

NATIONAL BUREAU OF STANDARDS REPORT

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A STUDY OF GILSULATE AS UNDERGROUND PIPE INSULATION

by

Selden D. Cole Paul R. Achenbach Frank J. Powlitch

to

Office of The Chief of Engineers Department of the Army Washington, D. C.



U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

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Heating and Air Conditioning Section Building Technology Division

to

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ABSTRACT

At the request of the Office of The Chief of Engineers, laboratory and simulated field tests were made of Type B Gilsulate as a proposed insulation for underground steam lines. Gilsulate consists of specially selected, sized, and blended particles of gilsonite, a natural mineral found in Utah. Gilsulate is asphaltic in nature and changes in physical appearance and chemical composition when heated. It was found that rapid heating sintered the particles at a temperature of about 225°F and consolidated it into a voidless material at a temperature of about 360°F to 370°F. Slower heating or dampening of the raw Gilsulate before heating inhibited consolidation but permitted sintering to take place. Gilsulate swells and increases in volume on heating and cracks during repeated heating and cooling. Because of this cracking a Gilsulate envelope around a steam pipe permits penetration of water under conditions of high water table. The thermal conductivity of raw granular Gilsulate was found to be about 0.64 Btu/hr (sq ft)(°F/in) at a mean temperature of 130°F. A sintered and consolidated envelope on a steam pipe operating at a temperature of 350°F had an average thermal conductivity of about 1.33 Btu/hr (sq ft) (°F/in) when surrounded by dry earth and only slightly higher in damp earth because it is non-hygroscopic. The test results indicate that Gilsulate might be an economical and useful insulation for underground steam lines in dry climates although conditions for corrosion might develop temporarily even in arid regions during periods of heavy rainfall because of its demonstrated tendency to crack under repeated heating and cooling.

1. INTRODUCTION

In response to the request of the Office of The Chief of Engineers, Department of the Army, an investigation was made of the mineral Gilsonite as an insulating material for underground steam pipes operating at a pressure of 125 psig corresponding to a saturation temperature of 350 F.

Gilsonite is produced under the trade name of <u>Gilsulate</u> by the American Gilsonite Company of Salt Lake City, Utah, for insulating hot underground pipes and protecting them against

corrosion. Gilsulate consists of specially selected, sized, and blended particles of gilsonites ranging in diameter from dust to 3/16 in. and resembles asphalt in its appearance but differs from asphalt in its composition and characteristics.

Gilsulate is produced in three grades for complete coverage of three temperature ranges.

| Grade | Temperature range | | |
|--------|-------------------|--|--|
| Туре А | 220 F to 310 F | | |
| Type B | 300 F to 385 F | | |
| Type C | 385 F to 520 F | | |

A typical Gilsulate installation consists of a pipe, supported in a dry trench or form, covered with a minimum thickness of four inches of the material, packed under and around the pipe, and tamped on the top surface to eliminate voids.

If the installation is below the ground water table or is made during inclement weather, the granular Gilsulate should be heated by passing steam through the pipe before covering with a minimum six-inch back fill in order to consolidate and sinter the material. For type B material consolidation and sintering is accomplished by passing steam through the pipe for 4 hours at 385 F, or 8 hours at 350 F, or 36 hours at 300 F and then allowing the pipe to cool down normally. The structure thus formed around the pipe consists of three layers with different characteristics (1) the consolidated zone nearest the pipe, (2) the semi-consolidated or sintered zone outside of it, and (3) the unconsolidated or granular zone. The consolidated part loses all trace of the individual particles from which it formed and resembles a hard,

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black, glossy, glass after it cools. The semi-consolidated or sintered material consists of the individual particles adhering together rigidly. The unconsolidated part shows no change visible to the eye from the original material as first used. It is the consolidated core and the semi-consolidated zone of the structure which are reported by the manufacturer to be watertight and to afford protection against corrosion.

To obtain both basic and practical information on Gilsulate when dry or wet, laboratory tests and full scale trench performance tests were conducted for determining physical characteristics, chemical properties, and performance under simulated use conditions.

The laboratory phase consisted of (1) heating dry or wet Gilsulate surrounding a hot pipe, (2) comparing the effect when the material was gradually oven-heated to progressively higher temperatures with that resulting from a rapid heating at one temperature, (3) determining the chemical properties chromatographically, (4) determining the thermal conductivity by the hot plate method.

The full scale tests consisted of passing steam at 350 F through a 4-in. pipe insulated with Gilsulate when embedded (1) in dry dirt, (2) in moist dirt with the water table 1 foot below the pipe, (3) with the water table level with the pipe, and (4) with water covering the insulating conduit. A second pipe was buried in the ground and insulated (1) with dry Gilsulate, (2) with dampened Gilsulate. The second installation was subjected to alternate heating and cooling, with the trench being wetted at

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each phase change.

2. DESCRIPTION OF EQUIPMENT

Part of the laboratory apparatus used to simulate the use of Gilsulate as a pipe insulation consisted of two cement asbestos boxes with a pipe running lengthwise through the center of each.

One box was $9 \ge 9 \ge 24$ in. constructed so that either end or any side could be removed independently. A 1-in. diameter galvanized pipe extended through the center of the box and had electric resistance wire wound on one end for a heater. Energy input to this heater was controlled with a thin-walled thermostatic switch located at the hot end. In use, the temperature of the pipe decreased with distance from the hot end so that successive transverse sections of the surrounding insulation were thus subjected to different pipe temperatures in one test, the duration of which may be several days or weeks. Thermocouples were peened into the surface of the pipe, and were spaced every two inches from the hot end for the first fourteen inches and every three inches for the remainder of the specimen.

The second cement-asbestos box was $9 \ge 9 \ge 36$ in. in size containing a l-in. diameter pipe centered lengthwise in it which was heated internally and thermostatically controlled, so that the surrounding insulation was uniformly heated. Thermocouples were peened into the surface of the pipe.

The thermal conductivity of unconsolidated Type B Gilsulate was measured in an 8-in. guarded hot plate apparatus conforming with the requirements of Fed. Spec. LLL-F-321b and of A.S.T.M.

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Twenty-one aluminum boats, each $2 \ge 2 \ge 4$ in. were prepared, and random filled with Gilsulate. These filled boats were placed in an electrically heated oven for exposure to various levels of temperature for selected lengths of time.

An analysis was made on four samples of Gilsulate in accordance with the method described in N.B.S. RP 2577, "A Chromatographic Method for the Fractionation of Asphalt Into Distinctive Groups of Components".

The full scale trench test was conducted in a watertight box 4 x 4 x 12 ft in size. Along the longitudinal center line and pitched 1/4 in. to the foot a 14 ft long 4 in. 1PS test pipe was suspended (See Fig. 1). The inside of the 4-in. pipe was divided into three sections: two internal half moon baffles in the bottom and two half moon baffles in the top, one slightly downstream and one upstream of the lower ones isolated a 4 ft measuring section at the middle. Separate internal condensate drain lines were brought out from each section and connected to high pressure steam traps. The condensate from the measuring section could be directed through a sight glass bypassing the steam trap. In each case, the condensate from the traps, or sight glass, was passed through a water-cooled heat exchanger to reduce the temperature to near room temperature so it could be collected and weighed in open vessels without appreciable loss by evaporation.

An electrically heated steam boiler of potential capacity

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FIG.



ranging from 1.5 to 13 KW (5000 to 45,000 Btu/hr) produced steam at the necessary pressure. The water level of the boiler was maintained by a float control opening a solenoid valve in a line from a supply tank held at a higher pressure than that of the boiler. A check valve in the supply line before the tank and one in the line after the solenoid valve prevented loss of pressure in the system from back feed.

Steam was delivered from the boiler through a 3/4 in. pipe extending about 1 ft into the upper end of the 4 in. pipe. The 1 ft extension was capped off at the end and a series of 1/4 in. holes drilled in its side walls whose aggregate area equalled 1.5 times the area of the open end of the 3/4 in. pipe. This produced a spray of steam on the side walls at the inlet end instead of a jet down the longitudinal center of the pipe.

The box was filled with oven-dried dirt, a typical Chester loam, to approximately mid-height. At this point a wooden form was placed about the pipe to make a 12-1/2 in. square conduit when filled with Gilsulate. After filling the form with Gilsulate, dirt was added to the sides, the form removed and the remainder of the box filled with dry dirt. Both materials were tamped with a 1/4 in. thick 8 x 8 in. iron tamper as they were added.

Five thermocouples were peened into the top of the pipe at positions 23 in. apart starting at the center and extending in both directions. A thermocouple was fastened to the pipe at its entrance to and its exit from the box. In addition, thermocouples were

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located in the Gilsulate and dirt with 11 in each of three strings, in a plane perpendicular to the steam pipe, at its center. The thermocouples in the three strings, extending downward, laterally and in an upward direction from the pipe, respectively, were spaced 1 on the pipe, the next 1/2 inch from the pipe, 4 at 1 in. intervals, 3 at 2 in. intervals, and 2 at 4 in. intervals (See Fig. 1). Temperature stations were also located on the steam traps and on the boiler. All thermocouples were of 26 gage copper-constantan wire and were used with a Rubicon portable precision potentiometer to indicate temperatures.

In order that the water table in the box might be adjusted, six 1/2 in. diameter pipes were equally spaced along each side of the box extending vertically from the top to the bottom. A 12 in. length of 2 in. pipe was fastened to the top of each 1/2 in. pipe for ease in introducing water. Before the dirt was added to the box, a 100 ft length of 1/2 in. rope was placed on the bottom of the box to provide distributing channels for the water introduced through these vertical pipes. Two 1 in. diameter vertical pipes placed diagonally from each other 1 ft from the side and 1 ft from either end of the box served as wells for measuring the actual water level (See Fig. 1).

The 4 x 4 x 12 ft box and its necessary auxiliary parts were set up in a specially erected building, 12 ft wide, 24 ft long and about 10 ft high, constructed of prefabricated insulated panels. An air chiller was placed at one end of the inside of the building with capacity enough to freeze the contents of the box, if desired.

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The air chiller was used to hold a steady temperature in the building when a test was in progress. The building was supplied with a 100 ampere, 3 phase, 208 volt electric service and a 1/2 in. water line with 70 lb pressure.

The outdoor apparatus consisted of a trench about 13 in. wide, 18 in. deep and 22 ft long. A 20 ft piece of 4 in. pipe was supported at each end raising it 4 in. above the bottom of the trench. Both ends of the pipe were sealed off with 1/4 in. iron plates. In one end a 3/4 in. pipe 18 in. long was inserted through the plate and welded to it with about 12 in. projecting into the inside of the pipe and 6 in. on the outside. The 12 in. projection was drilled with 1/4 in. holes and the end was capped off to make a side delivery steam nozzle. At the other end of the 4 in. pipe, a short 1/2 in. nipple was welded to what would be the bottom of the pipe, and the hole drilled through the side wall of the 4 in. pipe. A steam trap was connected to the 1/2 in. nipple using about 3 ft of copper tubing.

The 3/4 in. pipe was tied into the steam boiler. A thermocouple was inserted in a well in the steam line at the inlet end of the pipe and in the condensate line at the outlet end of the pipe. A thermocouple was fastened to the pipe about 3 ft from the inlet end and 3 ft from the outlet end.

The trench was filled with Gilsulate until there was a minimum of 4 in. covering the top of the pipe. The trench was then back filled with 6 in. of dirt.

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3. TEST PROCEDURE AND RESULTS

Sintering and Consolidation Temperatures

In the laboratory, the $9 \times 9 \times 2^4$ in. box was filled during separate tests with Gilsulate, (1) as it came from the shipping bag, (2) after it had been soaked in a container of water, and (3) with the unconsolidated residue that had been subjected to some heating, from previous tests. A constant heating temperature of about 525 F, measured at the heated end of the 1-inch pipe, was maintained for several days in each test.

At the end of the first test an investigation of the envelope formed around the pipe revealed the following conclusions as presented on Fig. 2A-2B and shown on the photograph Fig. 3.

1. Estimated initial sintering temperature was about 193 F. Material that showed sintering at temperatures lower than 226 F was too weak to withstand gentle handling.

2. The minimum temperature of pipe for sintering to obtain moderate strength and cohesiveness of the sintering annulus was 226 F. This sintered material showed only slight fusion of particles.

3. Glassy consolidation occurred at temperatures higher than 371 F. This material is a voidless "solidified liquid" when cool. A thin layer of glassy material up to 1/2 in. thick was found on the top surface of the pipe where the temperature was between 371 F and 491 F. At pipe temperatures above 491 F the material flowed from above the pipe leaving a void and collected under the pipe. The void was roofed with granular consolidated material with an inside

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film of glassy material.

4. The sintered material under the pipe, between 300 F and 371 F slumped away slightly, leaving a gap into which a flow of plastic material entered from the hotter end. This flow persisted until it reached a section of the pipe whose temperature was 310 F.

5. Pipe temperatures greater than 444 F caused considerable slumping of the unconsolidated granular material, due to shrinkage as it consolidated or melted at the pipe.

The second test in this same apparatus using Gilsulate that was soaked with water indicated that a consolidated core of glassy material could not be obtained at the temperatures recommended for Type B but that a sintered material only composed the envelope.

The third test using Gilsulate that had previously been heated, but not to the point of sintering, indicated that much higher temperatures were needed to obtain a sintering and consolidation equivalent to that observed with the original material.

The 9 x 9 x 36 in. box, with the heaters internally in the pipe, was filled with dry Gilsulate as it came from the shipping bag and the temperature of the pipe brought up to about 450 F for several hours. The system was then cooled completely to room temperature and the loose Gilsulate removed from the box, leaving the core exposed to room air conditions. When heat was again applied to the system the bottom half of the core dropped away from the pipe without having been softened by the heat.

Oven Tests

Further effects of heating Gilsulate were observed by placing

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the $2 \times 2 \times 4$ in. aluminum boats, random filled with Gilsulate as obtained from the shipping bag, into a thermostatically controlled electric oven with shielded elements. Thirteen samples were initially placed in the oven at room temperature. The temperature was increased in nine steps with the temperature being maintained constant at each successive level for 1 to 8 hours. At the beginning of each of the nine periods at constant temperature, an additional sample at room temperature was introduced into the oven and at the end of that period was removed with one of the initial 13 samples. The temperature of the oven was observed with a mercury in glass thermometer mounted through a hole in the center of the top surface.

Fig. 4 is a graphical presentation of the temperature each sample was subjected to during the oven tests and the length of the exposure. After the removal of the first four boxes, each removal thereafter consisted of two boxes, one box of the original 13, and one box exposed to a fixed temperature for a relatively short length of time. The exposure of each sample is listed below. For samples 1 through 13, each sample was exposed to the temperatures of all of the previous samples for the periods of time shown.

| Specimen l | was rea | noved aft | ter 22 | hours | at | 188 | F |
|-------------|-----------|-----------|---------|-------|----|-----|---|
| Specimen 2 | ** | 11 | " 24 | 11 | 11 | 196 | F |
| Specimen 3 | 11 | tt i | " 27 | ** | 11 | 230 | F |
| Specimen 4 | ** | TT 1 | " 40 | 78 | 11 | 302 | F |
| Specimens 5 | & 14 were | removed | after 4 | ** | ** | 358 | F |
| Specimens 6 | & 15 " | 71 | " 8 | 11 | 11 | 358 | F |

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| Specimens | 7& | : 16 | were | removed | after | 1 | hour | at | 385 | F |
|-----------|-----|------|--------|---------|-------|---|-------|----|-----|---|
| Specimens | 8 & | ; 17 | 11 | 11 | *1 | 2 | hours | 11 | 385 | F |
| Specimens | 9 & | 18 | н | ** | ŦŦ | 3 | ŦI | Ħ | 385 | F |
| Specimens | 10 | & 1 | 9 " | ** | 11 | 4 | 11 | 11 | 385 | F |
| Specimens | 11 | & 2 | C " | 11 | 11 | 4 | 11 | 11 | 401 | F |
| Specimen | 12 | | was | ** | 11 - | 4 | ** | 11 | 455 | F |
| Specimens | 13 | & 2 | l were | e " | 11 | 4 | TT | 11 | 507 | F |

The visual effect of time-temperature treatment upon Gilsulate is shown in the photographs number 5-9, inclusive. Fig. 5 shows that sintering took place at the 302 F temperature level with specimen 4. Figs. 6, 7, 8, 9 show that the initial 13 specimens sintered but did not consolidate at any temperature up to 507 F regardless of the length of time of exposure but specimens 14 through 21 consolidated starting at 358 F and 4 hours duration.

This series of time-temperature tests indicates that a gradual increase in temperature changes the characteristics of Gilsulate so that it does not flow together into a homogeneous consolidated mass, whereas a sudden exposure for a short length of time at any temperature above 358 F causes complete consolidation, if the material has not been previously heated.

Thermal Conductivity

The thermal conductivity of a sample of unconsolidated Gilsulate was measured by pouring the loose material, as taken from the shipping bag, between the plates of the hot plate apparatus and rodding the loose material to prevent voids.

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Thickness, as tested, inch 1.00 Thermal conductivity, Btu/hr ft²(deg F/inch) 0.636 Mean temperature of specimens, °F 130.9

Temperature gradient in specimens, deg F/inch 42.9

The thermal conductivity of the dry dirt used to cover the Gilsulate conduit was determined in the same manner as the Gilsulate. The loose dirt was slightly pulverized so that the largest granules were not over 1/8 in. and was then poured between the plates of the apparatus and settled by considerable rapping of the plates.

| | Density, as tested, lb/ft ³ | 65.5 | |
|-------|---|-------|-----|
| | Thickness, as tested, inch | 1.01 | |
| | Thermal conductivity, Btu/hr ft ² (deg F/inch) | 1.55 | |
| | Mean temperature of specimens, °F | 128.1 | |
| | Temperature gradient in specimens, deg F/inch | 33.0 | |
| * | Loss of weight on drying, percent | 2.3 | |
| *Mate | erial was tested as received and then was air-dried | after | the |
| | | | |

test to constant weight in an oven at 215 F.

Controlled Underground Tests

For underground studies of the heat transmission factor of Gilsulate with controlled conditions the manufacturer's recommended directions for forming the conduit were followed, and simulated conditions of actual field use were applied in completing the investigation.

With the Gilsulate poured around the pipe and the test box filled

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with dry dirt to a level approximately 2 feet above the center line, steam between 325 F and 355 F was turned into the system for a period of about one week. The steam temperature was then raised to 380 F for a period of four hours after which the steam was turned off.

Fig. 10 is a graphical presentation of this time-temperature combination used to form the consolidated core in the Gilsulate envelope.

After allowing the entire apparatus to cool to a steady state, steam at 350 F was admitted to the pipe and held at that temperature for eleven days at which time temperature stations and condensate rate indicated a steady condition prevailed throughout the Gilsulate conduit.

Water was added through the stand pipes until a free water level of 1 ft was measured in the box. The 1 ft level was maintained until the earth on the surface about 3 ft above the free water level became moist. Steam at 350 F was admitted to the pipe while the 1 ft water level was held constant. At the end of 22 days of constant steam temperature and water level, a steady condensate rate and temperatures in the conduit again prevailed.

Immediately at the conclusion of the test with a l-foot water table, a l in. core sample, from top to bottom of the test box, adjacent to the Gilsulate conduit was taken in increments and the percent moisture for each increment determined. A second core sample was taken from directly over the conduit center for comparison with the moisture content of the first core sample. The core was taken

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in measured increments with each increment placed in a separate weighed container. Each container and sample was weighed before heating in an oven at 215 F and again after heating when there was no more change in weight. Fig. 11 shows the percent of moisture to dry dirt for each increment of the two cores and the change of moisture content with height in the test installation. The short curve B shows the dirt to be slightly drier above the conduit than the dirt adjacent to the conduit. The percent moisture at both stations at the level of the top of the conduit and at the surface of the dirt was about 5 percent, increasing to about 10 percent a few inches above the conduit and dropping to zero percent at the pipe level. Below the conduit the percent moisture to dirt indicated by the core samples increased rapidly from zero at the conduit to about 38 percent at the 2 in. level. It is probable that water drained from the samples taken below the free water level as they were being withdrawn. Thus the 38 percent shown at the 2-in. level is probably not representative of the actual conditions.

The rates of condensation measured in the measuring section of the pipe for the tests with dry dirt and with a 1-foot water level were 0.503 lb per hour for the dry dirt and 0.765 lb per hour for the moist dirt.

To determine the thermal conductivity factor, k_m , the equation for steady heat transfer through an annulus was used

$$k_{\rm m} = \frac{q \ln \frac{1}{r_{\rm o}}}{2 \pi L \Delta T}, \text{ in which}$$

q = rate of heat loss in Btu/hr for the measuring section

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MOISTURE DISTRIBUTION



ln = logarithm to base e

r: = radius at outside surface of annulus, ft

T1 = 3.1416

 ΔT = difference in temperature measured at the two radii, °F k_m = thermal conductivity factor, Btu/hr (sq ft)(°F/ft)

Substituting the appropriate values obtained from the tests of the underground installation in the equation, the thermal conductivity factors for the Gilsulate envelope surrounded by dry earth and moist earth are shown below. The thermal conductivity of the dry dirt and moist dirt surrounding the conduit, expressed in Btu/hr (sq ft)(°F/in), are also shown below based on the temperature gradient observed in the dirt and assuming that the above equation is applicable approximately to this case.

Fig. 12 shows the average temperature in the Gilsulate and earth at the midpoint of the measuring section plotted against the ratio of the distances to each station and to the pipe surface as measured from the center of the pipe. The interface between the Gilsulate conduit and dirt lay between the 3-1/2 in. and 4-1/2 in. stations. The curve through the dry and moist dirt is a straight

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line in Fig. 12 on semi-log paper indicating steady conditions and a uniform material, and a uniform rate of change of temperature with the logarithmof the distance from the pipe. While the curve for the Gilsulate conduit in Fig. 12 approaches a straight line yet, there is deviation from a straight line indicating a difference in the thermal conductivity of the material, especially near the pipe, where a consolidation of some thickness was expected to exist.

Water was again added to the box until the 2 ft level above the bottom of the box was reached. At this height the water level was midheight of the 4 in. pipe and Gilsulate conduit. With all temperature in the system initially at room temperature steam at 350 F was admitted to the pipe and 15 minutes later the inlet and outlet pipe temperatures were 235 F and 131 F, respectively. Six hours later the temperatures were 239 F and 180 F, respectively. 3 days and 6 hours after starting the test the temperatures were 230 F and 224 F at the inlet and outlet of the pipe. During this time the boiler was using maximum energy input, the condensate rate from the measuring section was averaging 12.3 lb per hr and the pressure could only be maintained a little above atmospheric pressure. Temperatures taken 3 in. below the surface of the dirt or about 16 in. above the conduit surface and centered over the conduit ranged from 140 F to 144 F with the ambient temperature at about 75 F.

Water was again added to the box raising the water level to 3 ft from the bottom which completely covered the conduit. Steam at

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350 F was admitted to the pipe and one hour later the inlet and outlet pipe temperature read 218 F and 205 F. This drop in temperature took place with full energy input to the boiler and 5 days later the temperatures were 221 F and 214 F and the pressure was only slightly above atmospheric.

Lowering the water level below the conduit caused an immediate increase in pipe temperature indicating that the conduit was drying out whereas an increase in the height of the water level to cover the conduit caused an immediate drop in pipe temperature to about that of boiling water at atmospheric pressure.

Figs. 13, 14 and 15 show the temperatures observed at equal distances from the pipe in downward, lateral and upward directions when the conduit was surrounded with dry dirt, and with moist dirt and three levels of the water table in the box. The measured rate of condensate for the four-foot measuring section with dry dirt was 0.50 lb per hr; with the 1 ft water level, 0.76 lb per hr; and with the 2 ft and 3 ft water levels, about 12 lbs per hr with the boiler at full capacity.

Fig. 16 is a photograph of the Gilsulate conduit as it was being removed from the box after the tests were complete. The Gilsulate had sintered completely, with a relatively thin layer of glassy consolidated material surrounding the pipe. There was no evidence that the consolidated Gilsulate adhered to the pipe. Fig. 17 is a photograph of three pieces of the conduit showing the surface that was in contact with the pipe (large center piece) and

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the cracks that were observed on all pieces adjacent to the pipe. The upper left piece shows the narrow band of consolidation; the lower left piece shows the amount of sintering that occurred on the surface of the conduit. The entire conduit broke up into churks resembling coke in appearance as it was removed, revealing an extensive system of cracks and fissures in the Gilsulate envelope as indicated in Fig. 16.

Trench Tests

For the outside installation of the 20 ft pipe in a trench 18 in. deep and about 13 in. wide, the Gilsulate was moistened before it was placed around the pipe in the manner recommended by the manufacturer. The empty paper bags were used to cover ten feet of the conduit before backfilling with dirt. Thermocouples were peened into the pipe about 3.5 ft from either end, and in the same plane on the surface of the Gilsulate and 1 in. below the surface.

Steam at a temperature of about 375 F was turned into the pipe to consolidate an inner core. After four hours the steam was turned off and the system allowed to cool for several days. When steam was again turned into the pipe, the temperature was limited to 352 F automatically.

During the next three weeks a series of heating and cooling and wetting cycles took place. The time of heating and the time of cooling varied from one to three days and the amount of water added varied from complete flooding of the trench to a mild wetting. Each time the water table was raised, when the pipe was being heated

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steadily, the boiler temperature would drop from 350 F to about 212 F at full energy input. As the water table was lowered the pipe temperature and boiler temperature would increase. Fig. 18 is a photograph showing the conduit at the inlet end. The glassy consolidation near the pipe is hardly discernible, most of the core being a tightly sintered material. Fig. 19 is a photograph of the outlet end of the pipe showing the cracks in the portion of the conduit that was covered with paper.

A second installation was made using dry Gilsulate as it came from the shipping bag and curing it immediately after it was installed at 375-380 F for four hours. After the four hour curing time the steam temperature was reduced to 352 F and held for 65 hours. As the steam temperature was being reduced a small hole, large enough for observation purposes, was dug through the Gilsulate to the pipe. A second sampling and inspection was made 17 hours later and a third sampling made 40 hours after the first one.

The first sampling revealed a soft sticky plastic about 3/4" thick adjacent to and adhering to the pipe. The second sampling and inspection revealed a settlement groove along the center line of the conduit on the outside surface and the Gilsulate near the pipe was a sticky plastic with a hard crust on the outside. The third sampling revealed a coke-like substance on top of the pipe, a void space at the side of the pipe and apparent filling of a void at the bottom.

After cooling for 72 hours, about 4-1/2 ft at the inlet end

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was freed of all loose material leaving the rigid core exposed. Steam at a temperature of 350 F was admitted to the pipe. As the pipe and envelope warmed up, a longitudinal crack opened up on the top of the core followed by a similar one on the side. On adding water to the trench where the core was exposed for 4 to 5 feet, vapor was observed about as soon as the water touched the conduit. By the time the water was level with the center of the pipe the boiler steam temperature had dropped to about 212 F. Fig. 20 is a photograph showing the exposed hard core and the cracks that developed in it.

Chromatographic Analyses

Four samples of Gilsulation were analyzed chromatographically and are listed as follows:

- No. 1 "Raw". Proposed for use between 300 and 385 F. This sample is representative of the material as received before subjection to field test.
- No. 2 "Heated". This sample represents No. 1 after subjection to field test in which steam line was at 350 F.
- No. 3 "Severely Heated". This sample represents No. 1 after subjection to field test in which steam line was at 380 F.
- No. 4 "Raw No. 2". This sample represents the material submitted by the supplier after completion of the referenced field tests. This material proposed as a

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replacement for Sample No. 1 with the claim it was definitely known to be Type B.

Chromatographic Analysis of Gilsulate

| Sample No. | l | 2 | 3 | 4 |
|--------------------------------|-------------------|---------|--------|---------|
| Coke residue, %, ^a | 17.4 | 19.6 | 20.8 | 16.1 |
| Volatile Loss, %, ^b | 0.2 | 1.1 | 2.1 | |
| Composition, %: C | | | | |
| Asphaltenes | 79.2 | 88.8 | 97.6 | 78.2 |
| Water-White Oils | 2.6 | 1.4 | 0.8 | 3.0 |
| Dark Oils | 8.3 | 3.7 | 0.8 | 6.1 |
| Asphaltic Resins | 9.9 | 5.1 | 1.2 | 11.0 |
| Chloroform Desorbed | 0.3 | 0.2 | trace | 0.9 |
| Total | 100.3 | 99.2 | 100.4 | 99.2 |
| Ratio: Asphaltenes to Petro | olenes 3.8/1.0 | 7.9/1.0 | 41/1.0 | 3.6/1.0 |

^a A.S.T.M. Method D168-30

b 24 hours at 400 F

C N.B.S. Method RP2577.

Ordinarily, the hardness of a bituminous substance can be measured by determining either its consistency or softening point, or both. Unfortunately, the characteristic hardness and intumescent (swelling and increase in volume caused by application of heat) properties of gilsonite made the determination of either softening point or consistency impractical. An indirect method such as coke

residue at 1750 F was used. The results revealed that a progressive increase in coke residue, and therefore hardness, had occurred during the field tests.

The asphaltenes were hard, brittle, black solids which intumesce on heating. The petrolenes (combination of water white oils, dark oils and asphaltic resins) act as dispersing media for the asphaltenes and are believed to impart fusibility to the bitumen. In general, the lower the ratio of asphaltene to petrolenes the lower will be the softening point and therefore the more easily fusible will be the bitumen. These ratios for the samples marked No. 1 and No. 4 were 3.8/1.0 and 3.6/1.0. While the behavior during the test for volatile loss and the lower ratio of asphaltene to petrolene indicate improved fusibility of sample No. 4 over sample No. 1, it is doubtful whether the improvement would be significant.

Studies at the National Bureau of Standards and other laboratories have shown that asphalts, when subjected to heat, will show an increase in asphaltenes due to changes occurring mainly in the dark oils and asphaltic resins rather than to volatile losses. Sample No. 1 showed as much as an 18.4% increase in asphaltenes content and a corresponding 86.5% decrease in petrolenes content, after exposure to field tests as sample No. 3. Since the volatile loss after 24 hours of exposure to 400 F was 2%, compared with 0.2% volatile loss of No. 1, it can be assumed that the changes which occurred during the field tests were due to changes in composition rather than to volatilization losses.

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4. DISCUSSION AND CONCLUSIONS

The laboratory and simulated field tests of Type B Gilsulate indicated the following conclusions about the material tested:

- (a) The characteristics of Gilsulate change when heated. When dry Gilsulate is heated rapidly it sinters at a temperature of about 225°F and consolidates at a temperature of about 360°F to 370°F. When dry Gilsulate was heated slowly it sintered but did not consolidate at temperatures up to 507°F.
- (b) Wetting Gilsulate before heating inhibits consolidation.
- (c) Gilsulate is intumescent; i.e. it swells and increases in volume as a result of the application of heat.
- (d) Gilsulate cracks during repeated heating and cooling.
- (e) A hard consolidated core is not always formed upon heating. The Gilsulate does not soften upon heating except during the initial curing process.
- (f) Gilsulate does not adhere to steel pipe for the most part after initial curing and consolidation.
- (g) Gilsulate does not resist penetration of water after being heated and cooled down.
- (h) The thermal conductivity of raw granular Gilsulate is about 0.64 Btu/hr (sq ft)(°F/in) at a mean temperature of 130°F. The thermal conductivity of a sintered and consolidated envelope of Gilsulate on a steam pipe is about 1.33 Btu/hr (sq ft)(°F/in) in dry earth and only slightly higher in damp earth because it is essentially non-hygroscopic.

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(i) A Gilsulate envelope on a steam pipe permits water to come in contact with the pipe when the water table is at the level of the pipe or higher resulting in high condensation rates and possible corrosion.

These findings indicate that Gilsulate might be an economical and useful insulation for underground steam lines in dry climates where the water table does not reach the level of the pipe. In areas where the water table reached the level of the pipe Gilsulate insulation would permit high condensation rates and might not protect the steam line against corrosion.

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