

Automatic Freezing-and-Thawing Equipment For a Small Laboratory

WILLIAM A. CORDON, Associate Professor of Civil Engineering, Engineering Experiment Station, Utah State University, Logan

● **CONCRETE DURABILITY** is important in establishing performance of concrete in northern climates. Resistance to disintegration when subjected to alternate cycles of freezing and thawing is an accepted measure of durability.

Automatic freezing-and-thawing equipment in the laboratory provides a valuable tool in determining the performance and acceptance of proposed materials. Correlation between concrete strength and durability tests provides a means of establishing field control.

The cost and complicated nature of freezing-and-thawing equipment which automatically produces alternate cycles of freezing and thawing has discouraged extensive use of this test. Hand methods of placing specimens in freezing cabinets and thawing tanks are also costly and laborious. Hence, freezing-and-thawing tests are generally limited to research and performance tests in the larger laboratories.

Comparatively inexpensive automatic freezing-and-thawing equipment has been developed by the Engineering Experiment Station, Utah State University. This equipment produces up to 9 cycles of freezing and thawing in each 24-hr period. The cost of the equipment may be within the budget of most small laboratories.

This paper discusses the details of construction of the automatic freezing-and-thawing equipment and also presents typical test results obtained with the development model of this equipment.

AUTOMATIC FREEZING-AND-THAWING EQUIPMENT

Figure 1 shows a general view of the apparatus. The cabinet is 6 ft 10 in. by 2 ft 10 in. wide by 10 in. deep. All sides have 4 in. of insulation, including a 4-in. insulated lid. The freezing compartment is only 6 in. deep, 2 in. wide, and 6 ft 2 in. long.

Temperatures to 0 F are obtained during the freezing cycle by means of a 2- by 6-ft commercial cooling plate attached to a $\frac{1}{4}$ -horse-power commercial compressor (Fig. 2). Thawing is accomplished with electric resistance heaters rated at 500 watt, 220 v, placed along the sides of the containers and connected in parallel to a 110-v circuit.

Specimen containers are soldered copper trays. The soldered joints were cause for concern since it was feared that the alternate contraction and expansion of the ice during the freezing-and-thawing cycle would break the soldered joint. This was prevented, however, by making the copper container only $\frac{1}{4}$ in. wider than the concrete prism, leaving only $\frac{1}{8}$ in. of water on each side. The total expansion of this water was not significant. After more than one year's service, there has been no difficulty experienced with leaking or breaking the soldered joints. Specimens are blocked up $\frac{1}{8}$ in. to prevent direct contact between the metal container and the specimen.

The equipment does not limit the size or shape of specimens, since containers may be built to accommodate various specimen sizes. Two- by 2- by 10-in. prisms and 3- by 3- by 16-in. prisms have been tested successfully.

The length of cycle depends upon the time required to reduce the temperature at the center of the control prism (Fig. 3) from 40 to 0 F and back to 40 F. The length of a complete cycle averages about 2.7 hr. The freezing-and-thawing cycle is controlled by means of a thermostat placed in the center of a control prism and relays which start the compressor and turn off the heater at 40 F and reverse the procedure at 0 F. A gas-charged thermometer bulb and a recording thermometer keep a continuous record of the temperature at the center of the control specimen. It is possible to adjust the cycle at any time to take care of variable conditions.

The most unique features of the automatic freezing-and-thawing apparatus are undoubtedly its efficiency and simplicity. Rapid freezing of the specimens is accomplished through metal-to-metal contact between the cooling plate and the metal containers. Freezing progresses from three sides of the container to the center of the specimen. There is only a space of 6 in. of air above the specimens which is rapidly cooled and provides freezing conditions on the fourth side. The total amount of water used in the equipment is very small and, therefore, requires a minimum of heat exchange to freeze and thaw. For these reasons, $\frac{1}{2}$ -hp commercial compressor is adequate to provide the required refrigeration to freeze the specimens in accordance with ASTM specifications.

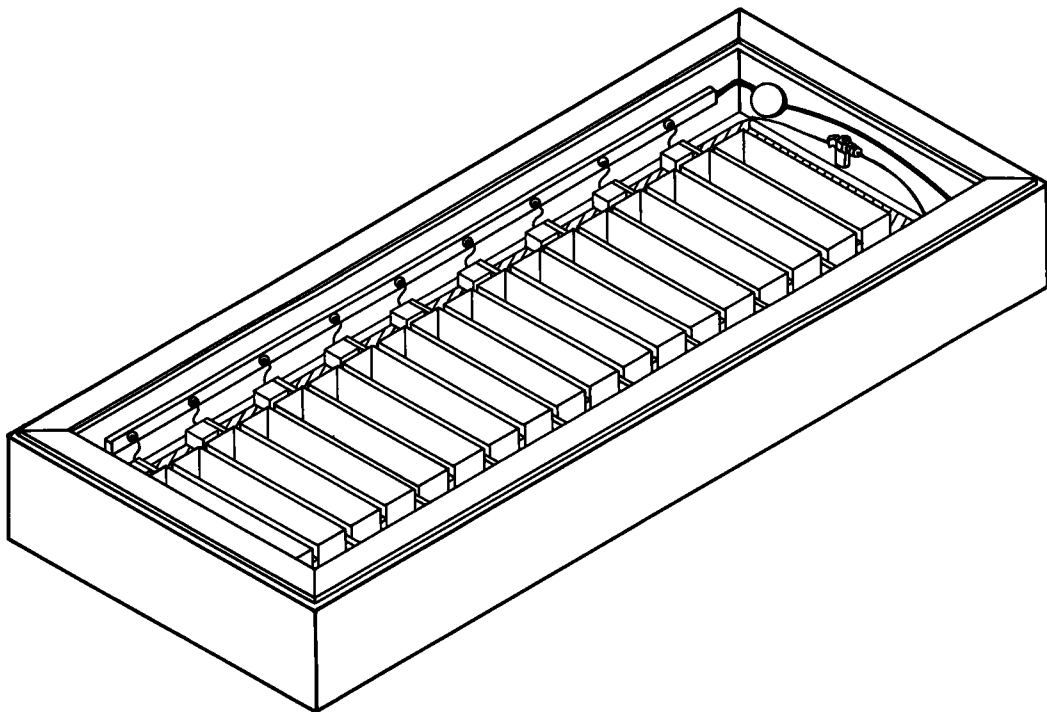
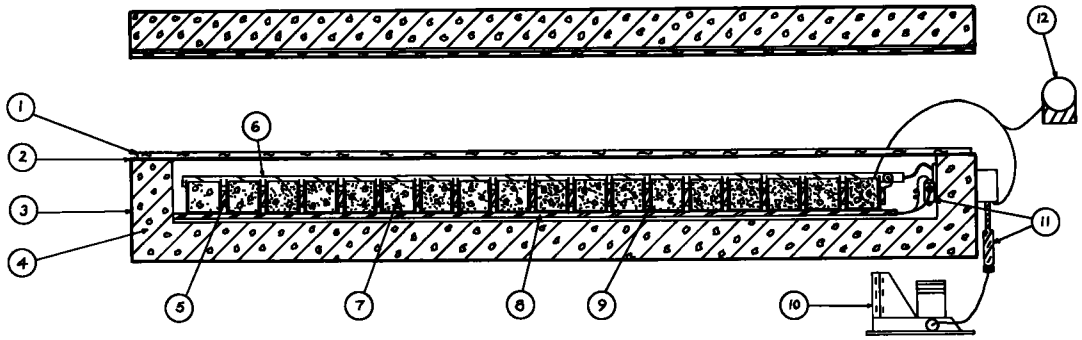


Figure 1. Automatic freezing-and-thawing equipment.

The thawing cycle is also efficient. Strip heaters are placed in direct contact with the sides of the metal containers. Here again, heat transfer is obtained with metal-to-metal contact. The ice surrounding the specimen is melted before the concrete is thawed. Thawing takes place directly from the sides and bottom of the metal container. The cooling plate itself becomes warm during thawing action. The amount of heat developed during the thawing cycle can be controlled by controlling the voltage to provide any desired rate of thawing. The simplicity of the apparatus provides flexibility of both thawing and freezing and undoubtedly will accommodate most requirements.

The specimens are rotated at the completion of each 50 cycles. At this time the water surrounding the specimens is changed. There is no assigned pattern for locating the specimens in the cabinet. Specimens are replaced at random after completion of each 50 cycles of freezing and thawing.

A typical graph of the recording thermometer during one 24-hr period is shown in Figure 4. This graph illustrates the variation in temperature with time for the freezing cycle and also the thawing cycle. Excellent uniformity among cycles of freezing and thawing at different times during the day is indicated. It is interesting to note that



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|-------------------------|-------------------------------|-----------------------------------|
| 1 - RUBBER SEAL | 5 - HEATING UNITS | 9 - SPECIMEN CONTAINER |
| 2 - MASONITE | 6 - ELECTRICAL OUTLET BAR | 10 - COMPRESSOR |
| 3 - SHEET METAL | 7 - CONCRETE SPECIMEN 3x3x16" | 11 - COOLING SYSTEM |
| 4 - INSULATION MATERIAL | 8 - COOLING PLATE | 12 - TEMPERATURE RECORDING DEVICE |

Figure 2. Sectional view of the automatic freezing-and-thawing equipment.

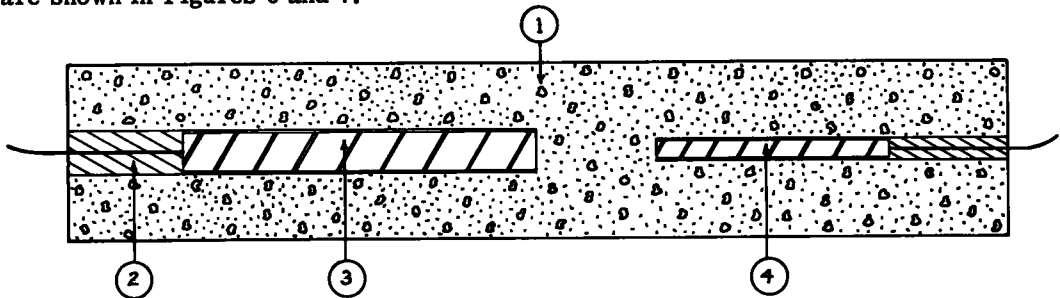
the recording thermometer faithfully records the pause in the cycle at 32 F during the time that ice is being melted and also the time the water must be frozen before the drop in temperature continues.

The uniformity of the temperature throughout the cabinet is shown in Figure 5. The time temperature curve is close throughout most of the cycle, and the only significant difference being at the end of the thawing and beginning of the freezing cycle.

Investigation showed that the strip beaters did not fit snug against the metal container in all cases. This has been corrected by holding the beaters against the containers with stainless steel clips. The probes were also placed in the containers in water, one near the surface and the other two near the bottom. The temperature time curve above freezing was, therefore, influenced by the rate and amount of ice melted and does not necessarily represent the temperature of the specimen.

RESULTS OF TESTS WITH FREEZING-AND-THAWING EQUIPMENT

In the final analysis, the value of testing equipment is determined by the results of tests performed with the equipment. Automatic freezing-and-thawing equipment should differentiate between the durability of various concretes. The results of various tests are shown in Figures 6 and 7.



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|--------------------------------|
| 1 - CONCRETE SPECIMEN 3x3x16" |
| 2 - WAX SEAL |
| 3 - THERMOSTAT BULB |
| 4 - RECORDING THERMOMETER BULB |

Figure 3. Automatic freezing-and-thawing control specimen.

The comparable durability of concrete with and without pozzolanic materials is illustrated in Figure 5. Because the purpose of these tests was to determine the relative durability of concrete containing each of the various pozzolans and to differentiate between the concretes containing pozzolans and a control mix with no pozzolan, there were no air-entraining agents used. Without entrained air one would expect the deterioration of concrete due to freezing and thawing to be rather rapid. Rapid deterioration was indicated from these tests which showed a 25 percent weight loss occurring at approximately 80 cycles in concrete containing pumice and about 150 cycles in concrete without pozzolan. The test adequately differentiated between the different types of concrete. In the lower part of the figure the same tests are presented showing the drop in dynamic modulus of elasticity. In this case, failure occurred at a minimum of approximately 40 cycles with fly-ash and a maximum of 90 cycles in concrete in which pozzolans were not used. These tests are considered typical of the durability of concrete before air-entraining agents were generally used.

The durability of concrete containing adequate air entrainment is illustrated in Figure 6 and Table 1. The purpose of these tests was to differentiate between the dur-

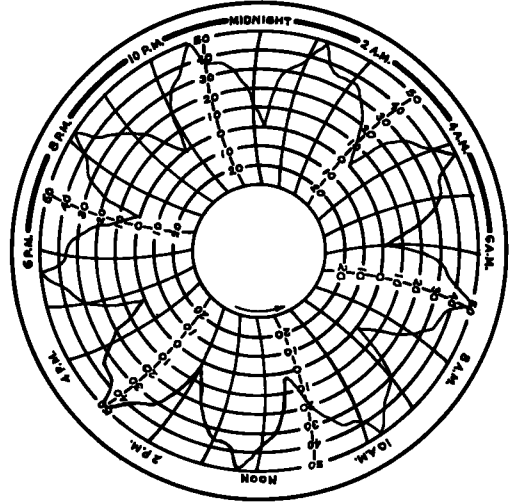


Figure 4. Typical graph of the recording thermometer.

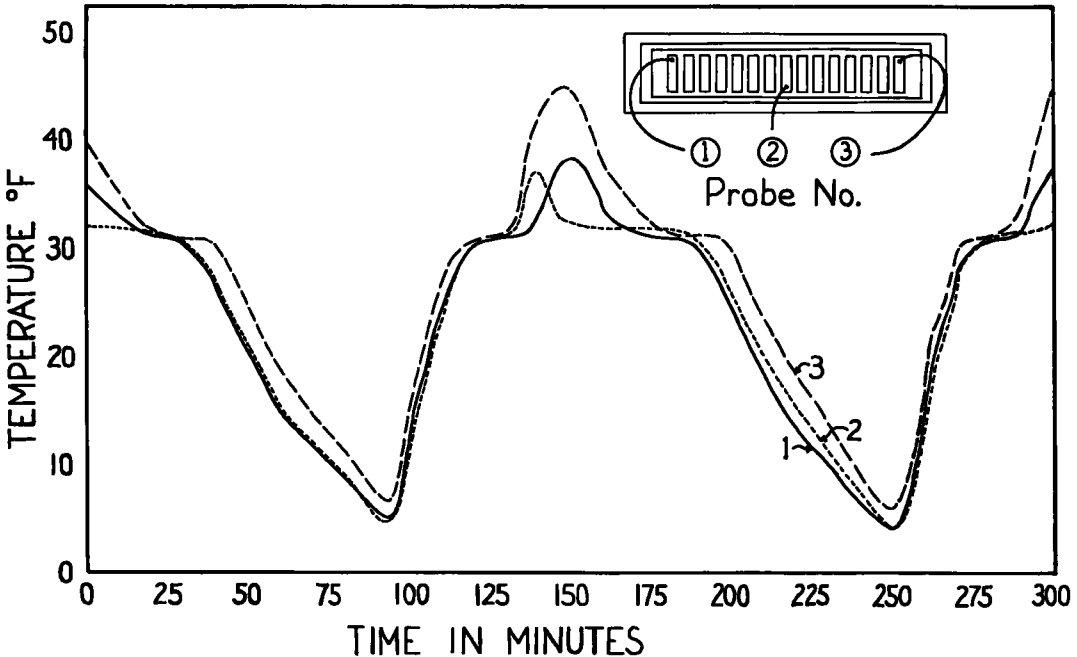


Figure 5. Time-temperature relation at three locations in the automatic freezing-and-thawing cabinet.

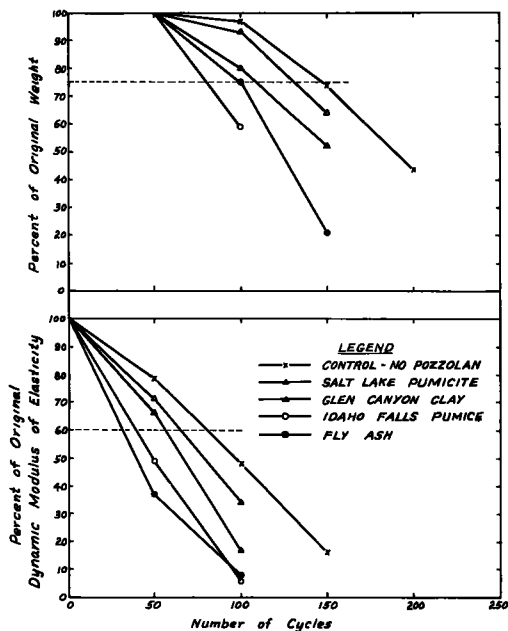


Figure 6. Freezing-and-thawing durability 2- by 2- by 10-in. concrete bars containing pozzolans.

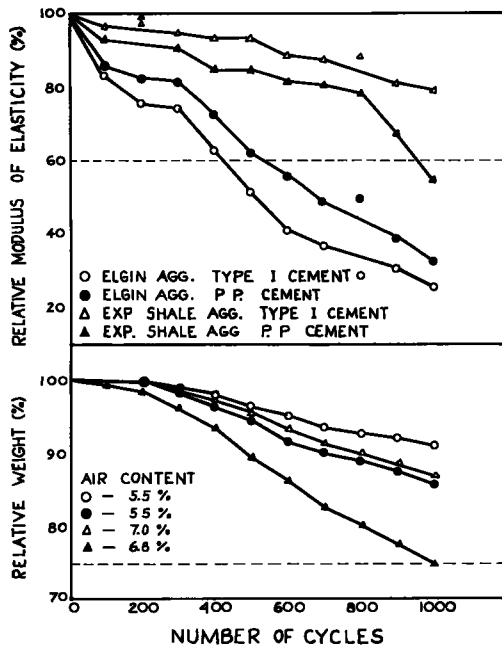


Figure 7. Durability of concrete with natural and lightweight aggregate—Type 1 portland pozzolan cement.

ability of concrete containing combinations of (a) natural aggregate from Elgin, Ill., (b) expanded shale lightweight aggregate, (c) Type I portland cement, (d) portland-pozzolan cement.

None of the concretes tested sustained a 25 percent weight loss before 1,000 cycles of freezing and thawing. These results are comparable to what might normally be expected with high quality air-entrained concrete.

REPRODUCIBILITY OF TEST RESULTS

Another measure of the adequacy of laboratory tests is the ability to produce similar results with similar samples. One method of measuring reproducibility is to analyze the range of results obtained with specimens fabricated from the same sample

TABLE 1
REPRODUCIBILITY OF FREEZING AND THAWING DURABILITY TESTS

Cement	Aggregate	Age in Days	Relative Weight, %—1000 Cycles					Relative Dynamic E, %—300 Cycles				
			Bar 1	Bar 2	Bar 3	Mean	Range	Bar 1	Bar 2	Bar 3	Mean	Range
Type I	Elgin	28	90.6	92.4	90.3	91.1	2.1	75.0	71.4	76.6	74.3	5.2
Type I	Exp. shale	28	86.4	86.1	88.1	86.9	2.0	93.7	94.4	96.7	94.9	3.0
P.P.	Elgin	28	85.2	87.1	86.2	86.2	1.9	88.0	75.5	81.9	81.8	12.5
P.P.	Exp. shale	28	76.1	74.1	74.6	74.9	2.0	85.7	88.8	97.2	90.6	11.5
Type I	Elgin	90	95.2	93.9	90.6	93.2	4.6	63.8	74.8	60.8	66.5	14.0
Type I	Exp. shale	90	86.9	90.9	89.8	89.2	4.0	91.8	91.0	88.1	90.3	3.0
P.P.	Elgin	90	90.4	84.6	87.4	87.5	5.8	66.9	70.7	-	68.6	3.8
P.P.	Exp. shale	90	86.0	84.9	87.3	86.1	2.4	83.0	85.8	81.5	83.4	4.3

¹Within test standard deviation and coefficient of variation.

Range	$\bar{R} = 3.1$	Range	$\bar{R} = 7.1$
Mean	$\bar{X} = 86.9$	Mean	$\bar{X} = 81.3$
Standard deviation	$\sigma = 1.83^1$	Standard deviation	$\sigma = 5.91^1$
Coefficient of variation - V	$= 2.1^1$	Coefficient of variation - V	$= 7.3^1$

of concrete. Table 1 is a compilation of test results where three duplicate prisms were fabricated and tested from the same batch of concrete. The degree of uniformity of durability tests is indicated by the range, standard deviation, and coefficient of variation. These results are only academic since duplicate tests made with other equipment are not available. The relative weight loss among comparable specimens shows excellent uniformity, however, and the coefficient of variation is lower than obtained with strength tests. The dynamic modulus of elasticity tests do not show comparable uniformity. There is also poor correlation between the relative durability as indicated by weight loss and by the loss of dynamic modulus. Concrete made with expanded shale aggregate shows superior durability when measured by the relative modulus of elasticity, whereas the expanded shale aggregate is inferior to natural aggregates when measured by weight loss. This apparent discrepancy cannot be attributed to poor reproducibility of the freezing-and-thawing equipment since both tests were made with the same specimen. The poor correlation between testing methods in this case is caused by differences in aggregates. Further investigations of weight loss versus loss of dynamic modulus with natural and lightweight aggregates is warranted.

CONCLUSIONS

Experience in the construction, development and use of the automatic freezing-and-thawing equipment indicates the following:

1. Automatic freezing-and-thawing equipment discussed in this paper is satisfactory for durability tests and can be manufactured at a cost which makes its use practical in small laboratories.
2. Simplicity and efficiency of the proposed freezing-and-thawing equipment makes its use sufficiently flexible and practical for acceptance tests and research.
3. Results indicate that freezing-and-thawing tests have good reproducibility.
4. Further investigations should be made to determine the influence of the position of the specimen and to check the uniformities of temperature throughout the cabinet.