relative humidity and 14 additional days at near 50 percent relative humidity), the specimens are ready for testing. Specimens of the same age and curing conditions should be used for each set of tests.

8.10 Compressive Strength Control Test Specimens

To ensure the similarity of batches of concrete of the same mix design made at different times, three 2-in. cubes are to be made, cured, and tested for compressive strength after 28 days curing in accordance with Sections 7 and 8 of this method. ASTM C 109-90 states in paragraph 11, titled "Precision-Compressive Strength," that the results of two properly conducted tests in a single laboratory of single batches of mortar made with the same materials on the same day should not differ from each other by more than 10.7 percent of the average.

8.11 Test Procedure and Test Cell (See Figure B-6, page 216)

8.11.1 Labeling of Specimens

Test specimens should be labeled on an end surface area using a suitable permanent marker which will not be affected by water or deicer solution. A convenient marking scheme should be determined from the test matrix.

8.11.2 Initial Weights and Densities

Specimen weights should be determined to the nearest 0.1 g. Densities for each specimen should be calculated.

8.11.3 Presoaking of Specimens

As soon as possible after weighing, specimens should be placed in a suitable container and deionized water added until the tops of the specimens are covered by 1/4 in. of water. The container(s) should be covered. Specimens are to be presoaked in deionized water for 24 hr at 73° ± 3°F 22.8° ± 1.7°C) before being placed in actual test cell containers.

8.11.4 Preparation of Test Cells and Test Solutions

8.11.4.1 *Sponges*

Sponges to be used in test cells should be rinsed several times in deionized water, wrung out, and allowed to completely dry before use. One sponge is placed in each test cell.

8.11.4.2 *Test Solutions*

- Test solutions should be prepared with deionized water.
- Concentrations of candidate inhibitors should be on a weight percent of total solids basis.
- A 310-mL quantity of the specified test solution should be poured into the test cell.

8.11.5 Placement of Test Specimens

Four test specimens are placed on the sponge in each test cell with one end in full contact with the sponge. Cover the test cell with a tight-fitting lid, slightly compressing each test specimen into the sponge.

8.11.6 Freeze-Thaw Cycling

Place all test cells in a suitable freezer for 16 to 18 hr. Temperature should reach 0° ± 5°F (17.8° ± 2.3°C) to ensure complete freezing of the deicer test solutions. After the appropriate time interval, remove all test cells from the freezer and place in laboratory air (73° ± 3°F) to thaw for 6 to 8 hr. This freeze/thaw cycle is repeated for nine more cycles.

8.11.7 Evaluation of Test Specimens

After complete thawing following the tenth cycle, test specimens should be carefully removed from the test cell, individually rinsed under running tap water, and hand-crumbled to remove any material loosened during the freeze/thaw cycling. The largest intact part of each test specimen is then placed in lab air to dry for 24 hr (at 73° ± 3°F and 50 percent ± 5 percent relative humidity). After drying, test specimens are weighed and the final weights recorded. Collection, drying and weighing of sealed material may be used as an alternate method for quantitation.

8.11.8 Controls

8.11.8.1 *Deionized Water*

It is recommended that one test cell solution contain deionized water only. This test cell should be used to monitor the temperature during freeze/ thaw cycles. A small thermocouple should be cast in one test specimen to measure the core temperature during freeze/thaw cycling.

8.11.8.2 *Sodium Chloride*

A solution of 3 weight percent NaCl in deionized water should be used in at least one test cell. Specimens from these cells are used to compare the relative effects of other deicer materials on concrete.

8.12 Calculations

8.12.1 Density

The density of each freeze/thaw test specimen is calculated as follows:

$$\text{Density (g/cm}^3\text{)} = \text{Initial weight (g)/54 cm}^3$$

$$\text{Density (lb/ft}^3\text{)} = \text{Initial weight (g)} \times 1.16$$

8.12.2 Percent Weight Loss

- For each test specimen:

$$\text{Weight loss (\%)} = \frac{\text{Initial wt (g)} - \text{Final wt (g)}}{\text{Initial wt (g)}} \times 100\%$$

8.13 Report

The report on test results should contain the following:

- Cement content and water-to-cement ratio of the concrete used
- Curing procedure if other than standard
- Composition and concentration of test solutions

- Number of freeze/thaw cycles
- Mean and standard deviation of percent weight loss for specimens in each test cell
- Twenty-eight-day compressive strength of concrete test cubes for each batch of concrete used to make test specimens.

8.14 Example Test Results

An example of typical test data generated using this test method appears in Table B-17.

Table B-17. Example results of test for rapid evaluation of effects of deicing chemicals on concrete

Deicer solution or control	Number of F/T cycles[a]	Means weight percent of spalled material removed from test specimens[b] (g)	Standard deviation[c]
3 weight percent NaCl	5	10.7	±4.4
	10	31.2	±7.7
3 weight percent $CaCl_2$	5	2.7	±1.2
	10	9.3	±1.0
3 weight percent $MgCl_2$	5	0	0
	10	0	0
3 weight percent CMA	5	0	0
	10	0.7	±1.4[d]
Deionized water	5	0	0
	10	0	0

[a] Sears chest freezer.
[b] Prepared from concrete Batch Nos. 2 and 5.
[c] For four test specimens.
[d] One test specimen had weight loss and three test specimens had weight gain.

Figure B-6. Concrete spalling minitest cell.

SHRP H-205.9

Test Method for Evaluation of Scaling Effects of Deicing Chemicals on Concrete Surfaces

9.1 Scope

This test method covers evaluating the scaling effects on horizontal concrete surfaces exposed to freezing-and-thawing cycles in the presence of deicing chemicals. It is intended for use in evaluating this effect qualitatively by visual examination, and quantitatively by weight loss.

1. The values stated in metric units are to be regarded as the standard.
2. This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

9.2 Applicable Documents

9.2.1 ASTM Standards

- C 143-90a Test Method for Slump of Portland Cement Concrete
- C 156-89a Test Method for Water Retention by Concrete Curing Materials

- C 192-90a Method of Making and Curing Concrete Test Specimens in the Laboratory
- C 511-85 Specifications for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
- C 672-91 Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals

9.3 Significance and Use

- This test method can be used to evaluate and compare the effects of deicing materials on the scaling of a concrete surface exposed to freeze/thaw cycles.
- This test method is not intended to be used in determining the durability of aggregates or other ingredients of the concrete.
- No relationship has been established between specimens cut from hardened concrete and specimens prepared in the laboratory.

9.4 Apparatus

9.4.1 Freezing Equipment

An upright or chest freezer or room of sufficient size to hold the specimens and capable of lowering the temperature of the specimens to 0° ± 5°F (-17.8° ± 2.7°C) within 16 to 18 hr and maintaining this temperature with a full load of specimens.

9.4.2 Molds

1. Molds should be made of 3/4-in. (1.91 cm) plywood and should have inside dimensions of 6 x 12 x 3 in. (15.24 x 30.48 x 7.62 cm).
2. A plywood spacer block 4 1/2 x 10 1/2 x 3/4 in. (11.43 x 26.67 x 1.91 cm) is wrapped with Saran® wrap and centered on and secured to the floor of the mold using two 1 1/4-in. (3.175 cm) No. 8 flathead wood screws. The screws should be entered from the outside bottom of the mold and should be flush with the surface or countersunk. Care

should be taken to ensure that the Saran® wrap is smooth and tight on the surface of the spacer block facing up into the mold.

3. A 24-gauge (.025 in., 0.635 cm) stainless steel rectangular band having inside dimensions of 4 1/2 x 10 1/2 x 1 1/2 in. (11.43 x 26.67 x 3.81 cm), silver-soldered on one corner, should be fabricated and fitted around the perimeter of each spacer block, flush with the mold floor. If necessary, the spacer blocks can be sanded around the edges so that the steel bands fit. Fitting the stainless steel bands' should be done prior to wrapping and attachment of the spacer block.

4. Facilities for curing concrete; conforming to ASTM C 192-90a.

5. Slump cone, base, and tamping rod conforming to ASTM C 143-90a.

6 Scales conforming to the requirements of ASTM C 192-90a.

9.4.3 Concrete Mixer

A power-driven revolving drum, tilting mixer, or suitable revolving pan or revolving-paddle mixer capable of thoroughly mixing batches of the prescribed sizes at the required slump.

A pan mixer may be more suitable than a revolving drum mixer for mixing small batches of concrete. The rate of rotation, degree of tilt, and rated capacity of tilting mixers are not always suitable for laboratory-mixed concrete. It may be desirable to reduce the rate of rotation, decrease the angle of tilt from the horizontal, and use the mixer at somewhat less than the manufacturer's rated capacity.

9.4.4 Small Tools

Wood strike-off board, steel trowel, paint scraper, screwdrivers, electric drill, small crowbar, 1-in. (2.54 cm) bristle paint brush with bristles cut to 3/4-in. (1.91 cm) length, scrub tester brush.

9.4.5 Other Equipment and Materials

Plastic dish pan, glass funnel, filter flask, coarse filter paper, drying oven, wash bottles, rubber bands, polyethylene sheeting, thermocouples, digital thermometer, chart recorder, and glassware for mixing and storing solutions.

9.5 Concrete

1. Concrete with the following characteristics has been found useful for the purposes of this test method: (a) non-air-entrained; (b) cement content, 564 ± 9.4 lb/yd^3 (334 ± 3 kg/m^3); (c) slump 2.5 ± 1.0 in. (6.35 ± 2.54 cm); and (d) durable aggregate (Note 1) of 3/8-in. (0.983 cm) maximum size. If desired, air entrained concrete may also be used. Refer to ASTM C 672-91 for specifications.

   Note 1: Care should be taken to use an aggregate which has a good performance record in freezing and thawing exposure.

The mix design designated for this test is specified as follows:

Water (tap)	9.11 weight percent
Cement (Portland Type 1)	17.79 weight percent
Sand (50/50 coarse/fine)	31.13 weight percent
Aggregate (3/8-in. max. limestone, saturated (surface-dry)	41.97 weight percent
Water/cement ratio	0.51
Slump (ASTM C 143-78)	2.5 ± 1 in. (6.35 ± 2.54 cm)
Density	≈ 145 lb/ft^3 (2.3229 g/cm^3)
Compressive strength	> 4,000 psi (27.6 MPa)

Non-air-entrained.

2. Machine-mix and test in accordance with applicable provisions of method ASTM C 192-90a.

9.6 Test Specimens

- Specimens should have the following dimensions, 6 x 12 x 3 in. (15.24 x 30.48 x 7.67 cm). At least two specimens for each deicer or control solution should be tested.

9.6.1 Fabrications of Specimens

Specimens should be made in molds as described in Section 4.2. If possible, all specimens used in a given series of tests should be prepared from the same batch of concrete. Concrete should be placed in the molds carefully, filling areas between the stainless steel band and the mold walls first. Placing may be aided by a tamping rod or other suitable tool. Molds should be filled with a slight excess of material. After placing, the specimens should be consolidated by vibrating according to ASTM C 192-90a, paragraph 6.4.3, and then tapped on the sides and ends with a hammer to aid consolidation. After consolidation, the specimen surface should be leveled and struck off with a strike-off board or plate and finished with a steel trowel. Time between placing and finishing should be no more than 20 min. If it is desirable to monitor the core temperature of the concrete specimen during freeze/thaw cycling, a suitable thermocouple, with a small diameter, plastic insulated wire, should be embedded in the center of a specimen during placing.

9.6.2 Curing and Demolding of Specimen

Specimens should be cured 20 to 24 h in the molds at ambient conditions (73° ± 3°F, relative humidity 45 to 55 percent). After demolding, specimens are placed in moist storage (relative humidity ~ 100 percent) as provided for in Specification ASTM C 511-85. At 14 days, specimens are removed from moist storage and stored in air for an additional 14 days at 73° ± 5°F (23° ± 1.7°C) and 45 to 55 percent relative humidity. When demolding specimens, the wooden spacer block attached to the mold floor is also removed from the specimen, leaving a steel-lined reservoir embedded in the specimen which will serve to contain a deicing solution for this test.

9.7 Control and Test Solutions

- All solutions should be prepared using deionized water.

- It is suggested that sodium chloride at 3 weight percent be used as a control solution. This control should be included with each series of tests.

- Deionized water should also be run as a control. It is useful to embed a thermocouple in one of the test specimens for temperature monitoring.

9.8 Procedure

1. At 28 days, transfer 200 mL of the appropriate solution to the reservoir of the specimen. Label each specimen.

2. Cover each specimen with a polyethylene sheet secured with a rubber band. This inhibits water loss due to evaporation and helps protect against spills during handling and testing.

3. Place specimens in a freezing environment for 16 to 18 hr. At the end of this time remove them from the freezer and place them in laboratory air at 73° ± 3°F (23° ± 1.7°C) and a relative humidity of 45 to 55 percent for 6 to 8 hr. Deicer solution should cover the entire surface through the duration of the test. Repeat this cycle daily.

4. When specimens have thawed at the end of the fifth cycle, the following evaluations of the degree of scaling are made.

9.8.1 Gravimetric Evaluation

1. Remove plastic cover. Stand specimen on its end in a plastic dishpan, allowing test solution to run off. Flush the test surface with deicer solution from a wash bottle. Use the bristle brush or scrub tester brush to aid removal of any scaled material. In some cases, light mechanical abrasion with a spatula may be necessary. Flush surface again with deicer solution. Repeat as necessary until all scaled material has been removed. Test solution, rinse water, and scaled material should be contained in the dishpan.

2. Transfer contents of the dishpan to a suitable beaker and allow scaled material to settle for 5 min. Carefully decant the liquid from the beaker until about 200 mL of solution and scaled material remain.

3. Filter this material using standard filtration methods. Slight vacuum may be used. Water should be used to aid transfer of the material to the funnel and to wash the solids during filtering. Filter paper should be labeled and weighed to the nearest 0.1 g prior to use.

4. The filter paper with residual solids should be dried in a forced-air oven at ~ 122°F (50°C) overnight.

5. After drying, the filter paper and residual solids are weighed to the nearest 0.1 g. The initial weight of the filter paper is then subtracted to determine the quantity of scaled material. Two specimens for each condition are determined.

6. Fresh solution is placed in the reservoirs and the specimens are covered and subjected to additional sets of five freeze/thaw cycles. The gravimetric evaluation is repeated. The total weight of scaled material removed from each test specimen is determined. The number of freeze/thaw cycles may be extended to 50 or more (see ASTM C 672-91).

9.8.2 Visual Evaluation

At the end of each five cycles, after the scaled material has been removed from the specimen surface, a visual rating may be given to each specimen according to the guidelines in ASTM C 672-84:

Rating	Condition of surface
0	No scaling
1	Very slight scaling (1/8 in., 3.2 mm) depth, max., no coarse aggregate visible
2	Slight to moderate scaling
3	Moderate scaling (some coarse aggregate visible)
4	Moderate to severe scaling
5	Severe scaling (coarse aggregate visible over entire surface)

Photographs or a word description of the surface are also useful.

9.9 Calculations

$$\text{wt of scaled material removed} = \text{wt of paper and solids after drying} - \text{initial wt of paper}$$

$$\text{cumulative wt of material removed} = \text{wt of solids after first 5 cycles} + \text{wt of solids after second 5 cycles}$$

9.10 Report

The report should include the following:

1. Cement content, water-cement ratio, slump, and compressive strength of concrete.
2. Visual rating of the surface after 5 to 10 cycles in accordance with ASTM C 672-84.

3. Mean cumulative weight of scaled material for each deicer and control solution tested.

4. Deicers and concentrations tested.

9.11 Example Test Results

An example of test results generated using this test method is presented in Table B-18.

Table B-18. Example results of SHRP H-205.9 test for evaluation of scaling effects of deicing chemicals on concrete surfaces

Deicer solution or control	Number of F/T cycles[a]	ASTM visual grade	Avg. cumulative weight[b] of scaled material removed from test specimens[c] g)
3 weight percent NaCl	5	3 to 4	40.6
	10	5	144.6
	15	5	222.9
3 weight percent $CaCl_2$	5	3	25.7
	10	4 to 5	56.4
	15	5	92.3
3 weight percent $MgCl_2$	5	0 to 1	1.2
	10	1	2.7
	15	1 to 3[d]	3.5
3 weight percent CMA	5	0	< 0.1
	10	0	0.6
	15	1	1.7
Deionized water	5	0	ND
	10	0	0.1
	15	0	ND

[a] Sears chest freezer.
[b] Duplicate test specimens.
[c] Prepared from concrete Batch Nos. 2 and 5.
[d] Scaling was localized in pits.
ND = not determined.

SHRP H-205.10

Test Method for Evaluation of Frictional Characteristics of Deicer Chemicals

10.1 Scope

1. This method covers the procedure for measuring frictional characteristics of deicer materials using the British Pendulum Skid Resistance Tester. A procedure for checking the calibration of the tester is included in the method.

2. The British Pendulum Tester is a dynamic pendulum impact-type tester used to measure the energy loss when a rubber slider edge is propelled over a test surface. The tester is suited for laboratory as well as field tests on flat surfaces.

3. The values measured, British Pendulum (Tester) Number expressed as BPN, for flat surfaces represent the frictional properties obtained with the apparatus. The procedures stated herein and do not necessarily agree or correlate with other slipperiness measuring equipment.

10.2 Referenced Documents

10.2.1 ASTM Standards

- E 501-82 Specification for Standard Tire for Pavement Skid Resistance Tests

- E 303-83 Standard Method for Measuring Surface Frictional Properties Using the British Pendulum Tester

10.2.2 Literature References

C. G. Giles, Barbara E. Sabey, and K. W. F. Carden. "Development and Performance of Portable Skid-Resistance Tester," *Road Research Technical Paper No. 66*, Road Research Laboratory, Dept. of Scientific and Industrial Research, England, 1964.

H. W. Kummer and D. F. Moore. "Concept and Use of the British Portable Skid-Resistance Tester," *Report No. 6*, PDH-PSV Joint Road Friction Program, Dept. of Mechanical Engineering, The Pennsylvania State University, State College, PA 16802, June 1963.

10.3 Summary of Method

1. This method uses a pendulum-type tester with a standard rubber slider to determine the frictional characteristics of deicer materials on a standard test surface.

2. The test surface is cleaned and thoroughly wetted before testing.

3. Before conducting the test, the pendulum slider is positioned to barely come in contact with the test surface. The pendulum is raised to a locked position then released, allowing the slider to make contact with the test surface.

4. A drag pointer indicates the British Pendulum (Tester) Number (BPN). The greater the friction between the slider and the test surface, the more the swing is retarded, and the larger the BPN reading. At least six swings of the pendulum are made for each deicer material on the standard test surface.

10.4 Significance and Use

1. This method measures the frictional property of microtexture of surfaces in the laboratory.

2. By using a standard surface, thereby holding constant the surface microtexture, the frictional characteristics of liquid deicer materials can be determined and compared.

10.5 Apparatus

10.5.1 British Pendulum Tester

The pendulum with slider and slider mount should weigh 1500 ± 30 g. The distance of the center of gravity of the pendulum from the center of oscillation should be 16.2 ± 0.2 in. (411 ± 5 mm). The tester should be capable of vertical adjustment to provide a slider contact path of 4 15/16 ± 1/16 in. (125 ± 1.6 mm) for tests on flat surfaces. The spring and lever arrangement should give an average normal slider load between the 3-in. (76.2 mm) wide slider and test surface of 2,500 ± 100 g as measured by the method prescribed in the Annex of ASTM E 303-83. See Note 1.

Note 1: A simple method to confirm the slider load is to hold the pendulum perpendicular to the pan of an electronic balance, 3,000 g minimum capacity, and then deflect the slider spring about 1/8 in. by pressing the pendulum downward. The balance display should read 2,500 g ± 100 g.

10.5.2 Slider

The slider assembly should consist of an aluminum backing plate to which is bonded a 1/4- x 1- x 3-in. (6- x 25- x 75-mm) rubber strip for testing flat surfaces. The rubber compound should be natural rubber meeting the requirements of the U.K. Transport Research Laboratory or synthetic rubber as specified in ASTM E 501-82.

1. New sliders should be conditioned prior to use by making ten swings on No. 60 grade silicon carbide cloth or equivalent under dry conditions. See Note 2. The swings should be made with a tester adjusted as in Section 10.7.

Note 2: Material suitable for this purpose is available from 3M Co., St. Paul, MN, under the trade name Type B Safety-Walk.

2. Wear on the striking edge of the slider should not exceed 1/8 in. (3.2 mm) in the plane of the slider or 1/16 in. (1.6 mm) vertical to it.

10.5.3 Accessories

1. Contact path gauge should consist of a thin ruler suitably marked for measuring contact path length between 4 7/8 and 5.0 in.

2. Miscellaneous equipment, such as water container, surface thermometer, wash bottles, and sponge is recommended.

10.6 Test Surface

1. Laboratory studies have indicated that sandblasted glass gives the most consistent and reliable test results when comparing the frictional characteristics of different deicer materials. Peak to valley profile of the sandblasted glass should be 1.0 to 1.5 mils.

2. The test surface should be clean and free of loose particles and should be held rigidly so as not to be moved by the force of the pendulum.

3. The test surface should have a surface area of at least 3 1/2 by 6 in. (89 by 152 mm).

4. Test surfaces prepared from paving materials, such as asphalt and concrete, are difficult to standardize, decontaminate, and replicate. Therefore, these test surfaces are not considered suitable for use in this laboratory test procedure.

10.7 Preparation and Calibration of Apparatus

Refer to ASTM E 303-83 for a complete description of the apparatus.

10.7.1 Leveling

Level the instrument accurately by turning leveling screws until the bubble is centered in the spirit level.

10.7.2 Zero Adjustment

Raise pendulum mechanism by loosening locking knob (directly behind pendulum pivot) and turn either head movement knob at center of tester to allow slider to swing free of test surface. Tighten locking knob firmly. Place pendulum in release position and rotate the drag pointer counter clockwise until it comes to rest against adjustment screw on pendulum arm. Release pendulum and note pointer reading. If reading is not zero, loosen locking ring and rotate friction ring on bearing spindle slightly and lock again. Repeat test and adjust friction ring until the pendulum swing carries the pointer to zero.

10.7.3 Slide Length Adjustment

1. With pendulum hanging free, place spacer under adjusting screw of lifting handle. Lower pendulum so edge of slider just touches surface. Lock pendulum head firmly, raise lifting handle, and remove spacer.

2. Raise slider by lifting handle, move pendulum to the right. Lower slider, and allow pendulum to move slowly to the left until the edge of slider touches the surface. Place the path gauge beside the slider and parallel to direction of swing to verify length of contact path. Raise the slider, using lifting handle, and move pendulum to left, then slowly lower until the slider edge again comes to rest on surface. If the length of the contact path is not between 4 7/8 and 5.0 in. (124 and 127 mm) on the test surface, measured from trailing edge to leading edge of the rubber slide, adjust by raising or lowering the instrument with the front leveling screws. Readjust level of instrument if necessary. Place the pendulum in release position and rotate the drag pointer counterclockwise until it comes to rest against the adjustment screw on pendulum arm.

3. Alternatively, the slide path length can be lengthened by lowering the pendulum or shortened by raising the pendulum, as detailed in Section 10.7.2.

10.8 Procedure

1. Apply sufficient solution of deicer material to cover the test area thoroughly. Wash bottles are useful for applying test solutions. Execute one swing, but do not record reading.

Note 3: Always catch the pendulum during the early portion of its return swing. While returning the pendulum to its starting position, raise the slider with its lifting handle to prevent contact between the slider and the test surface. Prior to each swing, the pointer should be returned until it rests against the adjustment screw.

2. Without delay, make five more swings, rewetting the test area with solution of deicer material each time, and record the results.

Note 4: If the BPN changes by more than one number over the course of the five swings, continue until five consecutive swings are within 1 BPN. Use the last five BPN for the average computation, e.g.,

—not acceptable—
| |
53, 53, 51, 50, 50, 50, 51
| |
—— acceptable ——

Note 5: Care should be taken that the slider remains parallel to the test surface during the swings, and does not rotate so that one end rather than the entire striking edge makes the initial contact. Available data indicate that tilting of the slider may cause erroneous BPN readings.

Installation of a small flat spring will relieve the problem. The spring can be inserted into a slot in the spring clip and the assembly secured by the cotter pin as shown in ASTM E 303, Figure 4. The free ends of the spring can rest on the slider backing plate to restrain the slider from tilting.

3. If more than one deicer or concentration is to be evaluated, the following procedure is recommended for cleaning the glass surface between tests: (1) sponge off residual solution from primary test; (2) rinse surface using deionized water and sponge three times (if desired, the BPN for deionized water may be redetermined to ensure the test surface has been cleaned); (3) rinse surface once with the next solution to be tested; and (4) reapply test solution.

4. Recheck the slide contact length in accordance with 1.7.3.

5. In any series of tests, the frictional values (BPN) of deionized water and 3, 10, and 25 weight percent sodium chloride should be determined for comparative purposes before evaluating other deicer test solutions.

10.9 Report

The report should include the following:

1. Individual values in BPN.

2. Temperature of the test surface.

3. Type, age, condition, texture and location of test surface, as applicable.

4. The BPN for a test location should be reported as the average of the five swings, to the nearest whole number.

Example:

	Swing No.					
Initial swing	1	2	3	4	5	Average
52	51	51	51	50	50	50.6 = 51

5. Number, type and age of the rubber slider.

6. Deicers and concentrations tested.

10.10 Precision, Bias, and Sources of Error

1. Repeated tests show standard deviations as follows:

British rubber sliders	1.0 BPN unit
Rubber sliders (conforming to ASTM specification E 501-82	1.2 BPN units

In both cases the upper quartile of variability is represented in prevailing test instruments. As there is no marked correlation between standard deviation and arithmetic mean of sets of test values, it appears that standard deviations are pertinent to this test regardless of the average skid resistance being tested.

2. The relationship, if any exists, of observed BPN units to some "true" value of skid resistance has not been and probably cannot be determined. As a result, precision and bias of this test in relation to a true skid resistance measure cannot be evaluated, and only repeatability is given for the method.

3. Determine the testing error as follows:

$$E = t\sigma n^{-\frac{1}{2}}$$

where: E = testing error
t = normal curve of 1.96 or 2.0 rounded
σ = standard deviation of individual test results (BPN units)
n = number of tests

4. In order to ensure that the testing error stays within 1.0 BPN unit at a 95 percent confidence level (corresponding to a normal curve of 1.96 or 2.0 rounded), the following sample sizes are needed:

British natural rubber sliders:	4
Synthetic rubber sliders (conforming to ASTM Specification E 501):	5

5. The main sources of error are (1) adjustment and calibration of apparatus, (2) setting zero adjustment, and (3) setting and resetting contact path length.

10.11 Example Test Results

A typical example of test data obtained using this test method is given in Table B-19.

Table B-19. Example test results for frictional characteristics of various deicer materials on sand-blasted glass at 20°, 30°, and 75°F

Deicer/control	Deicer concentration (weight percent)	Friction value (BPN)[a]		
		75°F	30°F	20°F
Deionized water	–	66-67		
Sodium chloride	3	–	65	–
	10	66	63	–
	25	62	59	57
PCI-inhibited sodium chloride	10	66	67	62
Rock salt	10	64	64	–
	25	60	60	59
Sodium formate	10	66	63	–
	25	61	56	56
Calcium chloride	10	66	66	–
	25	61	62	58
Magnesium chloride	10	61	60	58
	25	52	52	51
Urea	10	66	73	–
	25	64	62	61
CMA	10	63	61	–
	25	49	45	38
Ethylene glycol	10	64	63	–
	25	61	59	57
	100	31	28	21

[a] BPN = British pendulum number; rounded mean of five consecutive BPN readings that vary by no more than 1 BPN.

SHRP H-205.11

Test Method for Preliminary Assessment of Ecological Effects of Deicing Chemicals

11.1 Scope

1. Test methods are presented for determination of the potential ecological effects of deicing chemicals on aquatic systems and seed germination.

2. Aquatic tests include chronic and acute toxicity studies with representative aquatic species including species collected from local or potentially affected sites.

3. Seed germination tests encompass representative seeds with test results based on percentage germination and radicle elongation.

11.2 Applicable Documents

EPA/600/4-85/013	Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms
EPA/600/4-89/001	Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (2nd Edition)
EPA/560/6-82/002	Environmental Effects Testing Guidelines—Part One and Part Two
EPA/560/5-75/008	Test Methods for Assessing the Effects of Chemicals on Plants

EPA/540/09-88/035	Aquatic Mesocosm Test to Support Pesticide Registration
FDA 223-802352	Standardized Aquatic Microcosm Protocol
ASTM E 729-88a	Standard Practice for Conducting Acute Toxicity Tests With Fishes, Macroinvertebrates, and Amphibians
ASTM E 978-84	Standard Practice for Evaluating Environmental Fate Models of Chemicals
ASTM STP 920	Community Toxicity Testing

11.3 Significance and Use

1. The test methods may be employed primarily for initial screening and for delineation of potential impacts of chemical deicers and for comparison of alternative deicers with common deicers exemplified by sodium chloride and calcium chloride.

2. The tests involving aquatic species are conducted in small standard static test systems. Test results accordingly reflect a relatively narrow range of conditions encountered in an actual environment.

3. The construction and general mode of use of an aquatic flow-through system is presented in the Annex. Tests in this dynamic system can be expected to yield more definitive results with regard to deicer impacts under environmental conditions encountered in naturally occurring systems.

4. These tests may involve hazardous materials, operations, and equipment. These test methods do not purport to address all of the safety problems associated with their use. It is the responsibility of whoever uses these tests to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

11.4 Summary of Test Methods

11.4.1 Acute Toxicity Tests

These tests provide initial screening of chemical deicers for their acute toxicity to representative aquatic animals.

11.4.1.1 *Range-Finding Test*

Specimens from a selected species are placed in beakers containing solutions of a deicer at four concentrations plus one control with no deicer. After 24 hr the mortality rate is calculated. Water quality parameters are measured at the beginning and end of the test.

11.4.1.2 *Main Test to Determine LC_{50}*

Specimens from a given species are placed in beakers containing solutions of a deicer at six concentrations, plus one control with no deicer. Concentrations are chosen from the range-finding test data to span from nearly zero to 100% mortality. Four replications are done at each concentration. After 24 hr the mortality rate for each concentration is noted and used to determine the LC_{50} (the concentration of deicer at which 50% of the specimens die).

11.4.2 Chronic Toxicity Test

This test provides initial screening of chemical deicers for their long-term toxicity to representative fish species. Fathead minnow larvae are placed in beakers containing solutions of a deicer at six concentrations, plus one control with no deicer. The highest concentration used is at the LC_{50} determined from the main acute toxicity test. Two replications are used for each concentration. Water quality parameters are monitored, dead specimens are removed, and the test solutions are replaced daily for 7 days. The mortality rate is noted for each concentration.

11.4.3 Seed Germination/Radicle Elongation Test

This test provides initial screening of chemical deicers for their effects on representative terrestrial seed plants.

Seeds of plant species, such as oats, sunflowers, soybeans, vetch, or fescue, are placed on filter paper in petri dishes. Three replications are used for each of six deicer concentrations, and a control with no deicer. A small amount of deicer solution is added to each dish. The dishes are covered and stored in the dark for 5 days. At the end of 5 days, the number of seeds germinating and the lengths of the radicles are recorded for each deicer concentration.

11.4.4 List of Tests and Test Species

1. Acute Toxicity Tests (static)

    a. Fathead minnow, *Pimephales promelas*; juveniles
    b. Common guppy, *Lebistes reticulatus*; young adults
    c. Bluntnose minnow, *Pimephales notatus*; juveniles
    d. Mayfly, *Heptageniidae* (family); *Stenonema* and *Stenacron*; nymphs
    e. Amphipods, *Gammarus*; adults

2. Chronic Toxicity Test (static, with renewal, daily for 7 days)

    a. Fathead minnow, *Pimephales promelas*; larvae

3. Seed Germination/Radicle Elongation Test

    a. Oats, sunflowers, soybeans, vetch, and fescue seeds

11.5 Equipment

1. Suitable glassware, aquariums, pumps, filters, aerators, temperature controllers, and laboratory equipment for preparing and storing solutions.

2. All equipment for handling and storing test species should follow EPA 600/4-85/013, Section 4—Facilities and Equipment, pp. 10-12.

3. Miscellaneous small tools—paintbrush (bristles, 5 mm wide and 10 mm long).

11.6 Acute Toxicity Tests (Reference Document EPA/600/4-85/013)

11.6.1 Acquisition and Maintenance of Species to Be Tested

11.6.1.1 *Fathead Minnows*

Juvenile fathead minnows (approximately 30 days old and 15 to 25 mm long, about half the average adult length of 50 mm) are obtained from the Environmental Monitoring and Support Laboratory Newtown Facility in Cincinnati, Ohio, or equivalent. The fish (500 to 1,000) are received and placed in a 30-gal aquarium at 68°F (20°C) which will also be the test temperature. The aquarium contains gravel and a filter and aeration system typical of systems obtained from commercial aquarium supply stores. Water for the aquarium is the same as the test synthetic dilution water and is prepared according to EPA/600/4-85/013, Section 6, pp. 22-23, as the "moderately hard" dilution water. Fish are maintained in the aquarium for a few days for any initial mortality to occur due to transport and transfer to the aquarium conditions. Generally, most fish arrive and remain healthy. Initial introduction into the aquarium should be gradual to equilibrate temperature (container suspended for a while in aquarium) and water quality (periodic partial mixing of aquarium water into container water after temperature equilibration) differences between shipping container and aquarium. With a healthy aquarium condition, fish to be used for tests can be maintained for a few weeks. Fish are fed in the aquarium, but not during 24-hr tests. Food consists of newly hatched brine shrimp (*Artemia*) nauplii which can be prepared from eggs obtained from an aquarium supply store following the label procedures or those described in EPA/600/4-85/013, p. 118. Because the brine shrimp are hatched in saltwater, they must be thoroughly rinsed with dilution water or distilled water to prevent a salt buildup in the aquarium. The fathead minnow can also be bred and reared at the testing facility as described in EPA/600/4-85/013. Before use, the aquarium and all other glassware are cleaned according to a standard EPA procedure presented in EPA/600/4-85/013, Section 4, pp. 10-12.

11.6.1.2 *Common Guppies*

Young adult common guppies are available from local aquarium supply stores. Handling is the same as described for fathead minnow, Section 11.6.1.1.

11.6.1.3 *Bluntnose Minnows*

Juvenile bluntnose minnows may be collected from a local stream. In the laboratory, they are handled and maintained as described for the fathead minnow, Section 11.6.1.1.

1. The bluntnose minnows are seined (small enough mesh size for the fish being sought) from a stream and transported to the laboratory in an aerated ice chest containing the stream water. One effective way of seining a small stream with minimal contacting of the fish with the net is to span the seine across most or all of the stream leaving slack in the net so as to form a downstream bag. Starting from a point upstream, one or more people walk downstream in a zigzag pattern, herding fish into the net which is then quickly lifted, tipping the bottom upward, trapping the fish in the bag. The portion of the net containing the fish can remain in the water, but brought close to the surface to examine and select the desired fish. Fish can be scooped out with a larger version of the cylinder with a net bottom described in Section 11.6.2.3.1.2. Once in the laboratory, these minnows are gradually transferred to an aquarium on the day of collection.

2. Certain procedures should be followed in making collections of aquatic animals which are similar, whether for fish, insects, or other small animals. The types of organisms selected for testing should be representative of the area in its uncontaminated state, avoiding organisms that have colonized contaminated sites and are thus likely to have some unusual tolerance, making them less representative of the organisms that may need to be protected. Particular care must be taken to minimize the shock of the transfer from the field to the laboratory. Gentle collection, whether by net, hand-picking, or live trap, is critical, as is maintaining the organisms in near ambient conditions of water quality (particularly temperature and oxygen) during the trip back to the laboratory. Insulated boxes (e.g., ice chests) with aeration (e.g., battery-operated minnow bucket aerators) are generally suitable. Once in the laboratory, the organisms should be gradually transferred to a suitable holding aquarium, comparable to the one described for the fathead minnows, containing the type of dilution water eventually to be used in the test. These organisms should be observed for a few days for initial mortality to optimize the establishment of healthy organisms for the test. The period of this initial holding and observation is a compromise between this relatively short period and a longer period which may introduce a slowly developing, though initially minimal stress due to the new environment to which the organisms are being exposed. Stressed test organisms may respond differently to the chemical, not necessarily identifiable from the control test organisms, thus producing unrealistic ecological effects predictions.

11.6.1.4 *Mayfly Nymphs*

1. The mayflies may be collected by hand from a local stream by lifting rocks at least 10 cm wide out of the water, then quickly examining the underside for moving individuals generally 5 to 15 mm in length. They can be picked up individually with a small paintbrush (bristles 5 mm wide and 10 mm long) by gently sliding the bristles under them, or sometimes just touching them will cause them to cling. If they are common on the stream substrate, hundreds can be collected by one person in an hour or so. As each is collected, it is placed in a 1-L plastic beaker filled with stream water and kept in the stream next to the collector. Some pieces of 10-mm diameter by 10-cm long nylon rope weighted with steel nuts (coated with silicone seal to retard rust and leaching of metals) are placed on the bottom of the beaker to provide substrate and prevent organisms from clumping together and harming themselves. After about 15 min the entire contents (including ropes) of the beaker are transferred to a 5-gal bucket (with a secure lid for the later trip to the laboratory) or ice chest set in the stream and filled with stream water and aerated. When the desired number of insects have been gathered, they are returned to the laboratory. They can be gradually transferred to an aerated aquarium (modified to contain the rope substrates, no gravel, and a depth of 15 to 20 cm) as described above or kept in the aerated bucket for 24 hr at the testing room temperature.

2. Procedures for making collections of aquatic fish and insects are as given in Section 11.6.1.3.2.

11.6.1.5 *Amphipods*

Amphipods of the genus *Gammarus*, are available from biological supply houses such as Ward Natural Science Supply, Rochester, New York.

1. When amphipods are received, they can be placed (gradual transfer as described for fish and insects) into an aquarium with 10 to 15 cm of synthetic dilution water over a layer of gravel and with aeration, but no filtering (due to small size of the organisms, approximately 5 mm). They can be fed brine shrimp or a fine settling fish food.

11.6.2 Range-Finding Test Procedure

11.6.2.1 *Test Organisms and Test Solutions*

For each chemical deicer, an initial range-finding test is performed to determine eventual main test concentration ranges. Each test consists of 24 hr of exposure. A total of 50 test organisms and 5 test beakers are required. The range test is composed of four test concentrations and a nonaddition control, each with 10 fish in separate 1-L Pyrex beakers (Pyrex No. 1040) containing 750 mL of the test solution. The four test concentrations are prepared in the following manner. The chemical deicer is weighed out in amounts calculated to prepare the desired concentrations in 750 mL of synthetic dilution water. Approximately half of the necessary dilution water is used to dissolve the chemical deicer by magnetic stirring 5 to 10 min; the remaining half of the water is used to transfer the chemical deicer solution to a 1-gal plastic jug. The jug is then capped and vigorously shaken for a few minutes, its contents emptied into the 1-L test beaker. When the four test concentration beakers and the control beaker are prepared, water quality parameters may be measured before addition of the test organism.

11.6.2.2 *Water Quality Parameters*

Though not required for the final mortality vs. concentration estimates, it is useful to measure water quality parameters prior to addition of test organisms to the test beakers, and again after the final count of surviving organisms is taken. pH, dissolved oxygen, and conductivity can be measured by standard instruments available from scientific instrument catalogs. These measurements provide insights into some water quality changes the chemical deicer may cause that may affect the test organism. Conductivity in the case of deicer chemicals provides some measure of the test concentrations that can be related to field measurements.

11.6.2.3 *Transfer of Test Organisms to Test Beakers*

Follow the loading limits (i.e., weight of organisms per volume of test solution) set by the EPA document at 0.8 g/L at 68°F (20°C) or less and 0.4 g/L above 68°F (20°C) to minimize depletion of dissolved oxygen. Ten organisms are used in each test beaker. If for some reason the EPA loading limits are not followed, then supplemental aeration for each test beaker must be provided, but must not interfere with the biological action of the chemical being tested. Transfer organisms as specified in the following subsections.

11.6.2.3.1 Fish (guppies, flathead, or bluntnose minnows). Ten fish are added to each of the five test beakers as quickly as possible, but not at the expense of handling them roughly. All fish can generally be added within 15 min of each other.

11.6.2.3.1.1 *Preferred EPA Method*. EPA, in its document, recommends concentrating the test species by siphoning the aquarium and then transferring some fish to a bowl or beaker using a large-bore, fire-polished glass tube (6 mm x 9 mm i.d. x 30 cm long) equipped with a bulb. Fish may also be dipped out of the aquarium with a bowl or beaker. Care must be taken when using these methods that as little water as possible is transferred with the fish to the test beakers, to minimize changes in the deicer concentration.

11.6.2.3.1.2 *Alternative Method*. To transfer fish to the test chambers, a small Plexiglas® cylinder (approximately 30 mm diameter, 25 mm long, with 80- to 110-µm mesh netting attached over one end) is used to "scoop up" several fish to be transferred to a test beaker. When the cylinder is lifted with fish, the water drains back into the holding beaker and then fish can be passed to the test beaker without adding any diluting water. Place the drained cylinder, held with forceps, gently into the test beaker. Allow it to fill with test water, resuspending the counted fish, and then tip the cylinder under the surface, releasing the fish.

11.6.2.3.2 Mayflies. Mayflies are transferred to test beakers by first transferring several to a Pyrex pie dish filled with aquarium water, or stream water if taken from the original bucket or cooler. If they are in the dish with stream water, an equal volume of dilution water can be added to provide some initial adjustment to new water quality conditions before final transfer to the test beakers. The transfer to the dish is accomplished by removing ropes on which insects are clinging and shaking, or gently brushing them into the dish. Individuals are then captured with a suction tube (greater than 5 mm i.d.) and placed in pairs in 20-mL plastic vials. In this manner 10 individuals are accumulated, then each vial is drained and then dipped (using forceps, keeping hands out of the test solution) into the test beaker solution and flushed out. A few rocks (inert composition so as not to affect water quality or the test chemical) should already be in place to serve as refuge.

11.6.2.3.3 Amphipods. To prepare test beakers several amphipods are sucked up with a tube as described for fathead minnows and placed in a petri dish containing aquarium water. From here they are transferred one at a time to the test beaker (500- to 1,000-mL size) containing 250 mL of the test solution. Because amphipods are sometimes cannibalistic on weaker individuals, a few small rocks (quartz or some relatively inert composition) can be added for refuge.

11.6.2.4 *Test Conditions*

The beakers are then placed under the same room conditions as the aquarium. Preferably this is in a dimly lit room or growth chamber at 68°F (20°C). The test beakers and aquarium can be provided with one tubular fluorescent bulb four or more feet away.

11.6.2.5 *Test Results*

After 24 hr, all surviving fish are counted while still in each beaker. A fish is considered alive if it shows any movement including gill movement. All survivors for a test can generally be counted within 15 min. All desired water quality parameters are again measured. Ideally, the results of the range-finding test should provide an indication of the concentration level where mortality begins, reaches 50%, and then 100%. A sample of data generated using this test appears in Table B-20.

Table B-20. Acute toxicity range-finding test results using NaCl and fathead minnows

		Dissolved oxygen (mg/L)		pH		Conductivity (μS)		
	Deicer concentration (g/L)	Initial	Final	Initial	Final	Initial	Final	Mortality (dead/total)
Control	1	7.8	5.9	7.30	7.03	250	280	0/10
	2	6.9	5.5	7.25	6.93	248	270	0/10
2.25	1	6.5	6.5	7.55	7.31	3,620	3,890	0/10
	2	7.4	5.6	7.51	7.27	3,620	3,720	0/10
4.5	1	6.8	5.8	7.70	7.37	6,400	7,200	0/10
	2	6.9	5.7	7.57	7.35	6,400	7,200	0/10
9.0	1	6.5	5.0	7.73	7.27	13,000	13,500	0/10
	2	7.5	4.2	7.65	7.26	13,000	13,200	0/10
18.0	1	6.6	4.8	7.68	7.33	24,000	24,500	10/10
	2	6.9	4.0	7.66	7.24	24,000	25,000	10/10

11.6.3 Test Procedure to Determine LC_{50}

11.6.3.1 *Test Concentrations*

Based on the range-finding test results, six deicer test concentrations are chosen, with the lowest causing almost no mortality, and the highest causing near 100 percent or 100 percent mortality. More or fewer concentrations may be tested if prior information warrants.

11.6.3.2 *Test Beakers*

Four test beakers are required for each concentration tested (4 x 6) = 24, along with four control beakers (no deicer), for a total of 28.

11.6.3.3 *Preparation of Test Solutions and Test Beakers*

Weigh the amount of chemical deicer which, when added to 3,000 mL of moderately hard dilution water (Section 11.5.1.1), will yield the appropriate test concentration. Place the weighed deicer in a large mixing beaker, add 1,500 mL dilution water, stir, and then transfer to a gallon jug. Rinse the mixing beaker with another 1,500 mL dilution water, and transfer this rinse water to the jug. Shake well. Add ~ 750 mL of this deicer solution to each test beaker.

11.6.3.4 *Water Quality Parameters*

These may be monitored as indicated in the range-finding test.

11.6.3.5 *Test Organisms*

Ten organisms per test beaker are required for a total of 280.

11.6.3.6 *Test Duration*

Test beakers are stored for 24 hr under the same conditions as indicated in the range-finding test.

11.6.3.7 *Test Results*

After 24 hr, mortality is assessed as in the range-finding test. The LC_{50} (concentration at which mortality is 50%) is determined.

11.6.3.8 *Data Analysis*

A detailed discussion of toxicity data analysis is given in EPA 600/4-85/013, Section 11, pp. 50-90. A sample of typical data generated from this test appears in Table B-21 and Figure B-7.

11.7 Chronic Toxicity Test (Reference Document EPA/600/4-89/001)

The chronic toxicity test allows for the development of the potential toxic effects of a chemical deicer on fish over a period of several days.

11.7.1 Test Duration

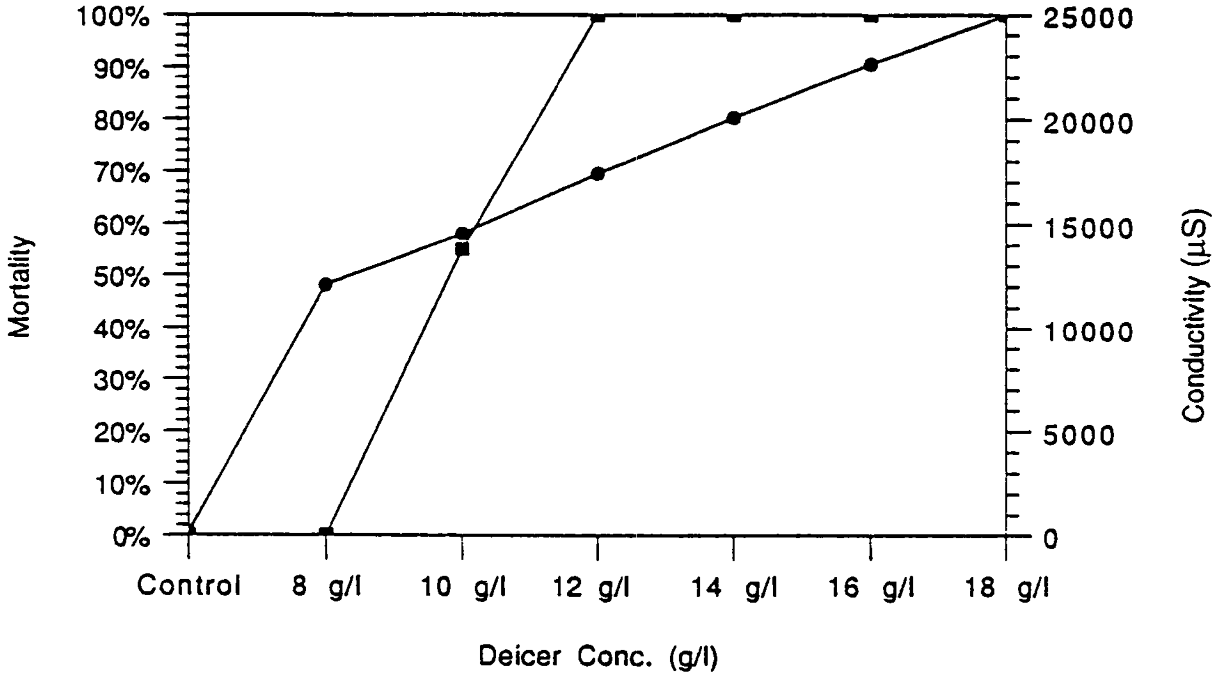
The test duration is 7 days.

11.7.2 Test Species—Fathead Minnow, *Pimephales promelas*; Larvae

Fish for this test are shipped as "eyed eggs" (fully developed embryo but still in the egg) from EPA in Cincinnati, hatch in the mail, and then arrive as less than 48-hr-old larvae. The test requires that the fish be less than 48 hr old at the start of the test. The fish are transferred to the test beakers on the day they arrive. The shipping container should be acclimated to room temperature before fish are removed.

Table B-21. Acute toxicity test results using NaCl and fathead minnows (LC_{50})

Deicer concentration (g/L)		Dissolved oxygen (mg/L)		pH		Conductivity (μS)		Mortality (dead/ total)
		Initial	Final	Initial	Final	Initial	Final	
Control	1	7.4	6.6	6.99	7.14	140	159	0/10
	2	7.4	6.0	7.11	7.13	140	151	0/10
	3	7.3	5.7	7.21	7.16	140	154	0/10
	4	7.7	6.2	7.21	7.18	139	158	0/10
8	1	7.9	5.5	7.47	7.17	11,700	12,300	0/10
	2	7.0	5.9	7.33	7.20	11,800	11,900	0/10
	3	6.8	5.2	7.43	7.12	11,700	12,100	0/10
	4	7.2	5.3	7.48	7.17	11,800	12,900	0/10
10	1	7.2	5.7	7.40	7.17	14,100	14,800	7/10
	2	7.4	5.6	7.44	7.13	14,100	15,000	5/10
	3	7.4	5.9	7.45	7.17	14,100	14,900	7/10
	4	6.7	5.1	7.51	7.09	14,100	15,000	3/10
12	1	6.8	3.9	7.35	7.07	16,500	18,000	10/10
	2	6.8	4.5	7.42	7.12	16,900	17,500	10/10
	3	7.2	2.9	7.55	6.99	16,900	18,000	10/10
	4	7.2	3.0	7.57	6.97	17,000	18,300	10/10
14	1	6.3	2.5	7.69	7.12	19,500	21,000	10/10
	2	6.5	2.7	7.84	7.15	19,500	20,900	10/10
	3	7.5	3.3	7.92	7.24	19,500	20,200	10/10
	4	7.1	3.2	7.94	7.27	19,700	20,200	10/10
16	1	6.7	5.0	7.88	7.42	22,000	23,400	10/10
	2	6.8	3.6	7.93	7.33	22,000	23,500	10/10
	3	6.5	3.3	7.91	7.31	22,000	23,200	10/10
	4	6.6	0.8	7.94	7.09	22,000	22,900	10/10
18	1	6.7	3.8	7.79	7.35	24,900	25,100	10/10
	2	6.5	1.1	7.81	7.11	24,800	24,900	10/10
	3	7.0	1.7	7.82	7.14	24,800	25,100	10/10
	4	6.7	2.6	7.80	7.19	24,800	25,500	10/10



Deicer Conc. (g/l)	Control	8 g/l	10 g/l	12 g/l	14 g/l	16 g/l	18 g/l
Conductivity	148	12025	14513	17388	20063	22625	24988
Mortality	0.0%	0.0%	55.0%	100.0%	100.0%	100.0%	100.0%

Figure B-7. **Results of NaCl acute toxicity test (LC_{50}) test fathead minnows. Percent mortality (squares) and conductivity (circles) are shown over a range of deicer concentrations.**

11.7.3 Test Concentrations

Six concentrations of the deicer are chosen. The highest concentration should be at the LC_{50} determined for the fathead minnow (see Section 11.6.3.7).

11.7.4 Test Beakers

Two beakers for each concentration (2 x 6 = 12), plus two control beakers (no deicer), for a total of 14.

11.7.5 Number of Larvae Needed

Ten per test cell, for a total of 140.

11.7.6 Test Procedure

Add 250 mL of the appropriate solution to each test cell. Using a suction tube (5 mm I.D.), remove approximately 30 fish from the shipping container and add them into a petri dish containing dilution water. Individual fish, only a few millimeters in length, are removed using a pipette with smooth edges (4 mm I.D.) and placed in the test beaker solution. The beakers are then maintained under the room conditions described above. The fish are fed three times each day at 4-hr intervals with newly hatched brine shrimp less than 24 hr old. By adding 0.1 mL of a concentrated shrimp suspension (rinsed to remove salt), about 700 to 1,000 shrimp are provided. At each successive 24-hr interval, excess food settled to the bottom is removed by suction (taking care not to remove live fish). Dead fish are removed and counted. Water quality parameters are measured carefully to avoid disturbing fish. The test solution is removed by siphoning so that about 20 percent remains (5 to 10 mm depth). New solution is added up to the 250-mL volume mark by gently pouring down the side of the test beaker. This procedure of cleaning, parameter measurement including mortality, and test solution renewal is performed at the end of each of the first 6- to 24-hr intervals. At the end of the seventh interval the test is concluded with the final parameter measurements and mortality observations. Live fish at the end can be grouped for each beaker and their dry weight measured as described in the EPA procedures. Refer to EPA/600/4-89/001, pp. 33-74, for supplementary information and data treatment.

11.7.7 Test Data

A sample of data generated using this test is given in Table B-22 and Figures B-8 and B-9.

11.8 Seed Germination/Radicle Elongation Test (Reference Document EPA/560/6-82/002)

11.8.1 Test Species

Seeds of oats, sunflowers, soybeans, vetch, or fescue. Other seed plants characteristic of the region in which the test is conducted may be chosen. Seeds are available from commercial sources.

11.8.2 Test Concentrations

Six concentrations of a given deicer, plus one control with no deicer.

11.8.3 Test Containers

Tall Pyrex® petri dishes with lid (100 x 80; No. 3250 or equivalent). Three containers per deicer concentration (3 x 6 = 18), plus three control containers (no deicer), for a total of 21.

11.8.4 Number of Seeds

Number of seeds per test container = 10 seeds of 1 species. Total seeds required = 21 x 10 = 210 seeds of 1 species.

11.8.5 Test Duration

Five days.

Table B-22. Results of NaCl chronic toxicity test using fathead minnows

Concentration (g/L)	Sample	Time	Dissolved Oxygen (mg/L)	pH	Conductivity (µS)	Mortality (dead/total)
Control	1	Day 1	6.1	7.60	272	0/10
		Day 2	5.9	7.51	300	4/10
		Day 3	6.1	7.62	302	5/10
		Day 4	6.1	7.52	350	5/10
		Day 5	5.8	7.55	348	5/10
		Day 6	6.2	7.68	291	5/10
		Day 7	4.0	6.55	170	5/10
		Day 8	4.9	7.14	312	
	2	Day 1	6.0	7.74	252	0/10
		Day 2	6.0	7.59	292	1/10
		Day 3	6.0	7.70	282	2/10
		Day 4	5.8	7.54	290	2/10
		Day 5	5.3	7.44	288	2/10
		Day 6	5.9	7.70	288	2/10
		Day 7	4.1	6.70	122	3/10
		Day 8	3.8	7.17	300	
0.5	1	Day 1	6.5	7.80	1,070	0/10
		Day 2	5.9	7.71	1,180	2/10
		Day 3	6.1	7.82	1,180	2/10
		Day 4	6.3	7.76	1,220	2/10
		Day 5	6.2	7.76	1,180	2/10
		Day 6	5.8	7.72	1,210	3/10
		Day 7	5.2	6.96	1,020	3/10
		Day 8	4.2	7.34	1,210	
	2	Day 1	6.0	7.82	1,040	2/10
		Day 2	6.0	7.78	1,120	4/10
		Day 3	5.9	7.87	1,140	5/10
		Day 4	6.3	7.82	1,200	5/10
		Day 5	6.2	7.81	1,170	6/10
		Day 6	6.0	7.77	1,190	6/10
		Day 7	5.4	7.04	1,020	6/10
		Day 8	5.4	7.61	1,180	

Table B-22. (Continued)

Concentration (g/L)	Sample	Time	Dissolved Oxygen (mg/L)	pH	Conductivity (µS)	Mortality (dead/total)
1.0	1	Day 1	6.5	7.81	1,810	0/10
		Day 2	5.7	7.76	1,900	1/10
		Day 3	6.0	7.95	1,870	4/10
		Day 4	6.1	7.79	1,900	4/10
		Day 5	6.0	7.80	1,890	4/10
		Day 6	6.1	7.85	1,890	4/10
		Day 7	5.5	7.23	1,850	4/10
		Day 8	5.8	7.72	1,910	
	2	Day 1	5.9	7.82	1,800	0/10
		Day 2	6.0	7.85	1,910	1/10
		Day 3	5.8	7.90	1,890	3/10
		Day 4	6.0	7.76	1,950	5/10
		Day 5	6.0	7.90	1,920	5/10
		Day 6	5.8	7.80	1,970	5/10
		Day 7	5.4	7.28	1,820	5/10
		Day 8	5.6	7.70	1,970	
2.5	1	Day 1	7.1	7.89	3,900	0/10
		Day 2	6.1	7.87	4,200	3/10
		Day 3	6.0	7.94	4,200	5/10
		Day 4	6.5	7.83	4,500	5/10
		Day 5	6.2	7.88	4,200	5/10
		Day 6	6.0	7.93	4,200	5/10
		Day 7	5.4	7.36	4,100	5/10
		Day 8	5.9	7.69	4,100	
	2	Day 1	6.0	7.92	3,900	0/10
		Day 2	5.9	7.85	4,100	3/10
		Day 3	6.2	7.95	4,200	5/10
		Day 4	6.1	7.79	4,200	6/10
		Day 5	5.9	7.78	4,200	6/10
		Day 6	5.9	7.87	4,300	6/10
		Day 7	5.5	7.37	4,100	6/10
		Day 8	5.7	7.69	4,200	

Table B-22. (Continued)

Concentration (g/L)	Sample	Time	Dissolved Oxygen (mg/L)	pH	Conductivity (µS)	Mortality (dead/total)
5.0	1	Day 1	7.0	7.90	7,400	0/10
		Day 2	6.1	7.88	7,500	1/10
		Day 3	6.0	7.94	8,200	2/10
		Day 4	5.6	7.55	8,900	6/10
		Day 5	6.0	7.77	8,200	8/10
		Day 6	6.0	7.89	8,700	9/10
		Day 7	5.8	7.65	8,400	9/10
		Day 8	5.7	7.66	8,500	
	2	Day 1	6.3	7.91	7,400	0/10
		Day 2	6.0	7.77	7,900	1/10
		Day 3	6.1	7.95	8,200	2/10
		Day 4	5.8	7.61	8,800	5/10
		Day 5	5.8	7.77	8,300	6/10
		Day 6	5.9	7.89	8,700	7/10
		Day 7	6.1	7.63	8,300	7/10
		Day 8	5.6	7.69	8,300	
7.5	1	Day 1	6.3	7.88	10,900	6/10
		Day 2	6.2	7.84	11,800	7/10
		Day 3	6.9	7.96	11,800	8/10
		Day 4	6.2	7.84	12,200	10/10
		Day 5	6.2	7.90	12,700	
	2	Day 1	6.3	7.87	10,800	4/10
		Day 2	5.9	7.86	11,000	6/10
		Day 3	6.2	7.92	11,400	6/10
		Day 4	6.3	7.84	12,200	6/10
		Day 5	6.5	7.94	12,600	9/10
		Day 6	6.2	7.92	12,800	10/10
		Day 7	6.3	7.74	11,900	
10	1	Day 1	6.4	7.87	14,000	10/10
	2	Day 1	6.5	7.88	13,900	10/10

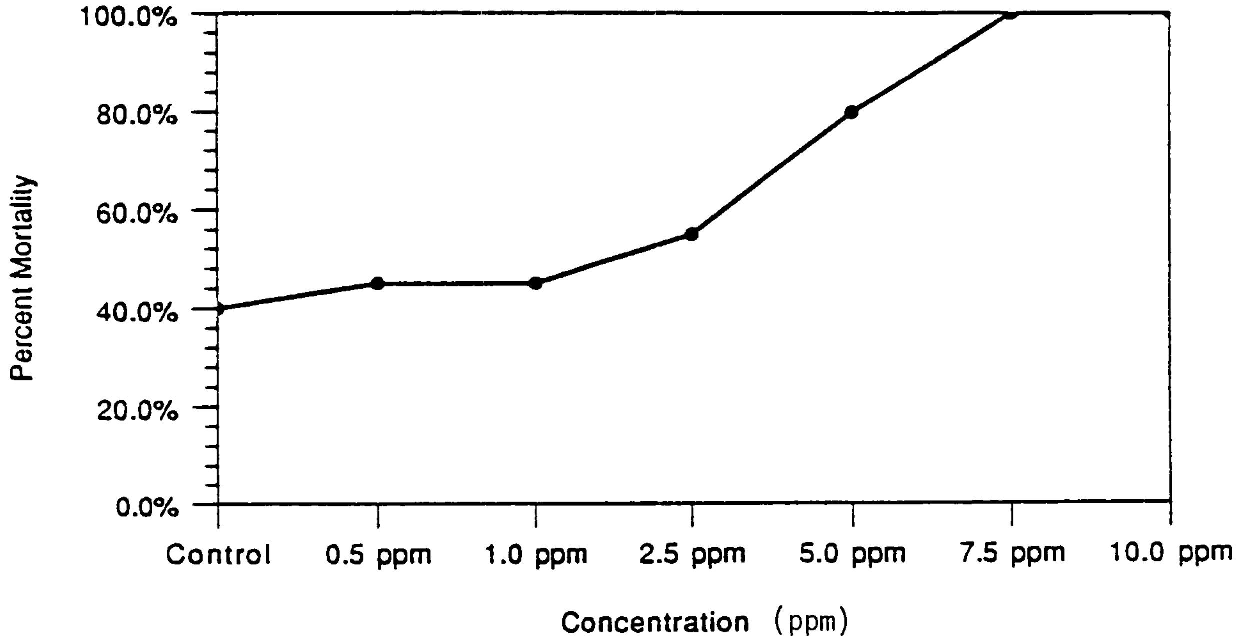


Figure B-8. Results of NaCl chronic toxicity test using fathead minnows expressed in mortality after a 1-week period.

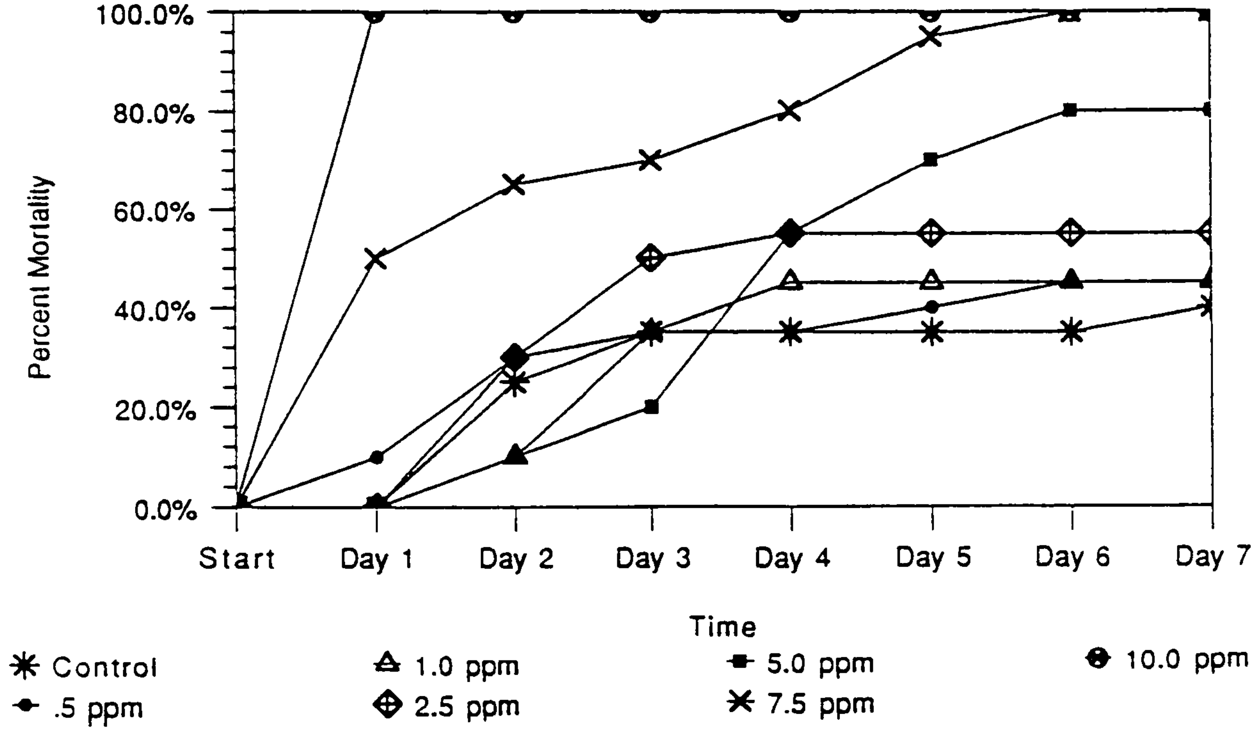


Figure B-9. Results of NaCl chronic toxicity test using fathead minnows expressed in mortality at six concentrations over a 1-week period.

11.8.6 Test Procedures

Two sheets of filter paper (inert and water absorbent) are placed in each test container. Ten seeds are grouped toward the center on the top sheet, but kept separated. Deicer solutions are prepared (as described above for the other tests) and 4 mL is added gently around the edges of each container so as not to dislodge the seeds. The lid is then placed on the dish. A wet paper towel is placed over the dish. The dishes are placed under the same room conditions as described above for the other tests, except no lights are provided. Thus germination is in the dark at 68°F (20°C). The seeds are checked daily to be sure drying is not occurring. If drying has occurred, all can receive another 1 to 2 mL. (Overly wet conditions are also undesirable.) The number of seeds germinated and the radicle length are determined after 5 days.

11.8.7 Test Results and Data (Reference Document EPA 560/6/82/002)

A sample of data obtained using the test is presented in Table B-23 and Figures B-10 and B-11.

Table B-23. Results of seed germination/radicle elongation test using NaCl and soybeans

Deicer concentration (g/L)		Germination (germ./total)	Radicle length (mm)	Average radicle length (mm)
Control	1	10/10	37,40,70,54,52,16,18,62,6,23	37.80
	2	10/10	49,75,32,51,61,38,21,31,13,35	40.60
	3	10/10	20,21,6,8,70,17,87,45,78,30	38.20
3	1	10/10	42,68,17,22,66,28,52,23,17,55	39.00
	2	9/10	15,7,31,15,52,63,56,10,47	32.89
	3	10/10	44,17,49,21,40,40,34,35,32,39	35.10
6	1	9/10	12,26,16,30,20,32,28,29,30	24.78
	2	9/10	41,44,29,34,36,33,24,37,11	32.11
	3	10/10	27,24,31,32,32,37,6,40,40,11	28.00
9	1	7/10	25,7,22,18,15,25,30	20.29
	2	8/10	27,9,9,7,28,7,17,12	14.50
	3	10/10	9,10,10,11,12,17,15,30,10,13	13.70
12	1	9/10	17,10,21,5,21,11,12,17,3	14.80
	2	10/10	20,3,11,5,21,19,6,13,16,20	13.40
	3	8/10	3,4,4,16,6,6,18,19	9.50
15	1	8/10	10,16,5,8,5,6,6,13	8.63
	2	7/10	6,4,11,18,10,11,11	10.14
	3	8/10	3,11,11,8,13,9,14,2	8.88
18	1	7/10	12,6,4,3,6,3,7	5.86
	2	5/10	5,5,9,10,10	7.80
	3	6/10	7,12,12,6,6,4	7.83

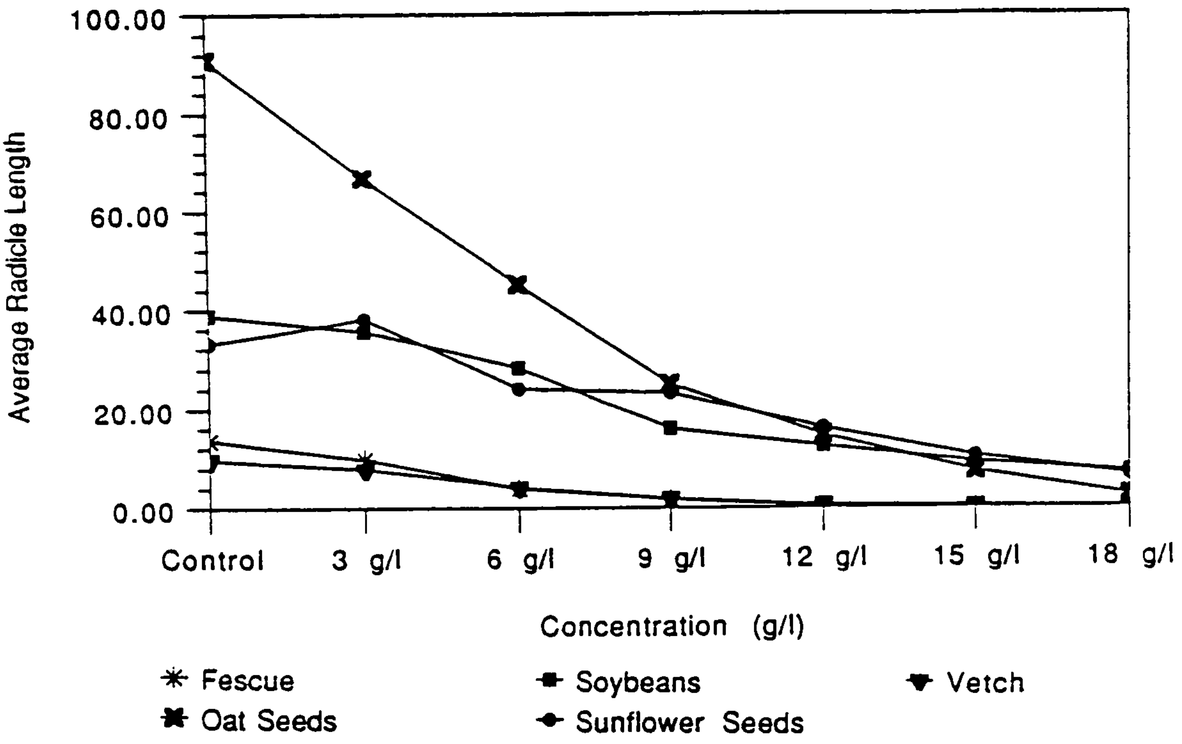


Figure B-10. Results of NaCl/radicle elongation test expressed in average radicle length of germinating seeds for fescue, oat, soybean, sunflower, and vetch seeds.

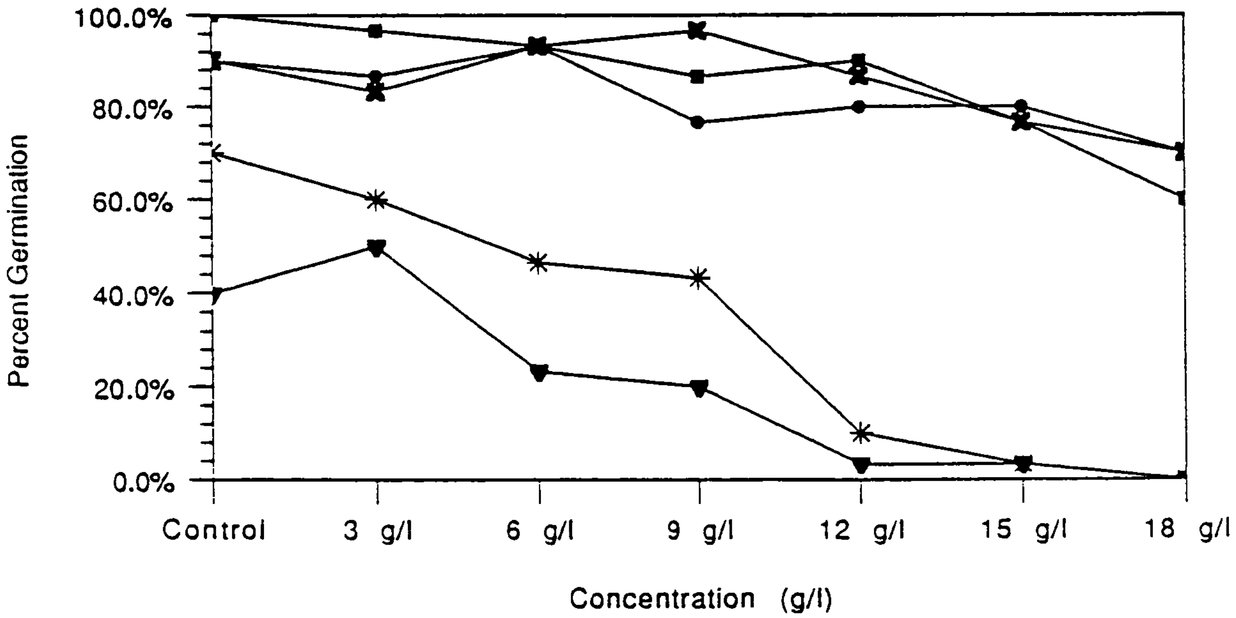


Figure B-11. Results of NaCl/seed germination test expressed in percent germination of fescue, oat, soybean, sunflower, and vetch seeds.

Annex

Design and Construction of a Simple Flow System for Maintaining Test Species in the Laboratory and for Potential Use in Additional Acute and Chronic Toxicity Screening Tests Using Chemical Deicers

Continuous flow systems can provide, particularly for stream organisms, more natural culture or maintenance conditions and may better simulate stream toxicant exposure. Such systems can also enhance the success of maintaining and then testing locally collected organisms more representative of local fauna and conditions, but not available from supply houses.

A detailed design for a simple flow-through system is presented in the two accompanying figures, which provide an appropriate layout to follow for construction. The entire functional unit, as shown in Figures B-12 and B-13, consists of the 3/16-in. (4.5 mm) thick Plexiglas® flow chamber (50 cm x 33.5 cm x 20 cm), subdivided into six individual flow channels (34.5 cm x 5 cm x 15 cm), and a pump inserted in a 3/4-in. flexible plastic tube entering one end of the chamber and exiting from the other. The main structure of the chamber is bonded with Plexiglas® fusing glue, but the walls separating the individual channels are held (not permanently) with silicone adhesive so that certain walls can be removed to form fewer, wider channels to accommodate more or larger organisms. For example, the basic six-unit chamber can be easily altered to three 10.45-cm-wide channels, two 15.9-cm-wide channels, or one 32-cm-wide channel. Flow rate depends on the rate through the pump (adjustable with an adjustable pump) and the hole size (shown as 2.25-cm diameter in second figure) between the common inflow section and the individual channels. Note that these possible adjustments will create a balanced depth in the common inflow section versus the channel depth. This is also influenced (thus flow as well) by the number and size of the holes (six 1/4-in. holes in each in this particular version) in the horizontal flow pipe extending across the width of the common inflow section and common outflow section. The pump used (others are possible) is a Little Giant 29038 Model No. 3-MD-SC (Tecumseh Products Company, Tulsa, Oklahoma). It is advisable to use a pump which employs a magnetic coupling between the motor and the impeller pumping the water that eliminates the conventional shaft seal (generally containing oil) and the possibility of leakage and contamination of the water with oil or other lubricant. A wide range of flow rates through the channels is possible depending on the adjustments already discussed, within the range of a few centimeters per second to over 50 cm/sec.

Larger systems also operate within this range which is an acceptable range for the purposes of this system discussed above.

In operating this system, in one chamber with its six (3, 2, or 1) channels, the same water treatment is experienced by each channel, thus they serve as replicates. Different treatments are provided with other duplicate chambers.

Temperature control of the system is generally desirable, particularly because pumps themselves may heat the water to various extents depending on the type of pump. One way to accomplish temperature control is to operate the tanks in a controlled temperature chamber of walk-in size or double-wide (approximately double-wide refrigerator size) reach-in size. Pumps can be placed on the outside of the growth chamber to reduce the heat load. Another way to cool, and perhaps one more readily available to some highway departments, is to insert a cooler of some sort in the flow line between the pump and the chamber on either side of the pump. The cooler should not come in direct contact (e.g., test water flowing directly over cooling coils) with the test water because of possible contamination.

One relatively simple method of cooling is to create a long coil of the 3/4-in. tubing on one side of the pump to be immersed into a container (20 to 40 gal, insulated) of ice water. By keeping a steady enough input of ice into the chamber so that its temperature remains constant at about 32°F (0°C) and then adjusting the length of the coil (thus contact with the coolant), the temperature of the channel water can be adjusted, given the relatively constant heat input from the pump. The chambers should be housed in a room of relatively constant temperature. A more easily adjusted cooling system, but more expensive and mechanically complex, is possible using a thermostatically regulated cooler with its coil immersed in the cooling container. Heavy-duty coolers of this type are used for cooling large fish boxes, particularly in commercial fish rearing operations. Suitable coolers are available from Frigid Units, Inc. (3214 Sylvania Avenue, Toledo, Ohio).

The operation of this flow system with locally collected insects (mostly mayflies, damselflies, and dragonflies) and fish (bluegill and various minnows and darters) was successful at maintaining these organisms for several weeks. Considering the maximum size of the organisms used in such a system, fish up to 4 cm in length adapted well to the smallest channels and larger fish to the wider channels, 6 cm long, 8 cm long, and 10 cm long in the three successively wider channels. The species of fish and insects, considering individual behavior and other requirements, is an important determining factor for the success of the system. Considerations such as food provided and substrate (most often gravel bottom, 1- to 2-cm depth of 5- to 8-mm diameter stones), including larger stones for refuge, are also important to suit the needs of particular species. For any species selected for testing, trial maintenance in the system is necessary before conducting a test with a chemical. This system can also be used to maintain organisms for a few days before use in static tests like those described in Sections 11.6 and 11.7 when aquarium maintenance is not as suitable. When attempting to adapt this flow system to the flow-through tests described by EPA in several of the documents cited in this report, it is important to carefully consider the requirements of each test to determine if they are adaptable to this system and still within the requirements set by EPA.

Schematic drawings of the flow-through system are presented in Figures B-12 and B-13.

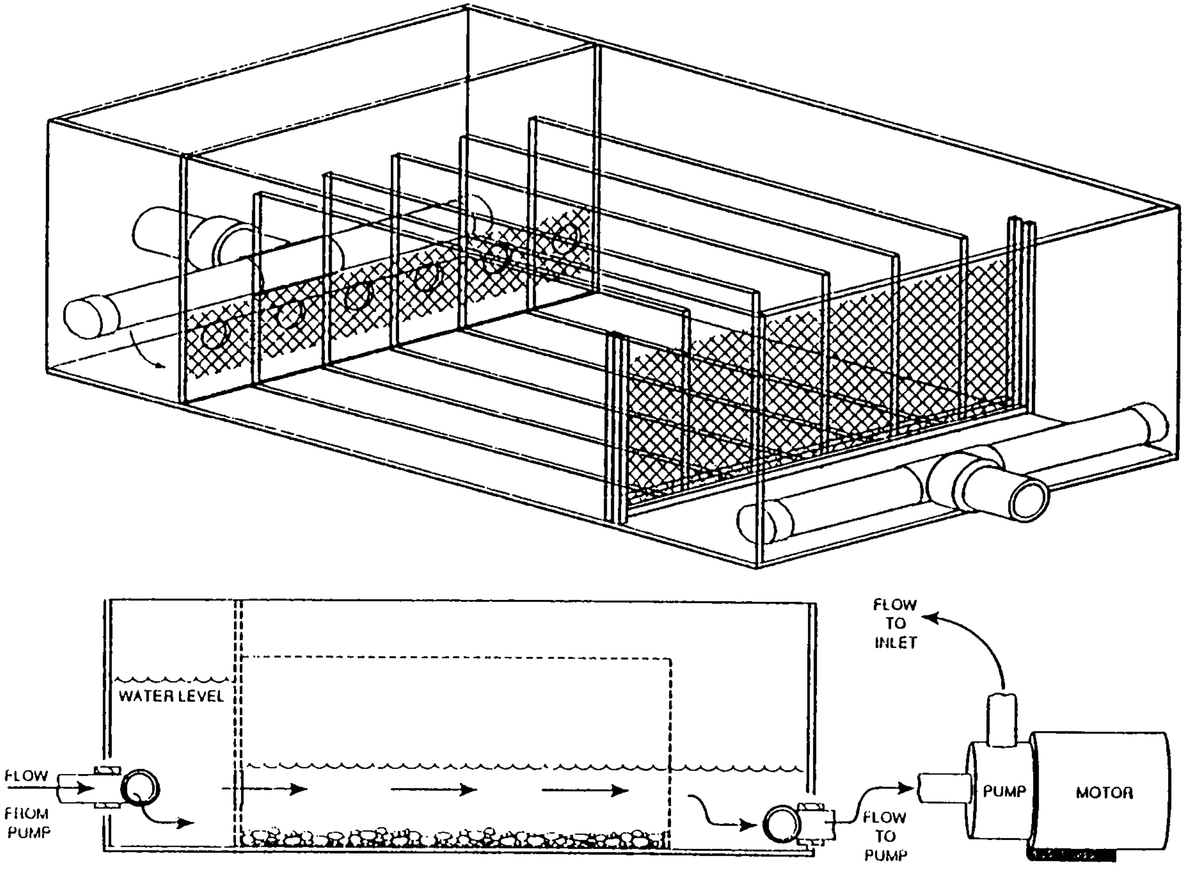


Figure B-12. Overview of the flow-through system.

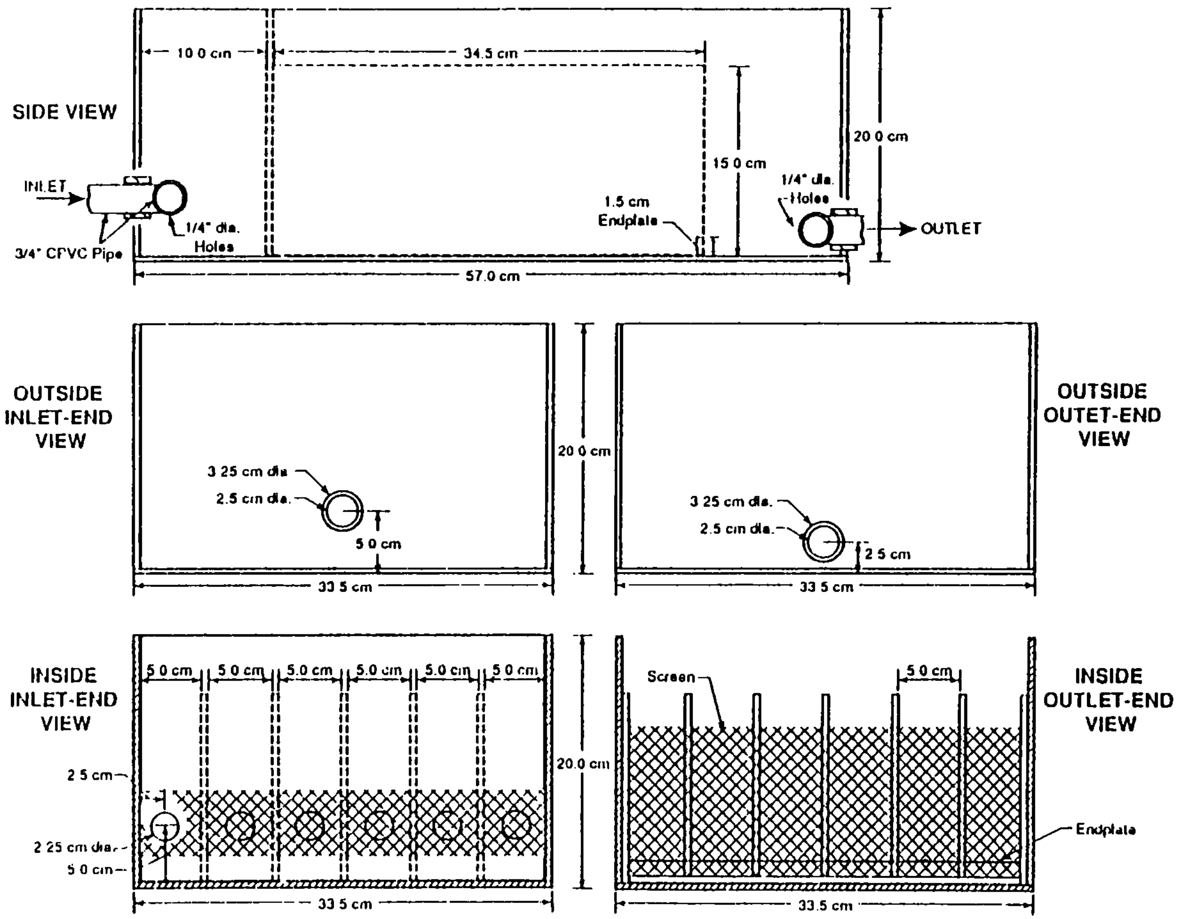


Figure B-13. Multiview of flow-through system.

SHRP H-205.12

Test Method for Evaluation of Corrosive Effects of Deicing Chemicals on Steel Reinforcement in Concrete

12.1 Scope

1. This test method is used to evaluate the effect of deicing chemical formulations on concrete reinforcement corrosion. The test method is based upon the ASTM G-109 method which has been developed recently for evaluating corrosion inhibiting admixtures.

2. A macrocell corrosion measurement technique is described in this test specification.

3. This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

12.2 Reference ASTM Standard Documents

• C 33-90	Specifications for Concrete Aggregates
• C 143-78	Test Method for Slump of Portland Cement Concrete

• C 173-78	Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method
• C 192-90a	Test Method for Making and Curing Concrete Test Specimens in the Laboratory
• C 231-90	Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method
• C 511-85	Standard Specifications for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
• C 876-91	Half-Cell Potential of Uncoated Reinforcing Steel in Concrete
• E 177-90	Standard Recommended Practice for Use of Terms "Precision" and "Accuracy" as Applied to Measurement of a Property of a Material
• G 109-92	Test Method for Determining the Effects of Chemical Admixtures on the Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments
• ASTM Research Report No. RR:G01-1009	Supporting Data for G 109-92, Test for Determining the Effects of Chemical Admixtures on the Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments, January 27, 1992

12.3 Method Summary

The test procedure involves measuring the macrocell current flowing between two bare steel rebar mats embedded in a concrete specimen and connected externally with a 100-Ω resistor. Specimens are subjected to alternating 2-week cycles of ponding with selected deicer test solutions and drying. IR drop across the 100-Ω resistor is measured at the beginning of the second week of each ponding cycle to monitor the corrosion rate of the rebar. Potential vs. a reference electrode may also be measured to determine the corrosive state of the rebar.

12.4 Significance and Use

1. This test method provides a simple straightforward procedure to evaluate the corrosive effects of deicing chemicals on reinforcing steel embedded in concrete.

2. This test method can be used to evaluate deicing chemicals containing corrosion inhibiting substances.

3. This test method can be used to evaluate the effect of admixtures and corrosion inhibiting admixtures in a chloride environment.

12.5 Test Specimens

Concrete test specimens are made according to ASTM C 192-90a using the same source of materials. Concrete should be portioned in accordance with ACI 211.1 and meeting the following parameters: 600 ± 5 lb/yd^3 of Type 1 or Type 2 cement; 0.5 ± 0.01 W/C ratio (ssd aggregates), and 6 ± 1 percent plastic air (ASTM C 231, C 173). The minimum slump shall be 2 in. (ASTM C 143-78), but W/C ratio shall not exceed 0.51. Top size aggregate shall be 1/2 in. (1.25 cm) maximum and conform to ASTM C 33-90. When admixtures are tested they shall be added to manufacturer's recommended dosages and a water reducer is allowed if needed to achieve the desired slump.

Test specimen size (length x height x width) is to be 11 x 6 x 4.5 in. (27.9 x 15.2 x 11.4 cm). Two deformed No. 4, 0.5 in. (1.27 cm) diameter steel reinforcing bars 14-in. long, are to be placed 1 in. (2.54 cm) from the bottom, and one bar is to be placed 0.75 in. (1.9 cm) from the top, as shown in Figure B-14. Other metal bars can be used if data are desired for nonsteel embedment. A chemical analysis of the metal bars used in preparing the test specimens should be obtained and reported.

All steel reinforcing bars shall be from the same heat, shall be pickled in sulfuric acid, washed, dried and then wire brushed. Metal bars are to be taped with electroplater's tape (such as 3M product No. 470) and tightly wrapped so that 1.5 in. (3.8 cm) of the bar are protected within each exit end from the concrete (to eliminate edge effects). This will expose the middle 8 in. (20.3 cm) of the steel within the concrete. Bars will be placed with the forming ridge horizontal. Furthermore, the ends of the bars protruding from the concrete specimens shall be protected to prevent rusting during curing.

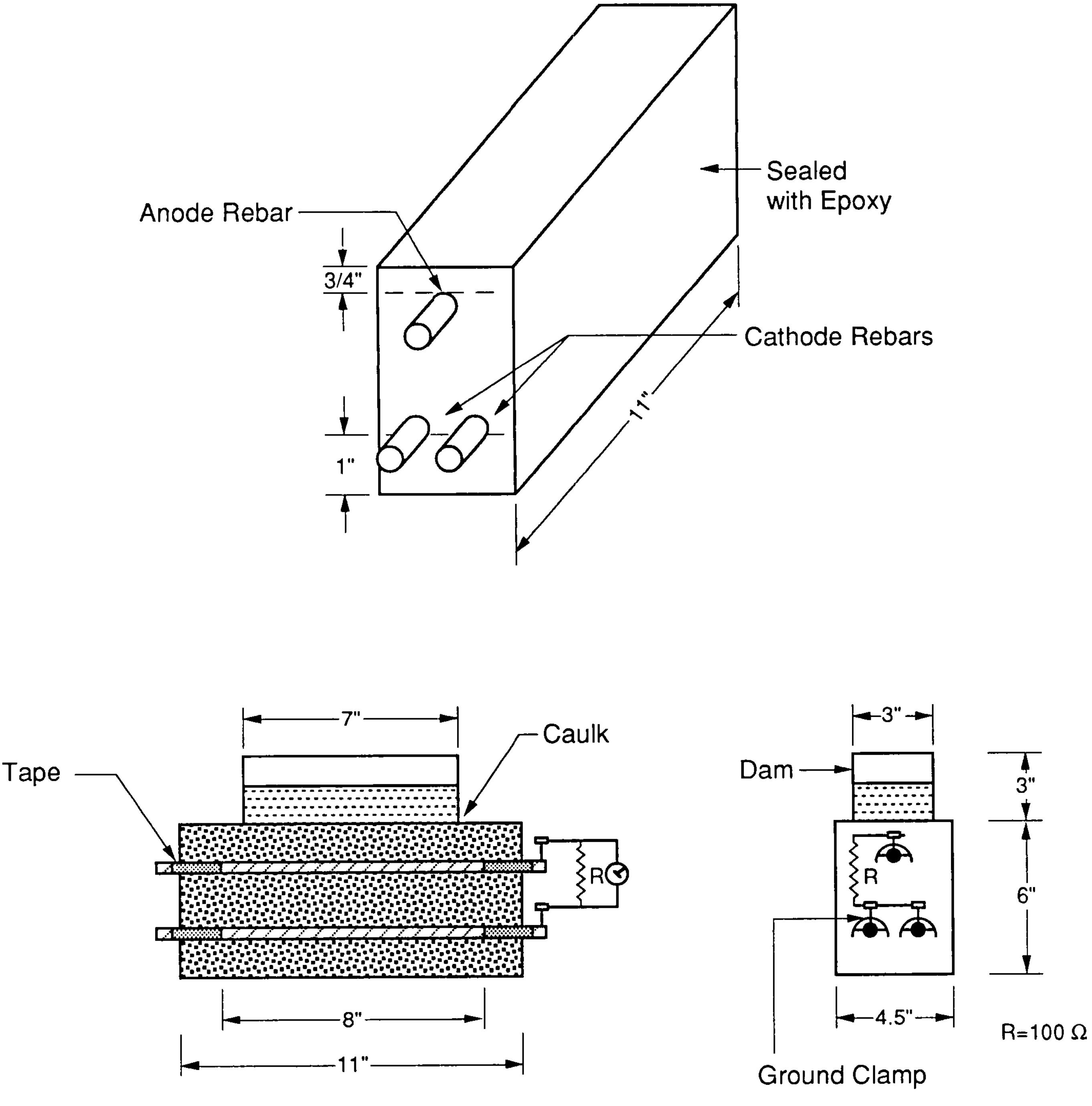


Figure B-14. Test specimen—macrocell configuration.

Samples shall be consolidated according to ASTM C 192-90a. A wood float finish will be applied. After removal from the forms, samples shall be cured for 28 days in the fog room according to ASTM C 192-90a and C 511-85.

Upon removal from the fog room samples shall be wire brushed on the concrete surface to be exposed (wood floated surface). The four vertical sides shall be sealed with a concrete epoxy sealer according to manufacturer's recommendations. A 3-in. (7.6-cm) minimum height dam shall be placed on the sample as shown in Figure B-14. The dam shall be 3 in. ID (7.6 cm) wide and be about 1/2 in. (1.25 cm) from each side and 7 in. ID (17.8 cm) long such that it does not extend over taped sections of the bars (Figure B-14). Plastic wall thickness shall be 1/8 ± 1/32 in. (0.32 ± 0.08 cm). A silicone caulk shall be used to seal the dam from the outside and epoxy sealer shall be applied to the top surface outside of the dam.

Ground clamps are then attached to each bar as shown in Figure B-14 and the two bottom bars are attached (shorted) with a grounding wire. A 100-Ω resistor (same for all samples) is placed between the bottom bars and top bars through the ground connectors.

Three test specimens shall be prepared for each chemical deicer formulation and test condition evaluated.

12.6 Test Procedures

Test specimens are to be supported so that air flows underneath. They are ponded for 2 weeks, at 22° ± 3°C, with the deicer test solution (approximately 400 mL) at the depth of 1.5 ± 0.1 in. (3.8 ± 0.3 cm). A loose-fitting plastic cover should be used to minimize evaporation. After 2 weeks, the solution is vacuumed off and the samples remain dry for 2 weeks. The voltage across the resistor is measured at the beginning of the second week of ponding using a voltmeter with a minimum of 1 MΩ input impedance. The galvanic current is the measured voltage divided by the resistance. Note that with the common on the bottom bar, negative voltages correspond to positive galvanic current (i.e., top bar is the anode). The current is monitored as a function of time (cycles) until such point that a clear difference is noted between the reference and deicer test solution and/or admixed concretes or until one set shows visible signs of corrosion. At the conclusion of testing, the test specimens shall be broken to note rebar appearance.

The macrocell current is measured at monthly intervals for each replicate test specimen. Three replicate test specimens shall be prepared and tested for every deicing chemical formulation and/or concrete admixture evaluated.

An example of the variation of the macrocell current with time is presented in Figure B-15 for four replicate test specimens ponded with 3 weight percent sodium chloride solutions. The average macrocell current vs. time of the same four replicates ponded with 3 weight percent sodium chloride solutions is shown in Figure B-16. A plot of the calculated total corrosion for the same test specimens ponded with 3 weight percent sodium chloride solution is shown in Figure B-17.

12.7 Calculation of Total Corrosion

The total integrated current (total corrosion) is calculated according to the following equation:

$$TC_j = TC_{j-1} + [(t_j - t_{j-1}) * (i_j + i_{j-i})/2]$$

where: TC = Total corrosion (coulombs).
t_j = Time (seconds) at which measurement of the macrocell current is carried out.
i_j = Macrocell current (amperes) at time t_j.

An example of the total corrosion calculation is given as follows:

Assume the following readings were obtained over a 90-day period of time.

Days	0	30	60	90
i_{mac} (μA)	0	20	27	35

At the end of the first 30-day period the total corrosion is:

$$TC_1 = 0 + [(30-0) * 86400 * (20+0)/2 * 10^{-6}] = 25.92 \text{ coulombs}$$

at the end of the 60-day period:

$$TC_2 = 25.92 + [(60-30) * 86400 * (20+27)/2*10^{-6}] = 86.83 \text{ coulombs}$$

At the end of the 90-day period:

$$TC_3 = 86.83 + [(90-60) * 86400 * (27+35)/2*10^{-6}] = 167.18 \text{ coulombs}$$

NOTE: Conversion factor from days to seconds = 24 x 60 x 60 = 86400

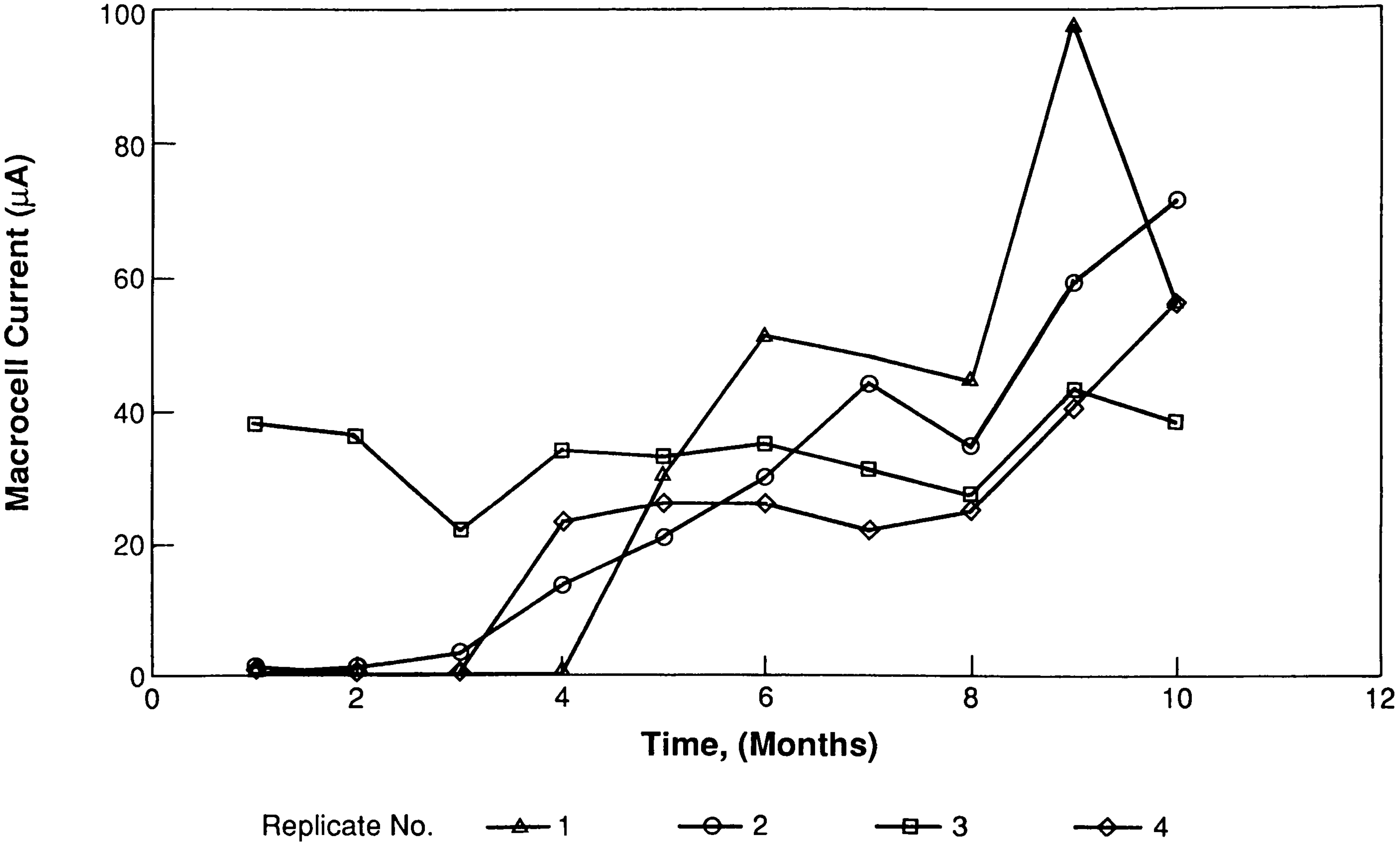


Figure B-15. Macrocell current vs. time for test specimens ponded with 3 weight percent NaCl solution.

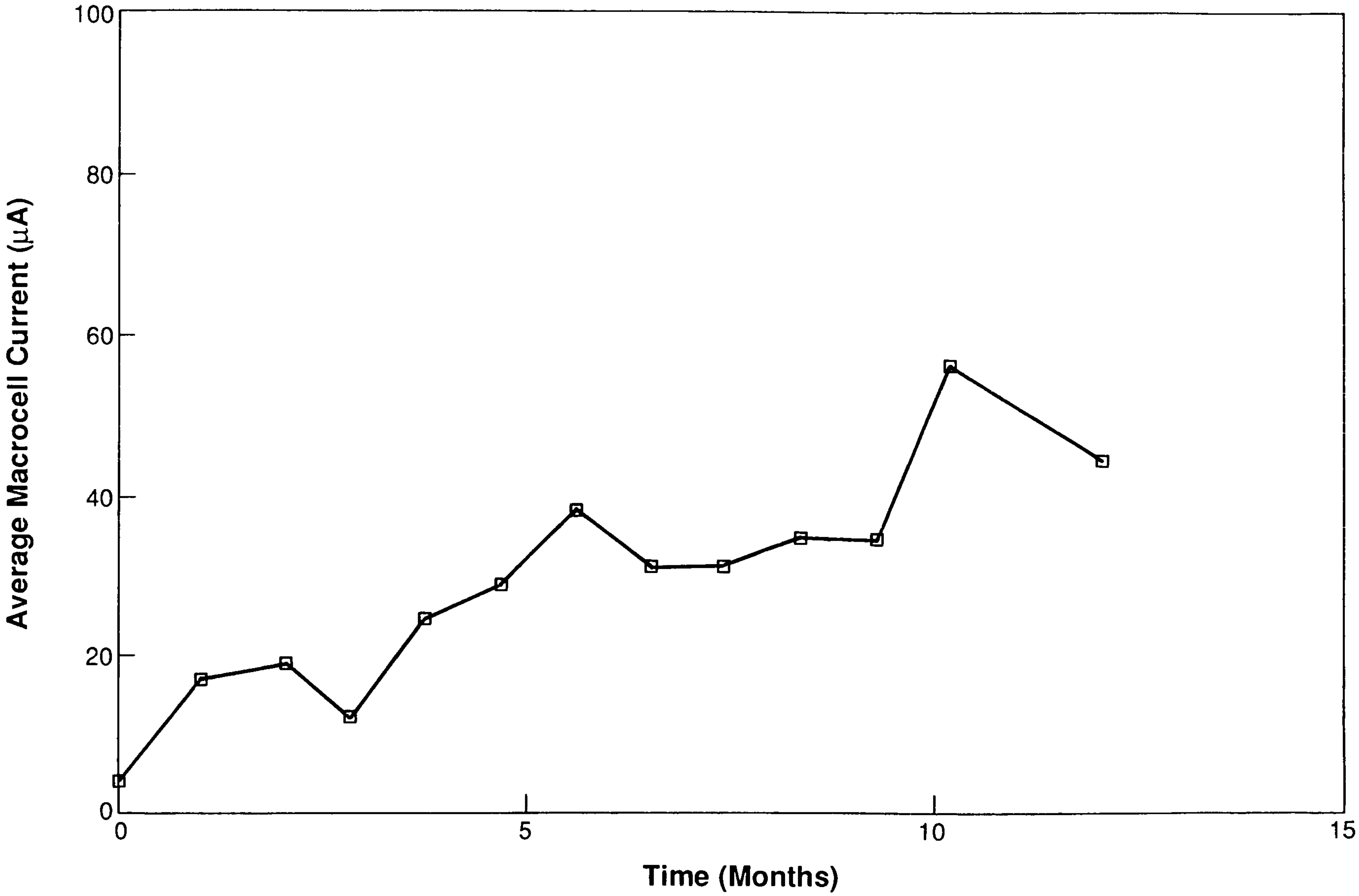


Figure B-16. Average macrocell current vs. time for four test specimens ponded with 3 weight percent NaCl solution.

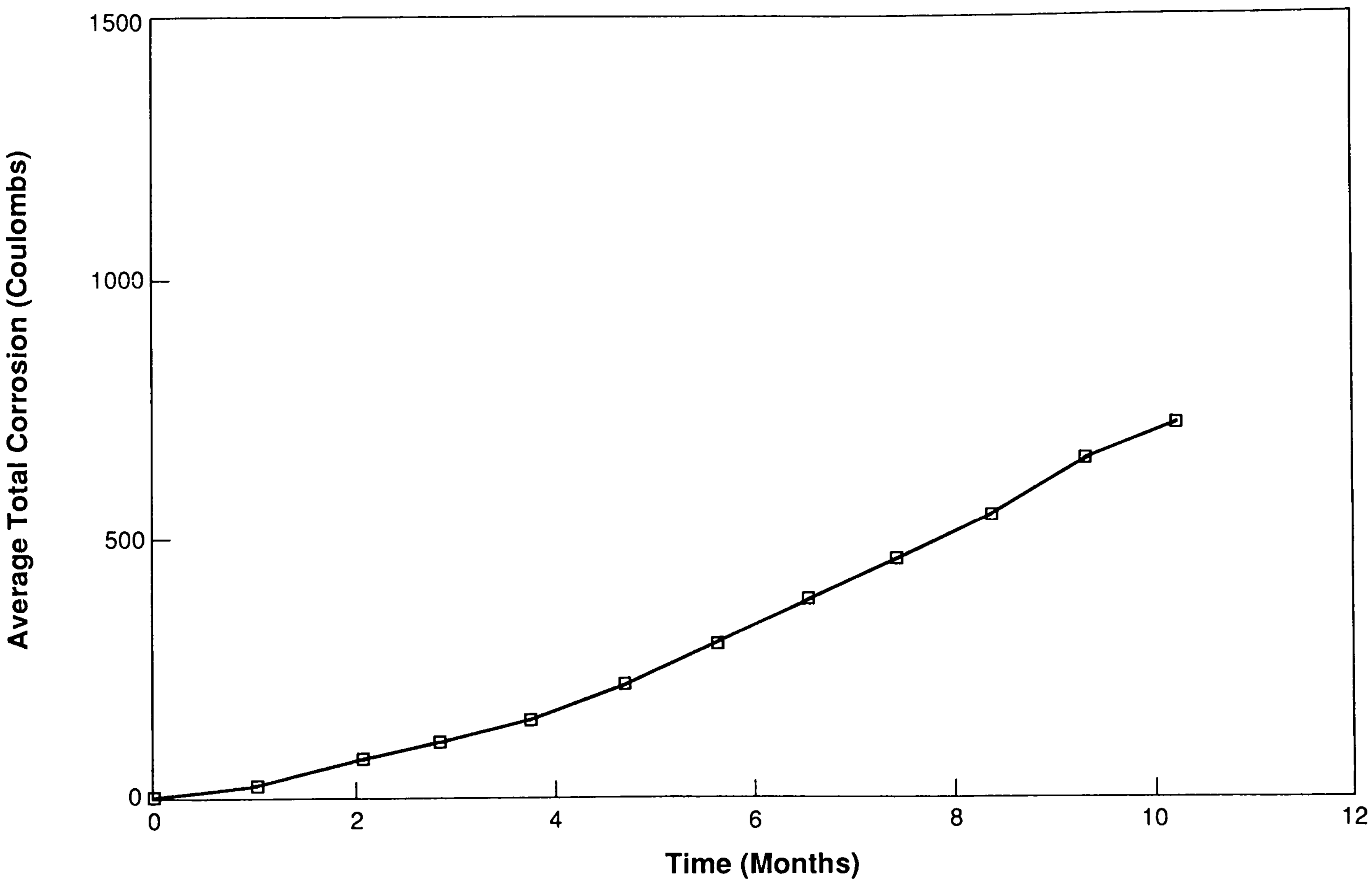


Figure B-17. Variation of average total corrosion with time for four test specimens ponded with 3 weight percent NaCl solution.

12.8 Statistical Analysis of Data

The statistical analyses of the measured macrocell current was carried out following Practice E 177-90. The following parameters are calculated for each time period and each specimen type from the raw data:

AVG = average of the macrocell current for three replicates

$$s = \text{standard deviation} \sqrt{\sum_{1}^{n} (x - AVG)^2/(n-1)}$$

where:
x = individual values of the macrocell current
n = number of replicates

12.9 Testing Guidelines and Recommendations

1. The standard deviation of the average of the measured macrocell current increases with increased average macrocell current.

2. The macrocell current is less than 1 μA for noncorroding specimens, and more than 9 μA after the onset of corrosion. The observed rapid increase in the macrocell current when corrosion occurs can usefully serve to establish a "time to failure" criterion.

3. The time to failure criterion is the time at which the average macrocell current for a given deicer solution exceeds 10 μA and at least half the samples show macrocell currents greater than 10 μA.

4. An interlaboratory study has revealed that after 6 months of testing, 88 percent of the test specimens ponded with 3 weight percent sodium chloride solutions had failed.

5. The test should be run until the control samples (ponded with 3 weight percent NaCl solution) have an average macrocell current of 10 μA, and at least half the control samples have macrocell current greater than 10 μA.

6. The tests are then continued for a further three cycles. At the end of this period, if the deicer formulation or the concrete admixture being tested functions as an inhibitor, the macrocell current should be less than 1 μA.

Appendix C

Supplementary Test Methods for Evaluating Chemical Deicers

ASTM B 117-90	Standard Method of Salt Spray (Fog) Testing
ASTM C 39-86	Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens
ASTM C 42-90	Method of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
ASTM C 136-84a	Sieve Analysis of Fuel and Coarse Aggregates
ASTM C 143-90a	Test Method for Slump of Portland Cement Concrete
ASTM C 192-90a	Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory
ASTM C 672-91	Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals
ASTM C 702-87	Standard Practice for Reducing Field Samples of Aggregate to Testing Size
ASTM C 876-91	Standard Test Method for Half-Cell Potentials of Uncoated Reinforcing Steel in Concrete
ASTM C 944-90a	Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating Cutter Method
ASTM D 75-87	Standard Practice for Sampling Aggregates
ASTM D 412-87	Standard Test Methods for Rubber Properties in Tension
ASTM D 445-88	Standard Test Methods for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
ASTM D 632-84	Specification for Sodium Chloride

ASTM D 638-90	Standard Test Method for Tensile Properties of Plastics
ASTM D 1122-90a	Standard Test Method for Specific Gravity of Engine Coolants by the Hydrometer
ASTM D 1177-88	Standard Test Method for Freezing Point of Aqueous Engine Coolant Solution
ASTM D 1186-87	Standard Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base
ASTM D 1568-63	Standard Methods for Sampling and Chemical Analysis of Alkylbenzene Sulfonates
ASTM D 1654-79	Standard Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments
ASTM D 1895-89	Standard Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials
ASTM D 2013-86	Standard Method of Preparing Coal Samples for Analysis
ASTM D 3177-89	Standard Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke
ASTM D 3178-89	Standard Test Methods for Carbon and Hydrogen in the Analysis Sample of Coal and Coke
ASTM D 3179-89	Standard Test Methods for Nitrogen in the Analysis Sample of Coal and Coke
ASTM D 3201-86	Standard Test Method for Hygroscopic Properties of Fire-Retardant Wood and Wood-Based Products
ASTM D 3665-82	Standard Practice for Random Sampling of Construction Materials

ASTM D 3682-87	Standard Test Method for Major and Minor Elements in Coal and Coke Ash by Atomic Absorption
ANSI/ASTM D 3683-78	Standard Test Method for Trace Elements in Coal and Coke Ash by Atomic Absorption
ANSI/ASTM D 3684-78	Standard Test Method for Total Mercury in Coal by the Oxygen Bomb Combustion/Atomic Absorption Method
ASTM D 4191-82	Standard Test Method for Sodium in Water by Atomic Absorption Spectrophotometry
ASTM D 4331-84	Test Method for Effectiveness of Dedusting Agents for Powdered Chemicals
ASTM E 70-90	Standard Test Method for pH of Aqueous Solutions with the Glass Electrode
ASTM E 104-85	Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions
ASTM E 303-83	Measuring Surface Frictional Properties Using the British Pendulum Tester
ASTM E 449-90	Method for Analysis of Calcium Chloride
ASTM E 534-86	Standard Test Methods for Chemical Analysis of Sodium Chloride
ASTM E 729-88a	Standard Practice for Conducting Acute Toxicity Tests With Fishes, Macroinvertebrates, and Amphibians
ASTM E 943-91b	Standard Definitions of Terms Relating to Biological Effects and Environmental Fate
ASTM E 978-84	Standard Practice for Evaluating Environmental Fate Models of Chemicals

ASTM E 1197-87	Standard Guide for Conducting a Terrestrial Soil-Core Microcosm Test
ASTM F 502-83	Standard Test Method for Effects of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces
ASTM G 1-90	Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
ASTM G 31-72	Standard Practice for Laboratory Immersion Corrosion Testing of Metals
ASTM G 46-76	Standard Practice for Examination and Evaluation of Pitting Corrosion
ASTM G 109-92	Test Method for Determining the Effects of Chemical Admixtures on the Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments
ASTM STP 920	Community Toxicity Testing
EPA/223-802352	Standardized Aquatic Microcosm Protocol
EPA/560/5-75/008	Test Methods for Assessing the Effects of Chemicals on Plants
EPA/600/14-84/017	Method 300.0, Ion Chromatography of Chloride, Fluoride, Nitrate, Nitrite, Phosphate, and Sulfate Ions
EPA/600/4-85/013	Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms
EPA/600/4-85/014	Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms
SAE/AMS-1425B	Deicing Fluid, Aircraft; Ethylene Glycol Base
SAE/AMS-1426B	Fluid, Deicing/Anti-Icing, Runways and Taxiways; Glycol Base
SAE/AMS-1427A	Deicing/Anti-Icing Fluid, Aircraft: Propylene Glycol Base

SAE/AMS-1430A	Urea Compound, Shotted
SAE/AMS-1431A	Compound, Solid Deicing/Anti-Icing: Runways and Taxiways