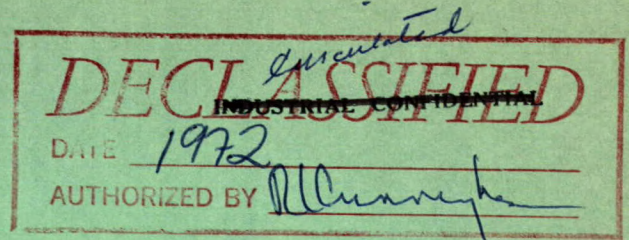


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MINES BRANCH INVESTIGATION REPORT

IR 71-43

June, 1971

IMPROVING LIGHTWEIGHT AGGREGATE PROPERTIES
OF A CLAY FROM EDMONTON, ALBERTA
THROUGH THE USE OF ADDITIVES

by

H.S. Wilson

Mineral Processing Division

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- - -

SUMMARY OF RESULTS

Seven of the twelve additives studied improved the bloating of the "lower" clay to various degrees. The best results were obtained with calcium-ammonium, calcium and sodium lignosulphonates, sodium carbonate, and flour.

The unit weights of graded coarse aggregates, produced from the clay using one and two per cent additions of the above compounds, ranged between 28 and 43 lb/cu ft. Crushing strengths of the aggregates compared favorably with those of some commercially produced lightweight aggregates.

Thermal gravimetric analyses indicated that a gas-producing additive will improve the bloating only if the formation of the glassy phase is promoted by increased fluxing.

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CONTENTS

| | <u>Page No.</u> |
|------------------------------------|-----------------|
| Summary of Results | i |
| Introduction | 1 |
| Composition | 1 |
| Stationary-Kiln Tests | 2 |
| Additives | 2 |
| Preparation of Materials | 3 |
| Stationary-Kiln Firing | 4 |
| Rotary-Kiln Tests | 17 |
| Scope | 17 |
| Preparation of Materials | 17 |
| Rotary-Kiln Firing | 17 |
| Physical Properties | 18 |
| Discussion of Results | 19 |
| Thermal Gravimetric Analysis | 21 |
| Conclusions | 35 |

LIST OF TABLES

| <u>No.</u> | <u>Page No.</u> |
|--|-----------------|
| 1. Chemical Analyses of Clays | 2 |
| 2. Rotary-Kiln Firings and Physical Properties | 19 |

LIST OF FIGURES

| <u>No.</u> | | <u>Page</u> |
|------------|-------------------------------------|-------------|
| 1. | No Additive | 5 |
| 2. | Additions of Fly Ash | 6 |
| 3. | Additions of Bindarene M | 7 |
| 4. | Additions of Bindarene LA | 8 |
| 5. | Additions of Lignosol XD | 9 |
| 6. | Additions of Sodium Carbonate | 10 |
| 7. | Additions of Sodium Silicate | 11 |
| 8. | Additions of Flour | 12 |
| 9. | Additions of Limestone | 13 |
| 10. | Additions of Gypsum | 14 |
| 11. | Additions of Sulphur | 15 |
| 12. | Additions of Potash | 16 |
| 13. | No Additive | 22 |
| 14. | No.additive | 23 |
| 15. | Bindarene M Additive | 24 |
| 16. | Bindarene LA Additive | 25 |
| 17. | Lignosal XD Additive | 26 |
| 18. | Sodium Carbonate Additive | 27 |
| 19. | Sodium Silicate Additive | 28 |
| 20. | Flour Additive | 29 |
| 21. | Limestone Additive | 30 |
| 22. | Gypsum Additive | 31 |
| 23. | Sulphur Additive | 32 |
| 24. | Potash Additive | 33 |
| 25. | Potassium Sulphate Additive | 34 |

INTRODUCTION

Lightweight aggregate is produced from a deposit of glacial clay at Edmonton, Alta. by Edmonton Concrete Block Co. Ltd. The deposit is composed of two clays, the upper layer being the raw material presently used by the company. The lower clay, although not greatly different in composition from the upper clay, is not a satisfactory lightweight aggregate raw material. The compositions and properties of these two clays have been studied previously and were reported in Mines Branch IR 69-3, "Comparison of Clays from Edmonton, Alberta, for Lightweight Aggregate."

The Mineral Processing Division was requested by the company to attempt to improve the bloating properties of the lower clay by the use of chemical additives.

COMPOSITION

The mineralogical compositions of the two clays were determined by Differential Thermal Analysis (DTA). This examination showed that the two clays are composed of basically the same minerals, namely illitic, chloritic, and montmorillonitic clay minerals, gypsum, quartz, dolomite, pyrite, and organic matter. They differ in that the upper clay contains less montmorillonoid, less quartz, and more calcite and dolomite than does the lower clay.

Further determination of composition through mineralogical analyses by X-ray diffraction patterns and X-ray diffractograms showed that the principal constituents in descending order of abundance are as follows:

Upper clay: quartz, feldspars, montmorillonoid, calcite, gypsum, dolomite, and amphibole (?)

Lower clay: quartz, feldspars, gypsum, montmorillonoid, mica, and dolomite.

The chemical analyses of the two clays are shown in Table 1.

TABLE 1

Chemical Analyses of Clays

| Element | Upper Clay | Lower Clay |
|--------------------------------|------------|------------|
| SiO ₂ | 60.00 | 68.70 |
| Al ₂ O ₃ | 15.02 | 12.20 |
| Fe ₂ O ₃ | 4.16 | 4.16 |
| FeO | 0.87 | 0.61 |
| CaO | 3.40 | 2.48 |
| MgO | 2.01 | 1.35 |
| K ₂ O | 2.18 | 1.87 |
| Na ₂ O | 0.77 | 1.01 |
| Total C | 1.07 | 0.79 |
| Elemental C | 0.69 | 0.69 |
| S | 1.15 | 0.86 |
| CO ₂ | 1.39 | 0.38 |
| Soluble SO ₄ | 2.77 | 2.49 |
| L.O.I. | 10.83 | 7.91 |

STATIONARY-KILN TESTS

Additives

In this preliminary phase of the study, various additives were used in amounts of 0.5, 1.0, 2.0 and 5.0 per cent of the air-dried weight of the clay. One additive, fly ash, was also used in amounts of 10.0, 20.0, and 30.0 per cent. The additives that were used are as follows:

- Fly ash - supplied by Western Fly Ash Ltd.,
Edmonton, Alta.
- Bindarene M - calcium lignosulphonate - ammonium
lignosulphonate mixture (sugar free)
supplied by Commercial Alcohols Ltd.,
Gatineau, Que.

| | |
|-------------------------|--|
| Bindarene LA | - calcium lignosulphonate (sugar free), supplied by Commercial Alcohols Ltd., Gatineau, Que. |
| Lignosol XD | - sodium lignosulphonate, supplied by Lignosol Chemicals Ltd., Quebec, Que. |
| Sodium Carbonate | - reagent grade |
| U-Brand Sodium Silicate | - soda:silicate ratio 1:2.4, supplied by National Silicate Ltd., Toronto, Ont. |
| Flour | - supplied by Robin Hood Mills. |
| Limestone | - supplied by Summit Lime Works, Lethbridge, Alta. |
| Gypsum | - supplied by Truroc Products Division, BACM Ltd., Amaranth, Man. |
| Sulphur | - supplied by Canadian Industries Limited, Montreal, Que. |
| Potash | - supplied by Alwinal Potash of Canada, Lanigan, Sask. |

Preparation of Materials

For all tests, the air-dried clay was stage-crushed to minus 16-mesh in a jaw crusher. For each test, 150 g (0.33 lb) of clay was mixed with 40 g (27 per cent) of water and the appropriate amount of additive. Additives that are soluble in water were dissolved in the mixing water before being added to the clay. The insoluble additives were ground to minus 100 mesh and mixed with the clay before the water was added. In each case, the clay was brought to the plastic state by hand mixing and, after wrapping in a polyethylene sheet, was aged for 12 to 18 hr to insure uniform dispersion of the water.

The clay was formed into pellets using a hand-powered extrusion machine. Small pieces of clay were fed into the barrel of the machine, the feed and die openings were sealed and the air was evacuated by a vacuum pump. The clay was then extruded through the multiple die openings by the screw driven piston. The 3/8-in. streams were cut into 1/2-in. lengths, and the resulting pellets were dried at 110°C (230°F).

Stationary-Kiln Firing

The materials were fired in an up-draft, gas-fired stationary kiln having a 4 by 8-in. hearth. The atmosphere in the kiln was oxidizing. The firing temperatures were 1095°, 1120°, 1150° and 1175°C (2000°, 2050°, 2100°, and 2150°F).

A charge of 8 pellets (in a preheated refractory sagger) was inserted into the kiln at the desired temperature and removed after 8 min. The firing temperature was maintained manually within 10 Farenheit degrees of the desired temperature.

The bulk specific gravity of the product of each test firing was determined. For this determination, the fired pellets were weighed to within 0.01 g, and the volume was measured, by the mercury displacement method, to within 0.1 ml. The bulk specific gravities are shown graphically in Figures 1 to 12. In each graph showing the effect of an additive, the specific gravity of the clay without additive is also shown so the effect of the additive can be clearly seen.

Temperature °F

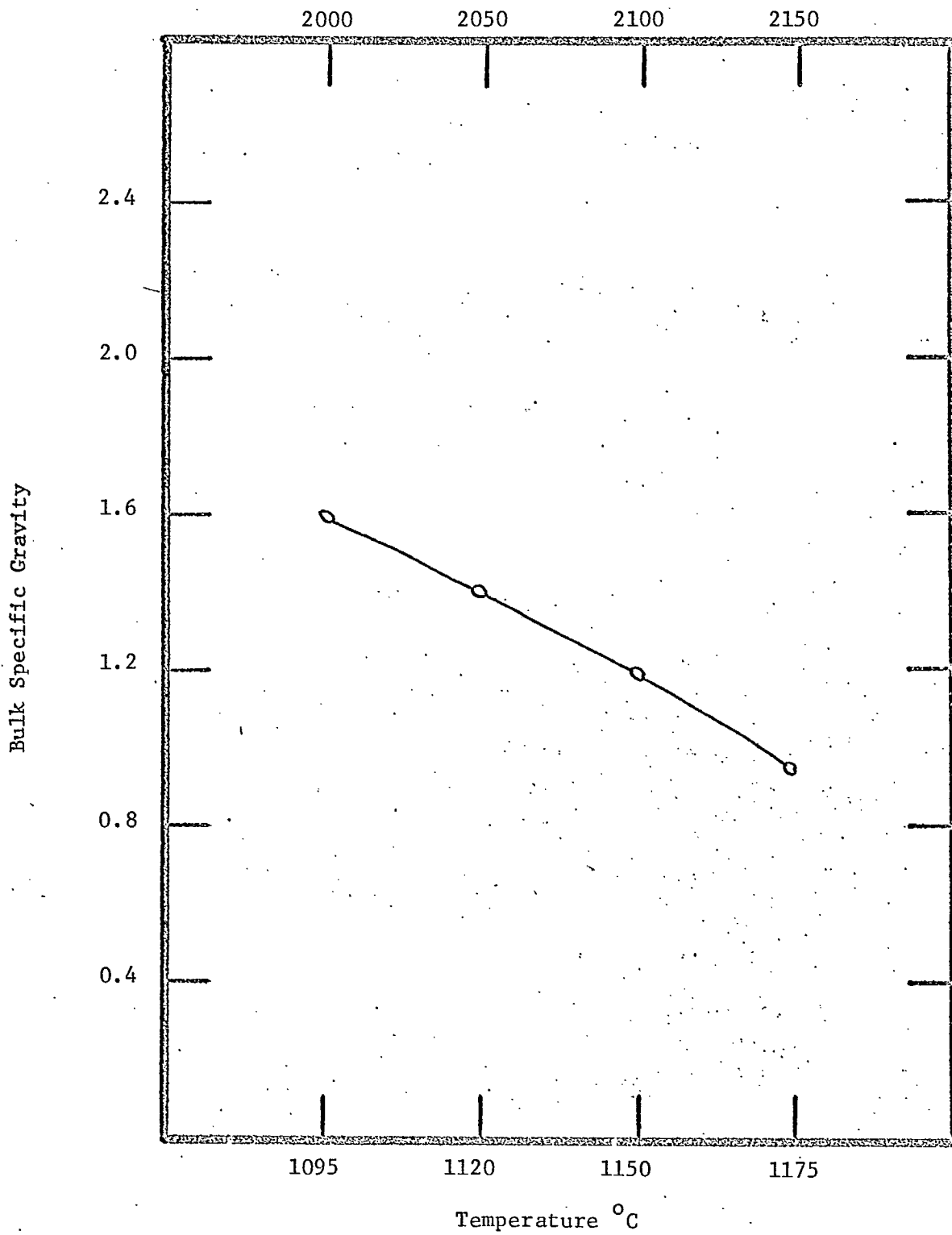


Figure 1: No Additive

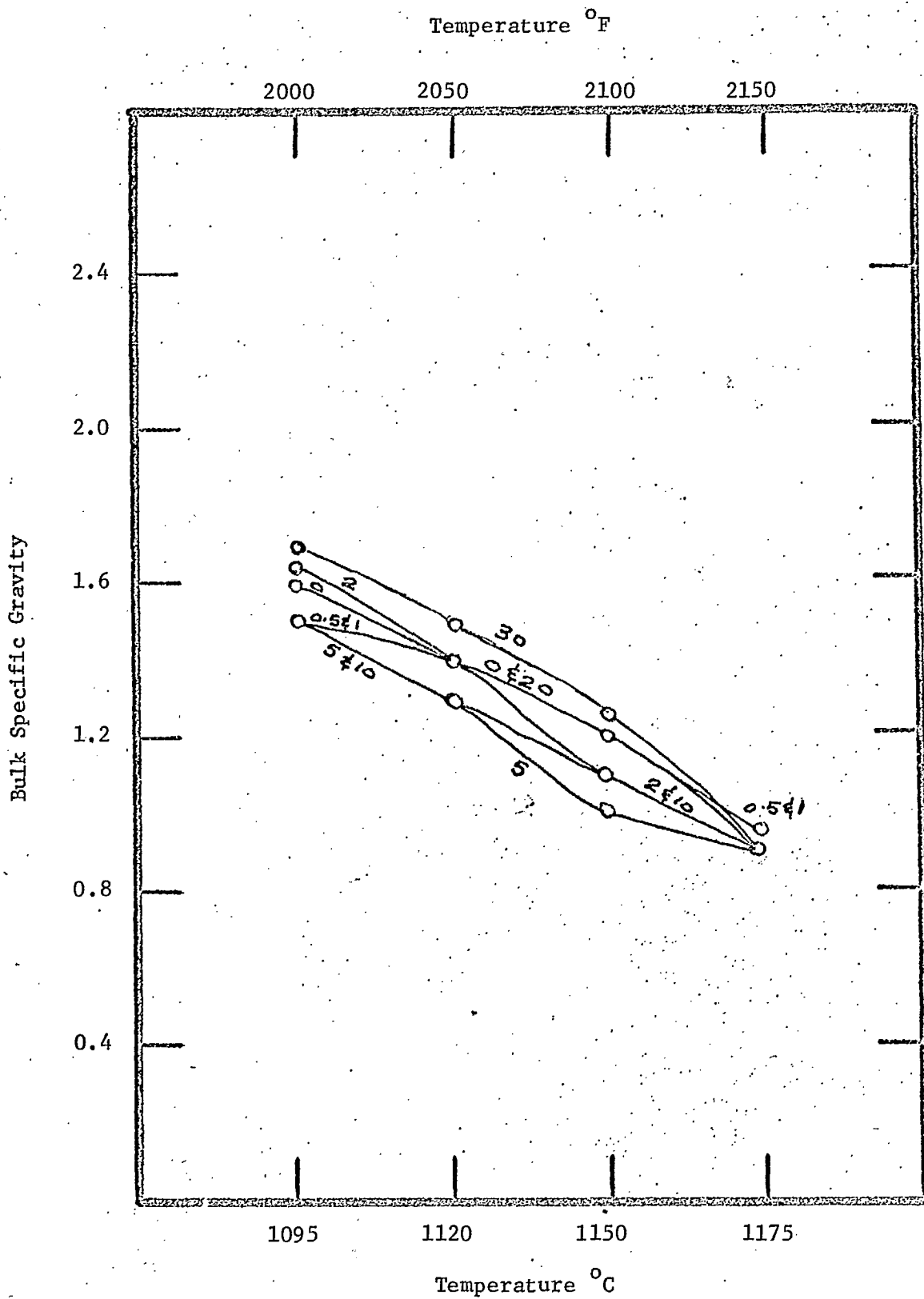


Figure 2: Additions of Fly Ash

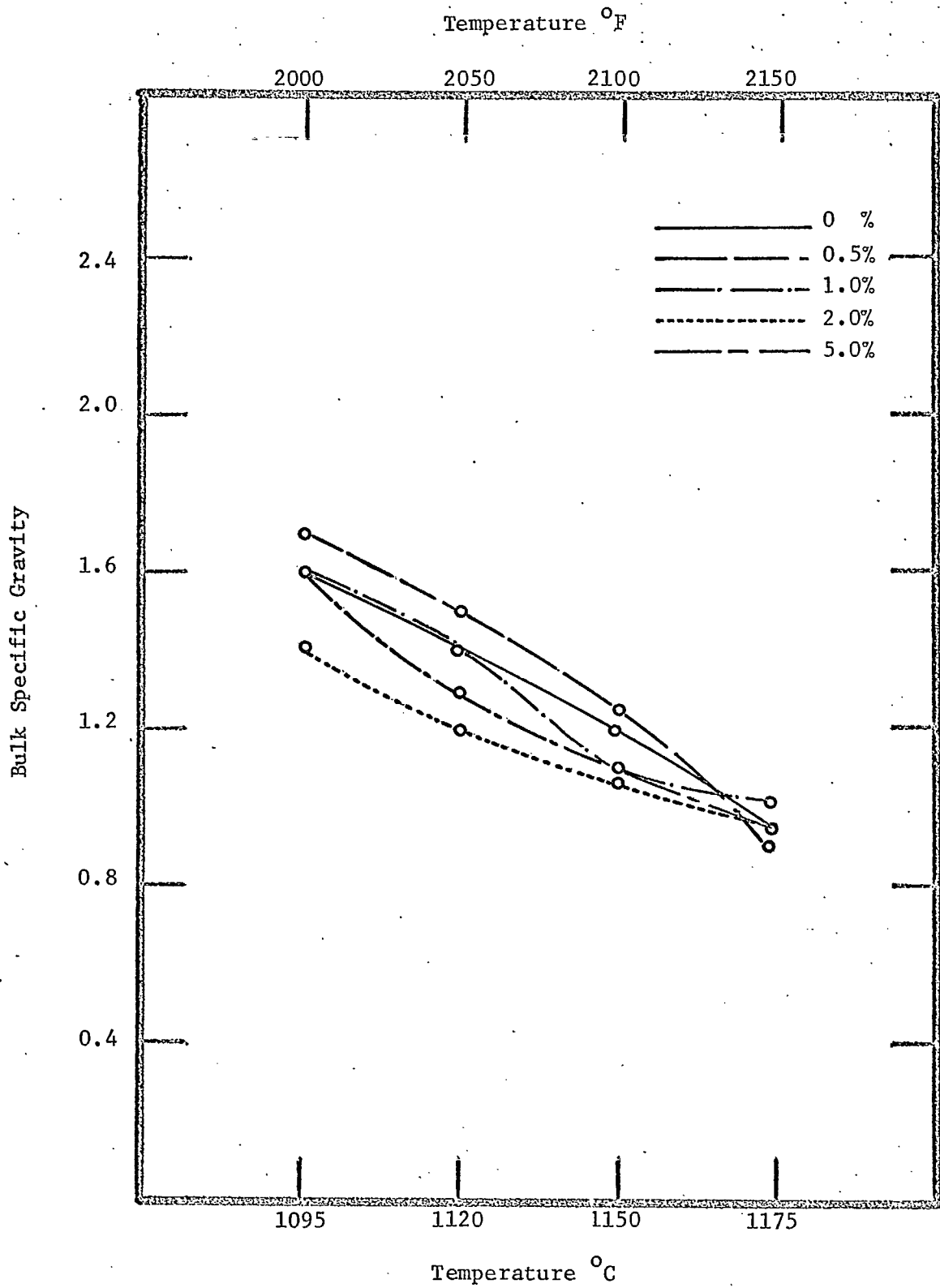


Figure 3: Additions of Bindarene M

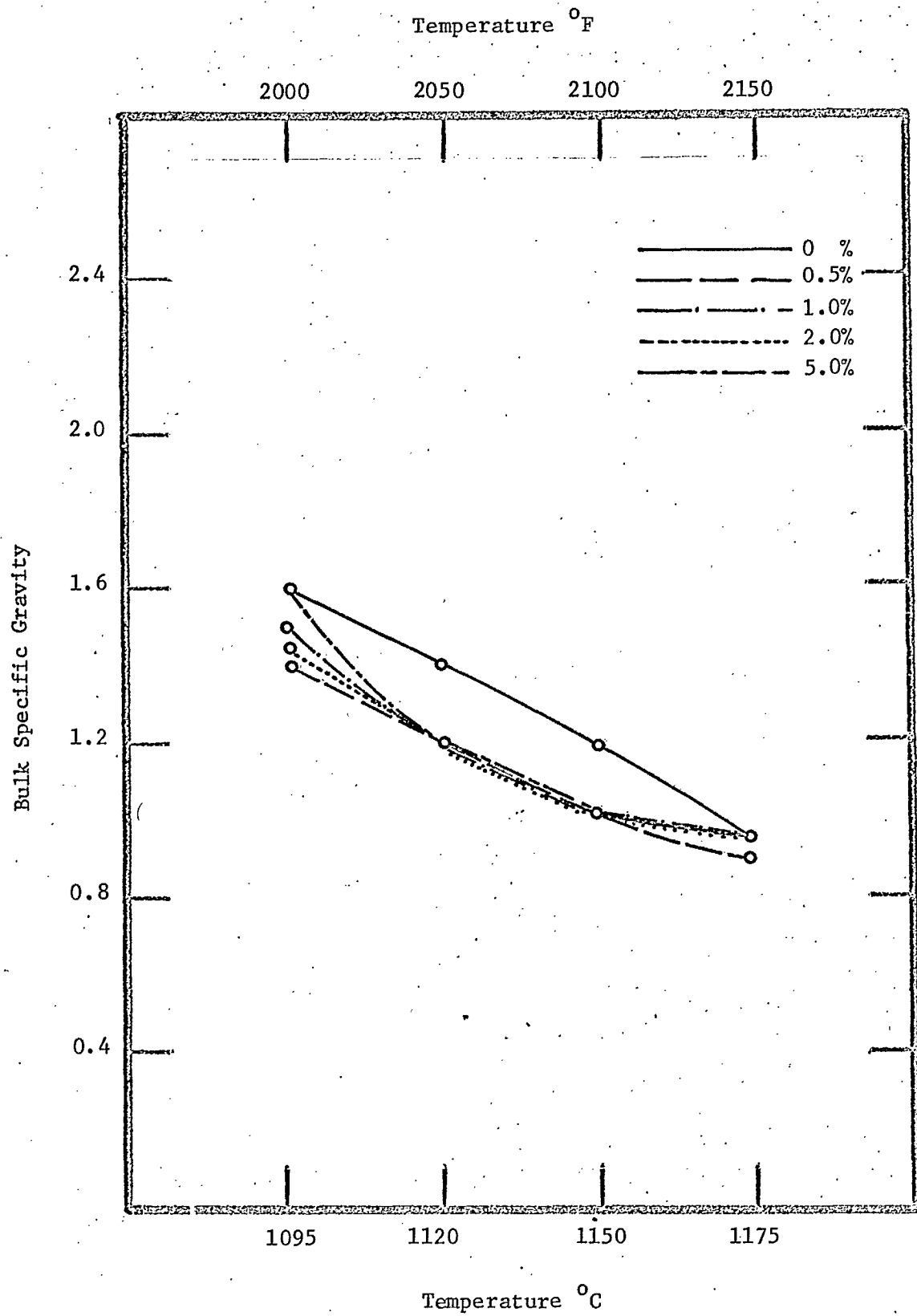


Figure 4: Additions of Bindarene LA

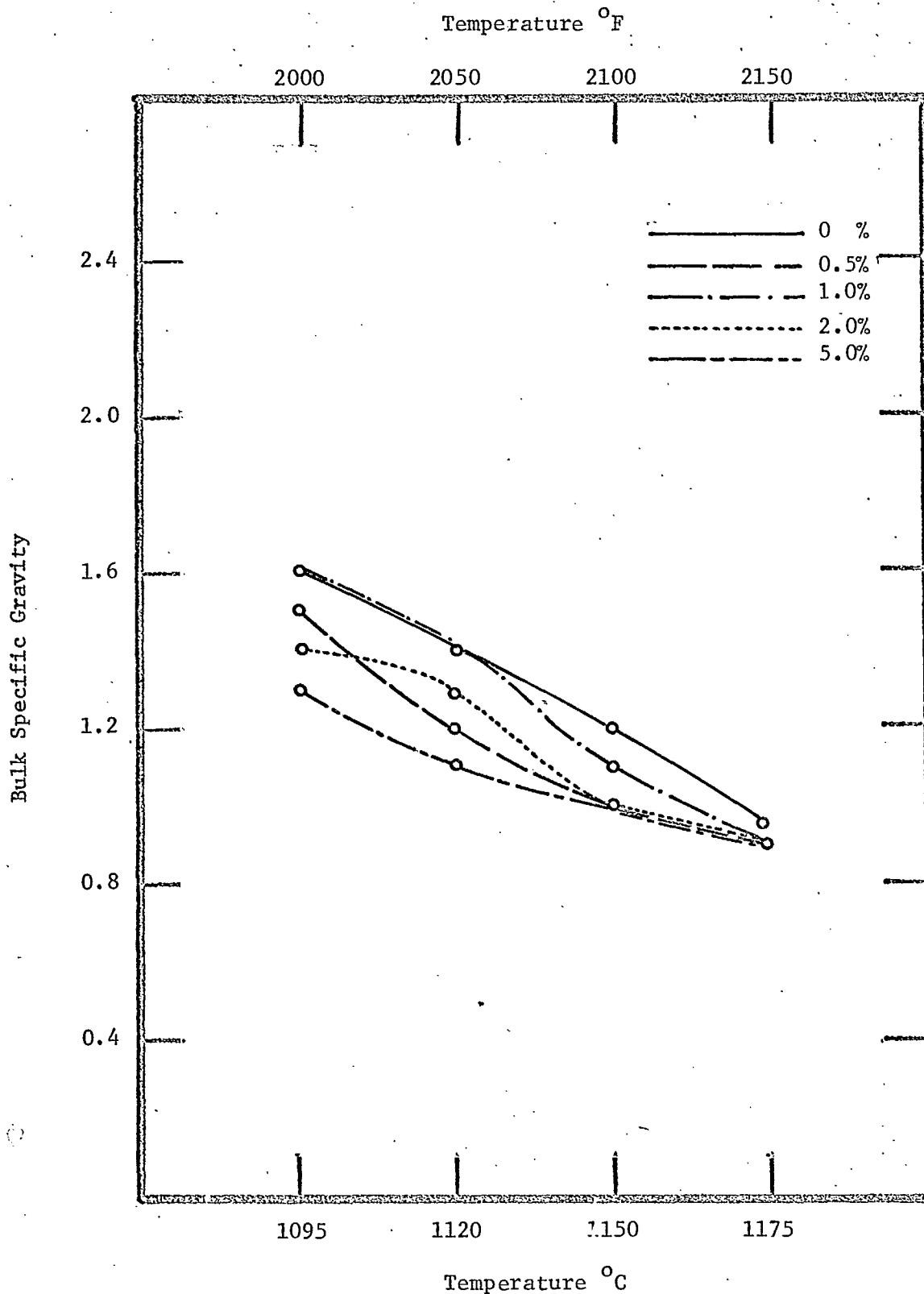


Figure 5: Additions of Lignosol XD

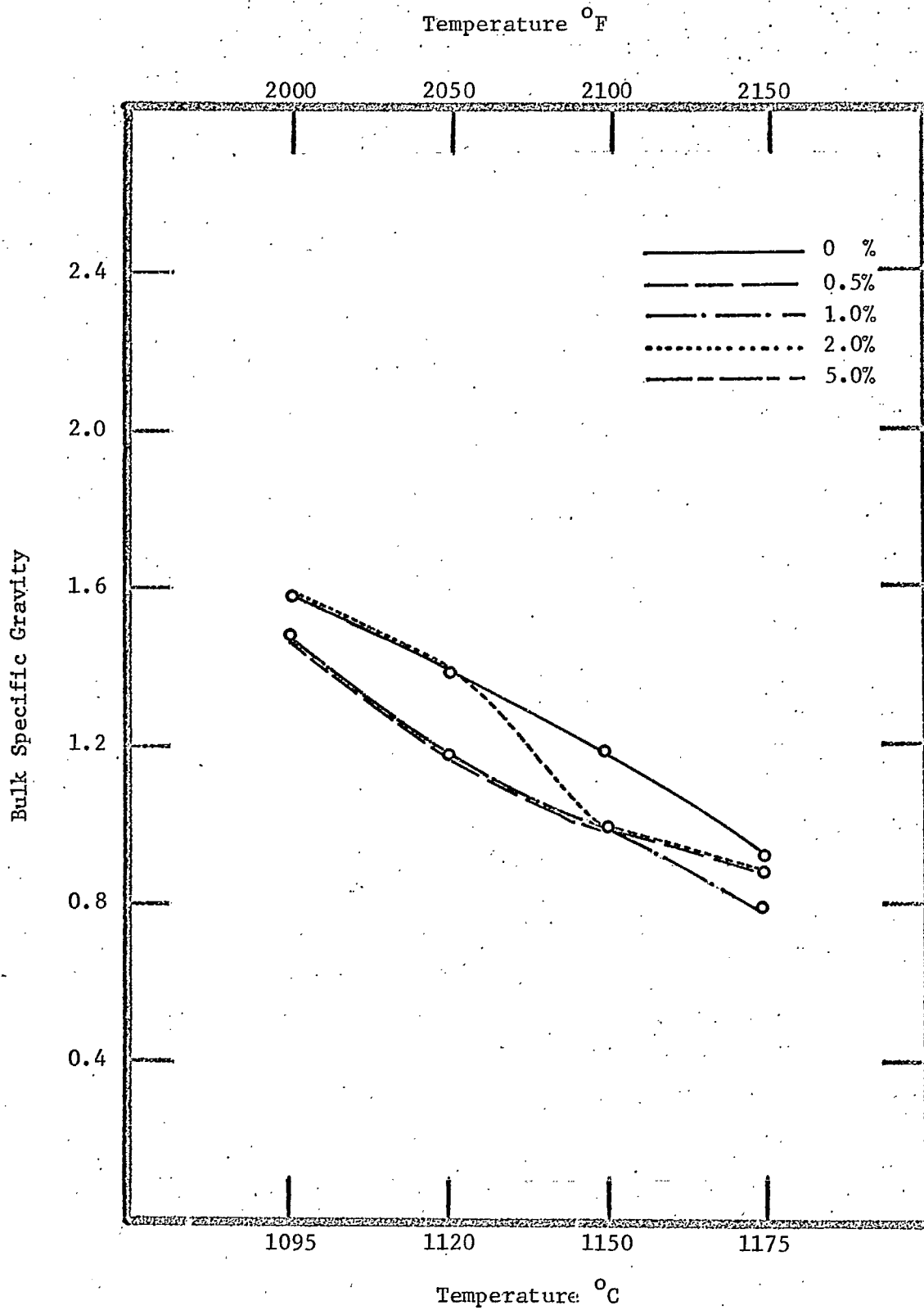


Figure 6: Additions of Sodium Carbonate

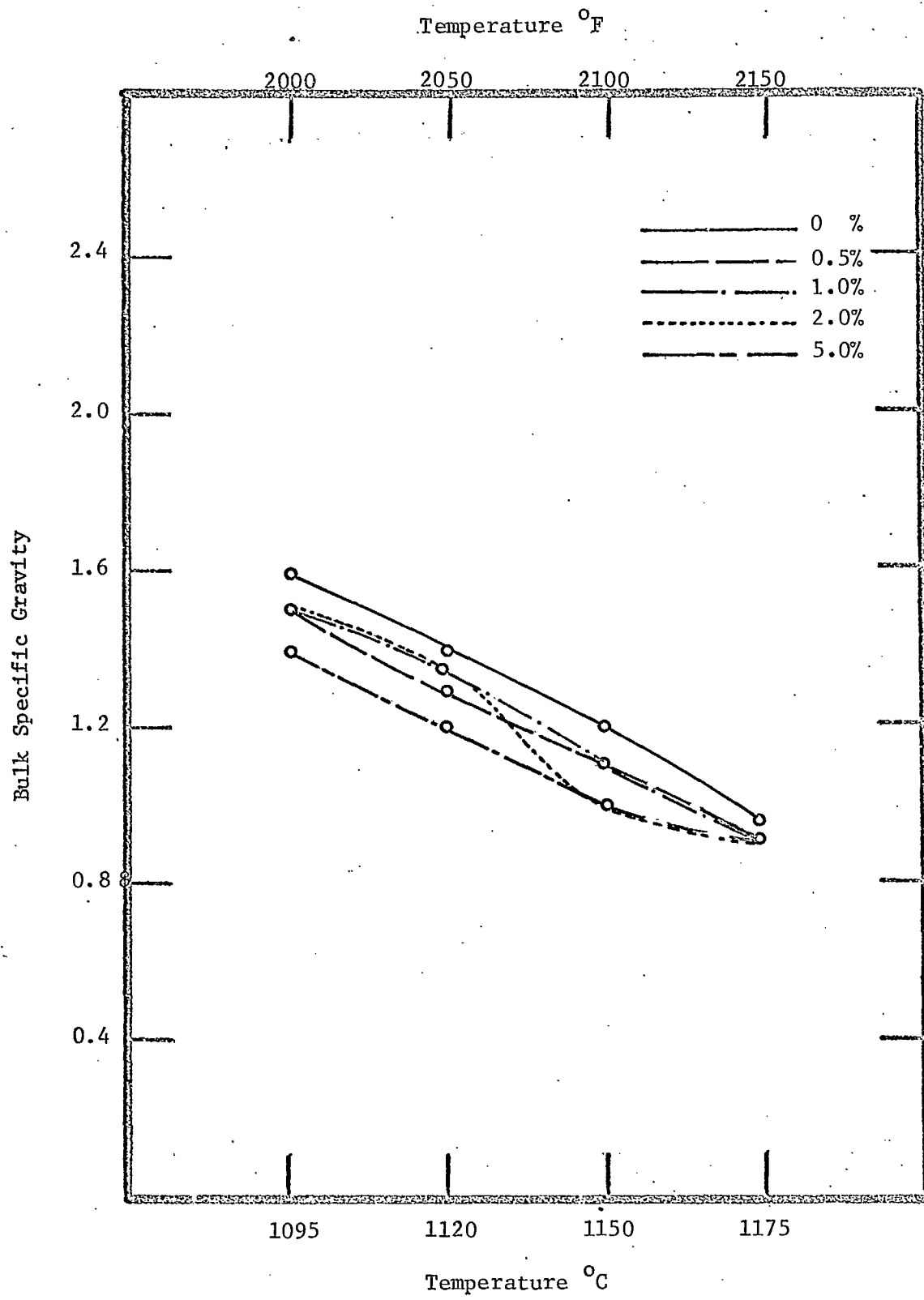


Figure 7: Additions of Sodium Silicate

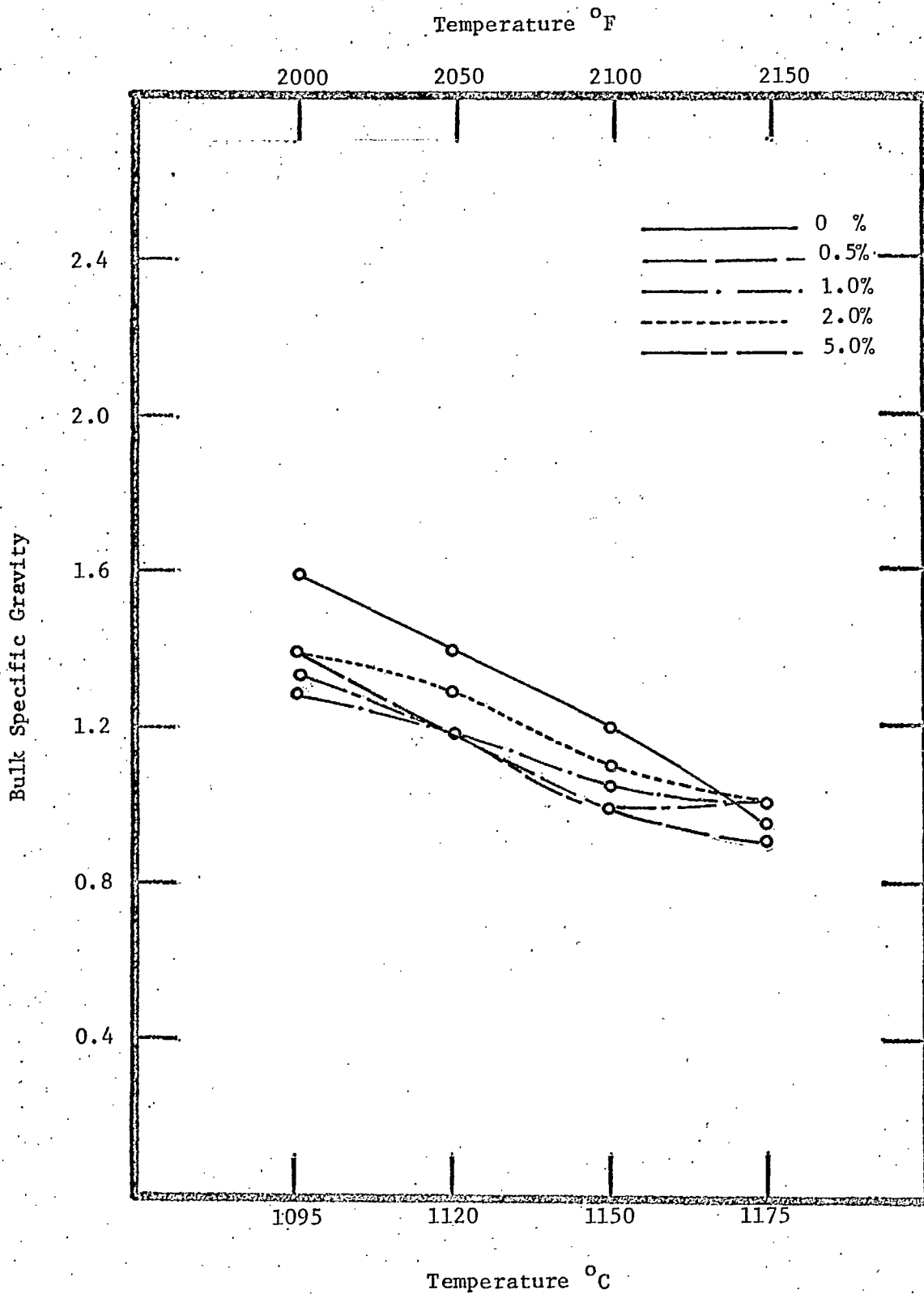


Figure 8: Additions of Flour

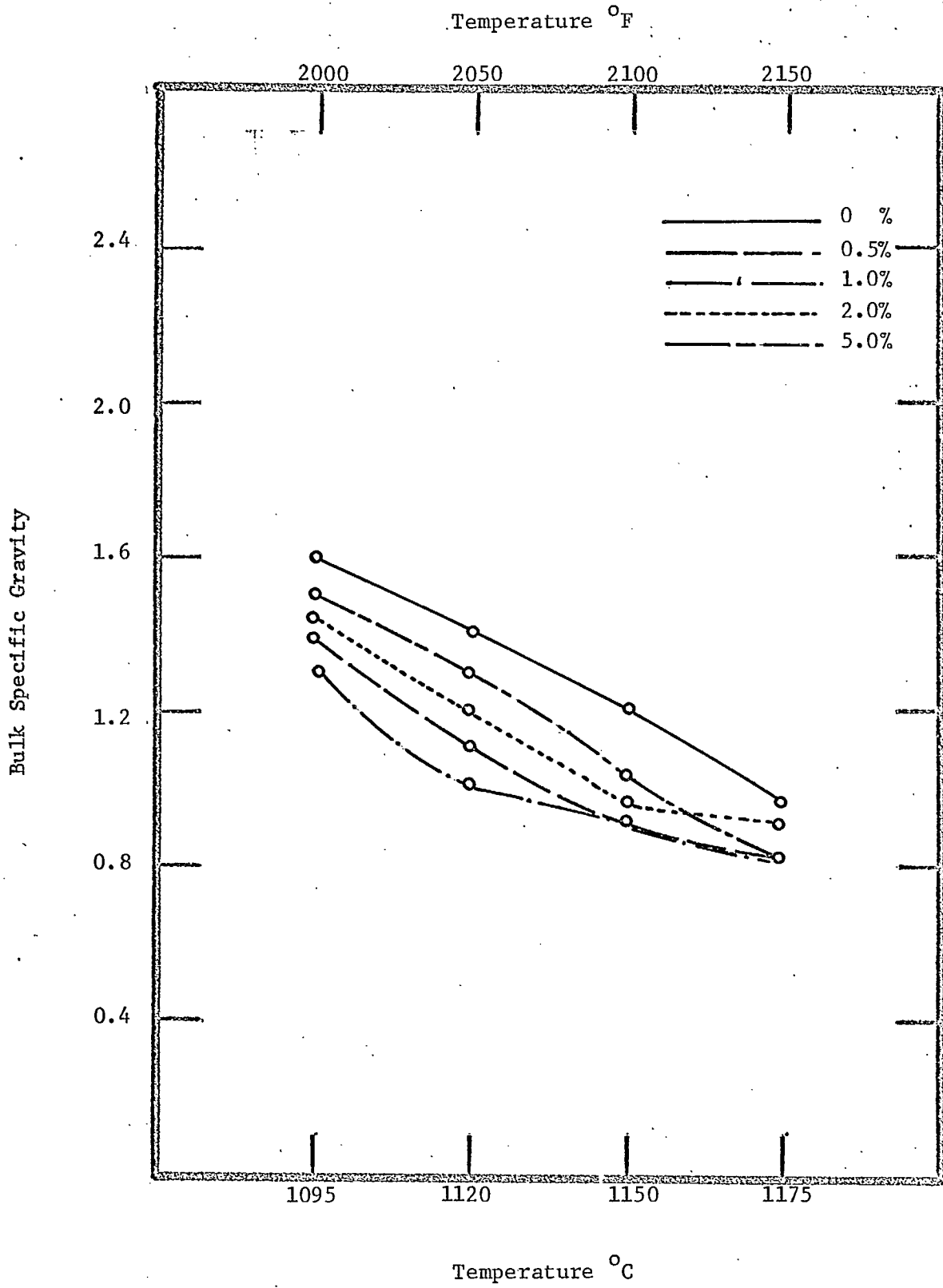


Figure 9: Additions of Limestone

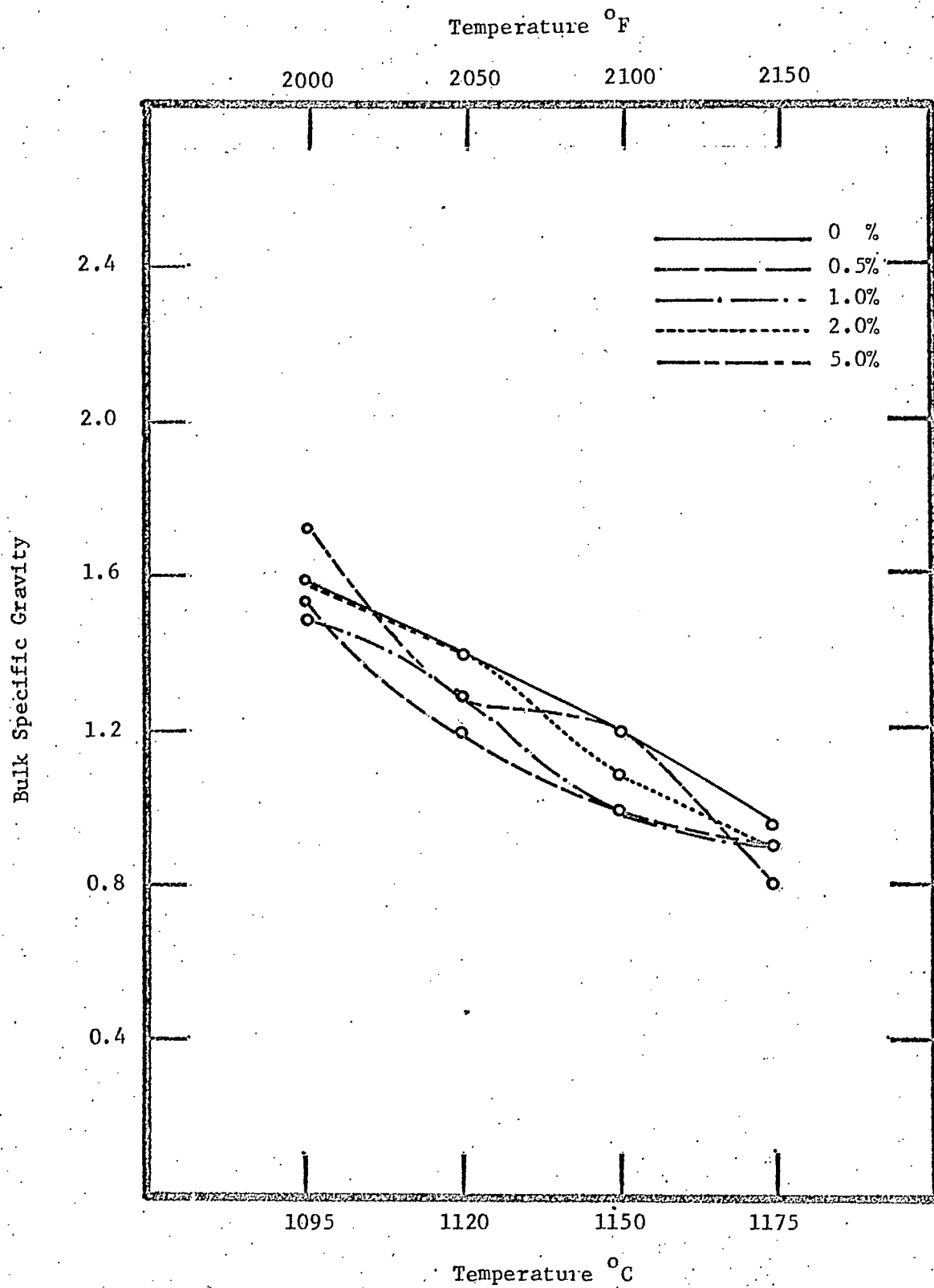


Figure 10. Additions of Gypsum

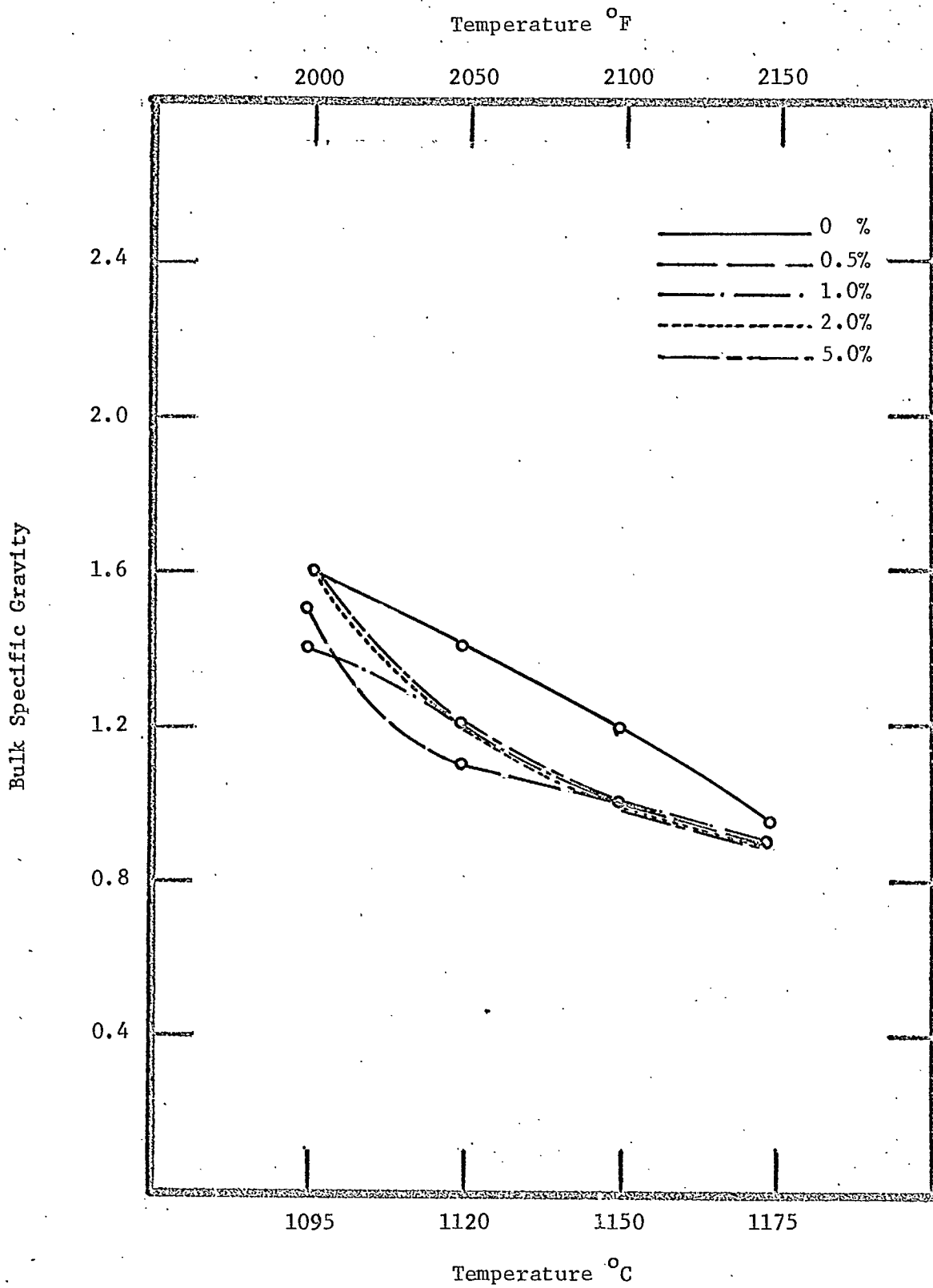


Figure 11: Additions of Sulphur

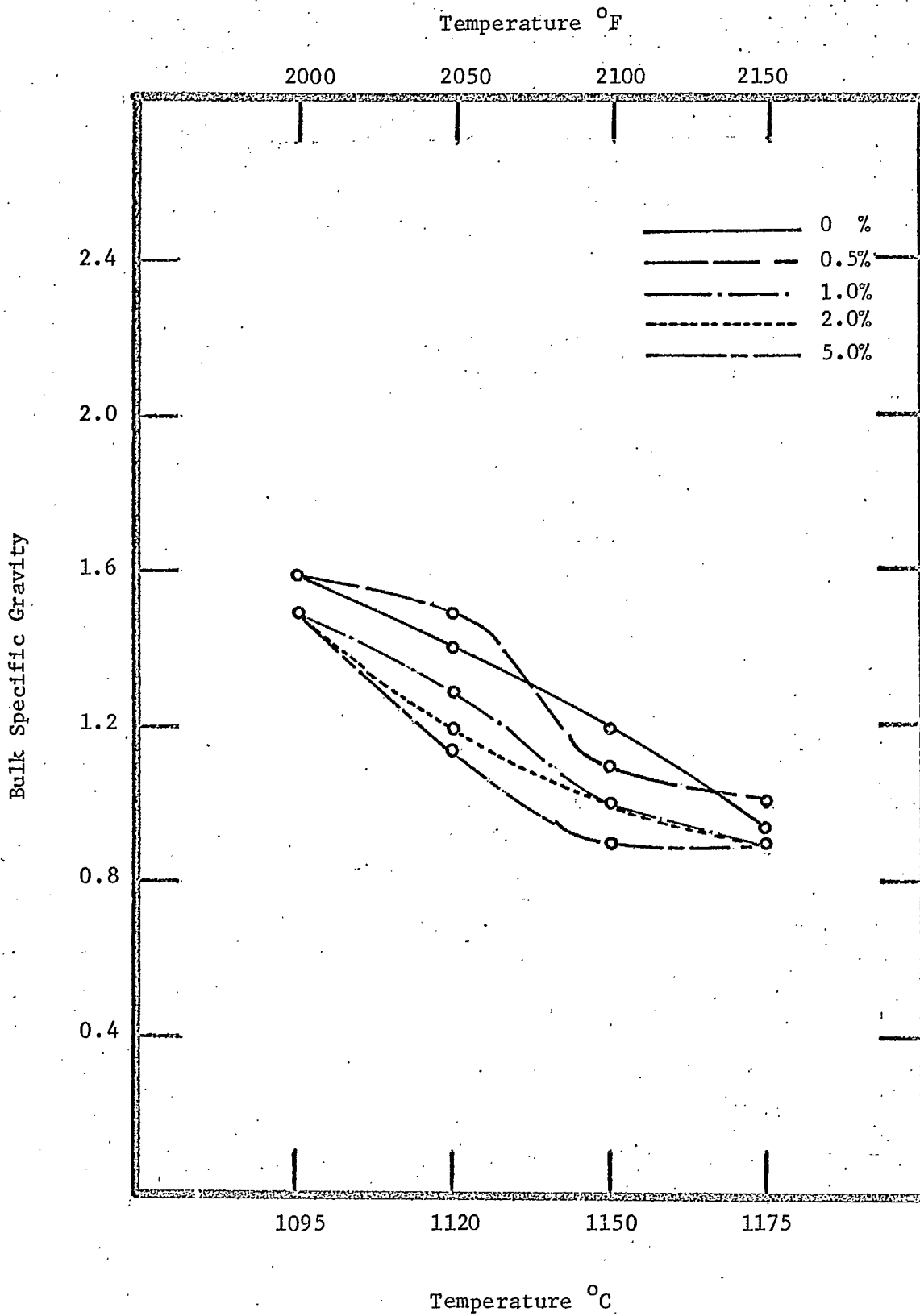


Figure 12: Additions of Potash

ROTARY-KILN TESTS

Scope

Of the additives used in the Stationary-Kiln Tests, only the fly ash did not appreciably improve the bloating of this clay. The other additives improved the bloating to degrees warranting further evaluation.

Keeping in mind the economics of using an additive in a commercial operation, a 2 per cent addition was selected as the maximum quantity acceptable. Consequently, tests were made using additions of 1.0 and 2.0 per cent of each of the additives except the fly ash.

Preparation of Materials

For each of the tests using 1 per cent additive, 3400 g (7.5 lb) of clay crushed to minus 16 mesh was used. For the tests using 2 per cent additive, to obtain more feed for the kiln, 3630 g (8 lb) was used. The method of preparing the plastic clay was the same as that used in the Stationary-Kiln Tests except that a Hobart, single-paddle mixer was used to mix the clay, additive, and water. Approximately 25 per cent of water was used to bring the clay to the plastic condition. After mixing, it was aged for 18 to 24 hr in a sealed polyethylene bag.

Pelletizing was done in a deairing International Vac-Air auger extrusion machine equipped with a multi-stream die. The deaired 3/8-in. streams of clay were cut into lengths of 1/4 to 3/4 in. by a cutter attached to the extrusion machine. Between 1 1/2 and 2 lb of the clay remained in the machine after each extrusion. This was cleaned out and discarded to prevent contamination of the next material. The pelletized materials were dried at 110°C(230°F).

Rotary-Kiln Firing

The rotary kiln (5-in. by 8 ft, inside) is fired by propane gas. The atmosphere in the kiln is oxidizing but the secondary air is restricted as much as is practical. The slope of the kiln was 1 in./ft and it rotated at 2.4 rpm. This gave the material a retention time of 10 to 15 min. The burner, which was

operated at a constant gas:air ratio, was aimed at the lining away from the bed of material. This prevented flame impingement which would over-heat the surface of the pellets. The temperature of the bed at the hottest zone was measured by an optical pyrometer. The material was fed to the kiln at a rate of between 10 and 15 lb/hr. For each test firing, 1/15 cu ft (approximately 4 lb) of material was used.

A preliminary test firing was made using the pelletized clay containing no additive. The temperature of the kiln was varied to determine the range, below the agglomerating temperature, through which appreciable bloating occurred. This was found to be 1115° to 1173°C (2040° to 2145°F).

All subsequent test firings were made at the approximate mid-point of this bloating range, 1150°C (2100°F).

Physical Properties

The volume expansion of the material during firing was measured. Each product was screened, crushed if required and recombined to result in the following grading:

40 per cent 1/2 to 3/8 in.
50 per cent 3/8 in. to 4 mesh
10 per cent 4 to 8 mesh

This grading meets ASTM specifications for 1/2-in. to 4-mesh lightweight aggregate.

The unit weight of each graded aggregate was determined by using a metal container (1/30 cu ft). The result reported is the average of four determinations.

The crushing strength of each graded aggregate was also assessed. For this determination, the graded aggregate was placed in a 3-in. steel cylinder to a depth of 5 in. A steel plunger was placed on the aggregate and pressure was applied. The crushing strength is represented by two figures: the pressures required to first compact the aggregate 1 in. and then through a total of 2 in. The bulk specific gravity and 24-hr absorption of the 1/2 to 3/8-in. uncrushed, coated aggregate from each product were also determined.

The firing temperatures and physical properties of the clay containing the different additives are shown in Table 2.

TABLE 2

Rotary-Kiln Firings and Physical Properties

| Additive | Firing Temperature (°F) | Volume Expansion (%) | Graded Unit Weight (lb/cu ft) | Crushing Strength (psi) | | Absorption (%) | Bulk Specific Gravity | |
|-------------------|-------------------------|----------------------|-------------------------------|-------------------------|------|----------------|-----------------------|-----|
| | | | | 1" | 2" | | | |
| Limestone | 1% | 2095 - 2110 | 15 | 50.3 | 1050 | 6100 | 11.2 | 1.4 |
| | 2% | 2095 - 2105 | 20 | 45.9 | 1090 | 4960 | 9.4 | 1.3 |
| Sulphur | 1% | 2090 - 2110 | 10 | 49.6 | 1120 | 6960 | 8.1 | 1.4 |
| | 2% | 2095 - 2100 | 15 | 49.0 | 1410 | >7000 | 8.1 | 1.4 |
| Sodium Silicate | 1% | 2095 - 2105 | 10 | 48.2 | 1040 | 5970 | 10.0 | 1.4 |
| | 2% | 2090 - 2105 | 15 | 48.0 | 1370 | 6220 | 8.0 | 1.4 |
| Gypsum | 1% | 2100 - 2110 | 10 | 47.6 | 1000 | 5850 | 11.1 | 1.4 |
| | 2% | 2090 - 2100 | 10 | 50.8 | 1270 | >7000 | 10.0 | 1.5 |
| None | | 2100 - 2110 | 25 | 47.5 | 990 | 5280 | 10.2 | 1.3 |
| Bindarene M | 1% | 2100 - 2110 | 20 | 45.9 | 930 | 5100 | 7.6 | 1.4 |
| | 2% | 2090 - 2120 | 45 | 38.3 | 670 | 2870 | 14.5 | 1.2 |
| Potash | 1% | 2100 - 2115 | 20 | 45.4 | 760 | 3610 | 6.4 | 1.3 |
| | 2% | 2095 - 2105 | 15 | 48.8 | 1090 | 5630 | 5.1 | 1.4 |
| Bindarene LA | 1% | 2100 - 2110 | 20 | 43.7 | 770 | 3750 | 10.9 | 1.3 |
| | 2% | 2100 - 2105 | 40 | 40.3 | 710 | 3010 | 12.8 | 1.2 |
| Flour | 1% | 2100 - 2105 | 25 | 42.9 | 800 | 3600 | 10.2 | 1.2 |
| | 2% | 2095 - 2100 | 25 | 44.9 | 1100 | 5370 | 10.9 | 1.3 |
| Sodium Carbonate | 1% | 2100 - 2120 | 25 | 42.7 | 880 | 3860 | 7.4 | 1.2 |
| | 2% | 2100 - 2050 | 25 | 46.5 | 880 | 4390 | 7.4 | 1.4 |
| Lignosol XD | 1% | 2100 - 2115 | 25 | 42.3 | 760 | 3540 | 9.7 | 1.3 |
| | 2% | 2090 - 2100 | 30 | 42.6 | 770 | 3430 | 11.3 | 1.3 |
| Potassium Sulfate | 2% | 2095 - 2105 | 5 | 54.7 | 1880 | >7000 | 8.7 | 1.6 |

Discussion of Results

In the series of tests where 1 per cent additive was used, 6 of the materials improved the bloating of the clay (resulting in lower unit weights), and 4 reduced it. The additives that improved the bloating were: Bindarene M, Bindarene LA, Lignosol XD, flour, potash and sodium carbonate. The materials that decreased the bloating were sulphur, gypsum, sodium silicate, and limestone.

By increasing the amount of additive from one to two per cent, bloating was further improved by Bindarene M and Bindarene LA. The higher quantity of potash, of flour, and of sodium carbonate decreased the bloating achieved with one per cent additions. The effect of Lignosol XD was about the same for one or two per cent additions.

Those materials which decreased the bloating, when using one per cent additions, showed no bloating improvement when 2 per cent of additive was used, with one exception that of the limestone additive.

The crushing strengths of the graded aggregates, in general, were related to the unit weights. In some cases, the strengths were somewhat higher than would be expected from the unit weight of the particular aggregate. This is probably due to the additive promoting increased reaction during heating thus giving a stronger ceramic bond within the pellets. This could be accomplished through deflocculation of the raw clay, resulting in denser pellets, and/or a fluxing action during firing. All the crushing strengths were comparable to or higher than some commercially produced lightweight aggregates.

Thermal Gravimetric Analysis

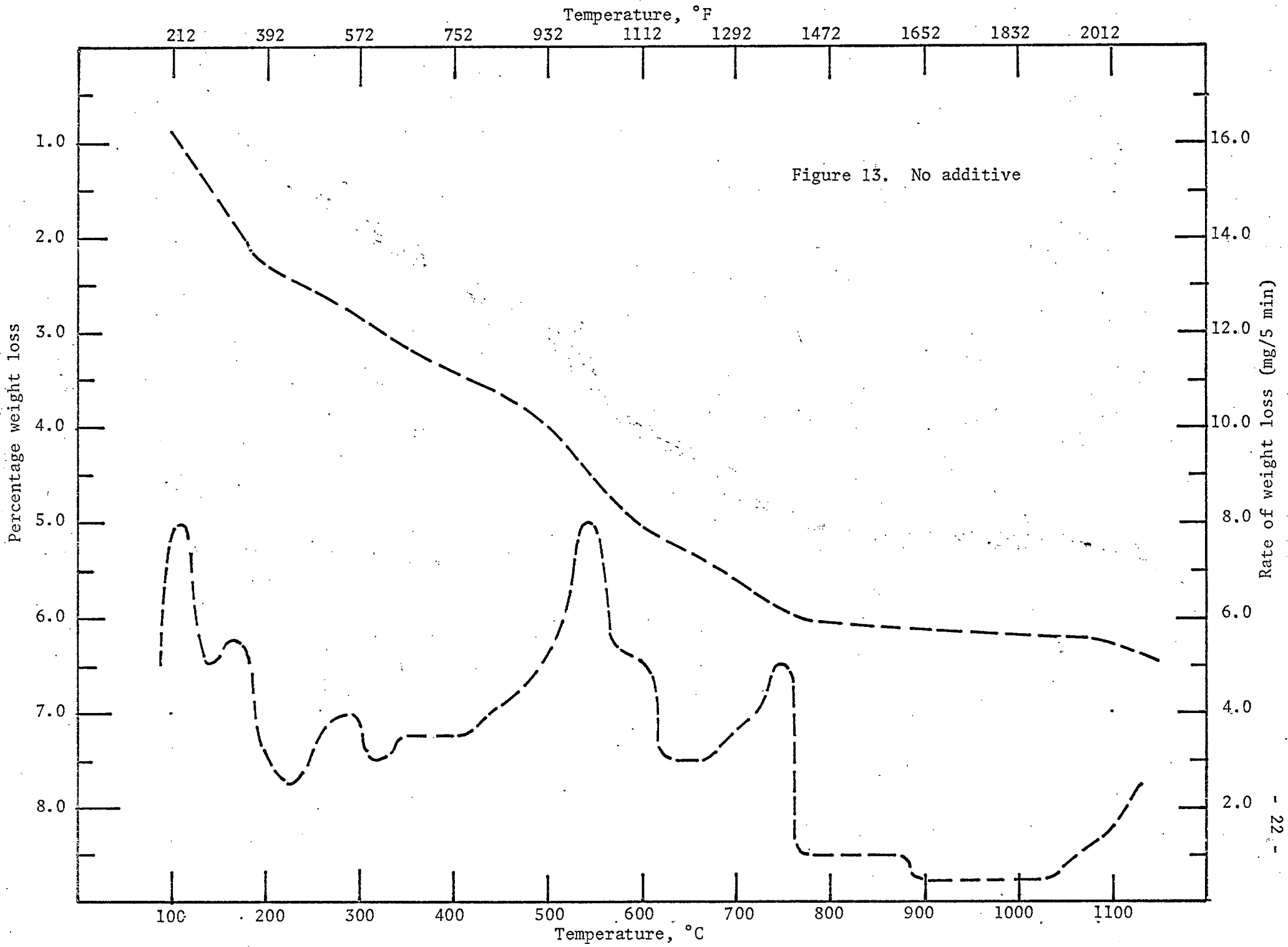
To understand better what happened to the various clay-additive mixtures in the kiln, thermal gravimetric analyses were made on each of the prepared materials. Tests were made in a Stanton thermo-balance. A few pellets of each of the prepared feeds for the rotary kiln were crushed to minus 8 mesh and representative 2.000-g samples were placed individually in the thermobalance. Each sample was heated at a rate of 6 Celcius degrees per minute to 1150°C (2102°F) during which time it was being continuously weighed; the sample weight was automatically recorded, providing a permanent record of weight change, during heating.

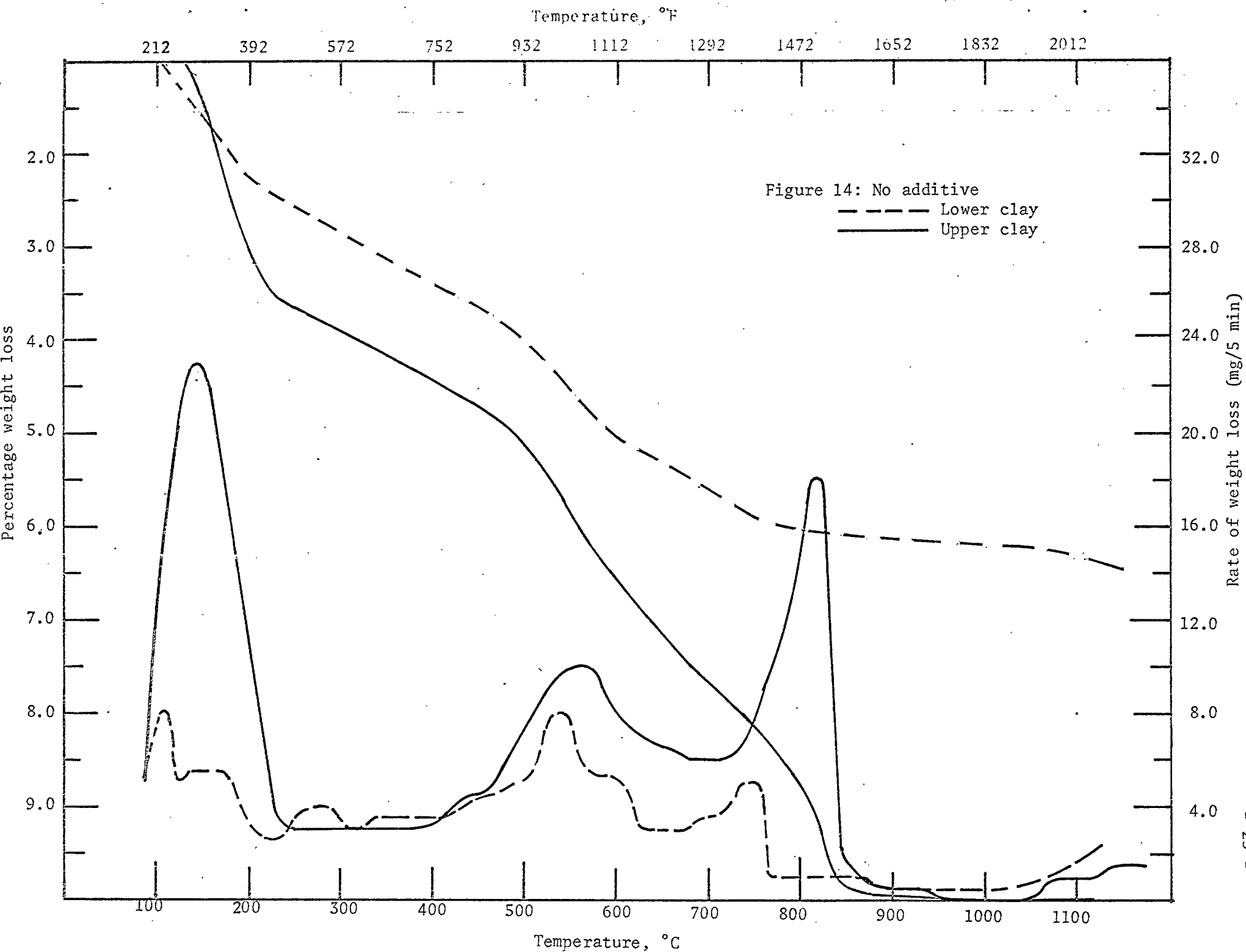
Each clay-additive mixture, the clay without additive, and the "upper" clay described on page 1 were analyzed in this manner. The weights recorded on the charts were re-plotted to show percentage weight loss at intervals of 50 Celcius degrees and rate of weight loss in mg/10 min at intervals of 60 Celcius degrees or at intermediate temperatures, if a change in rate was evident on the chart. These weight-loss curves are shown in Figures 13 to 25. The upper curves indicate percentage weight loss and the lower curves the rate of weight loss. It should be noted that Figure 14 is drawn to a different scale than the other figures. In each of the figures the rate of weight loss increases above 1000°C. This indicates that fusion had begun, probably accompanied by some volatilization of the melt formed.

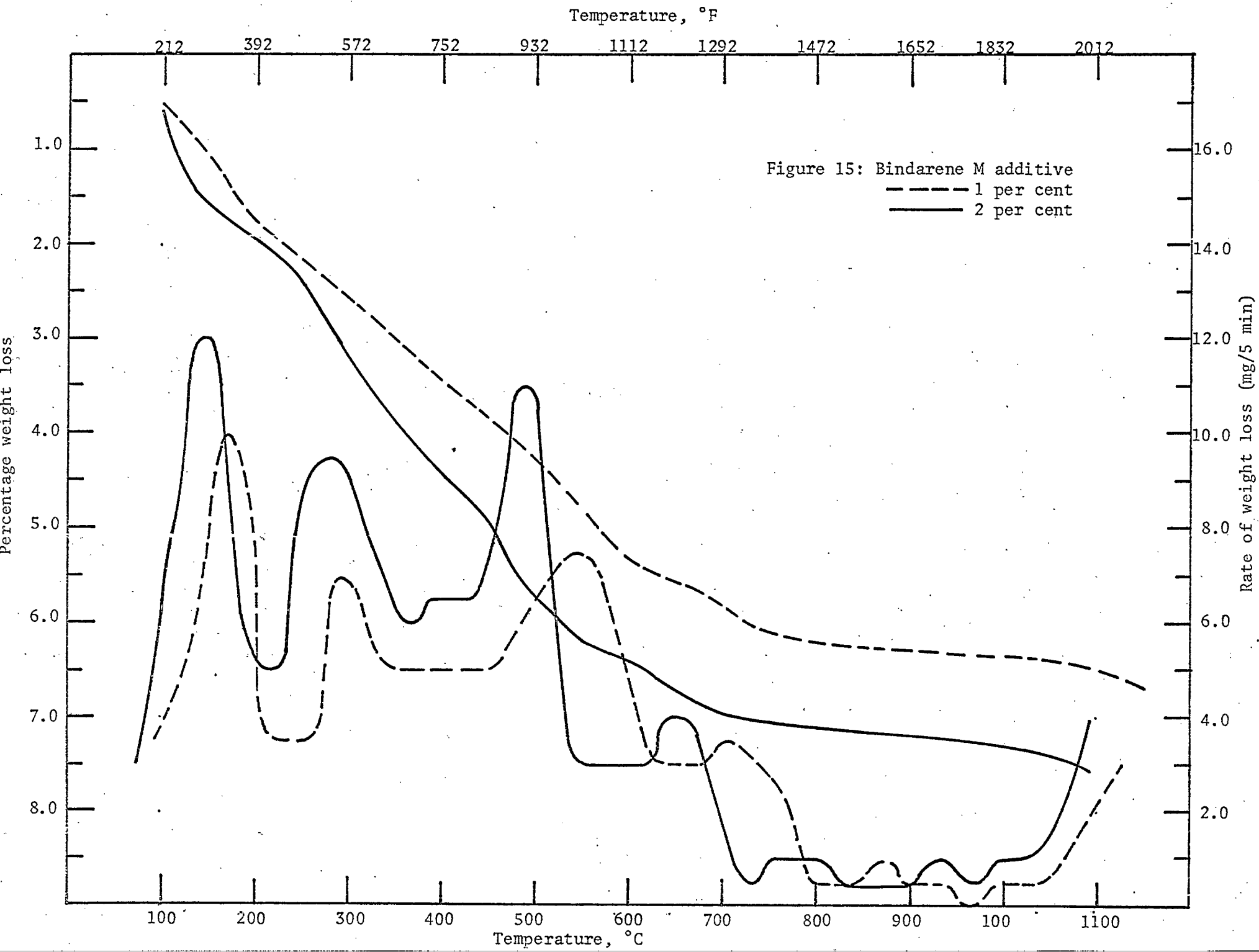
From Figure 14, it is evident that the significant differences between the upper and lower clays are one, a considerably greater total weight loss from the upper clay and, two, a higher rate of weight loss from the upper clay between 500° and 850°C (932° and 1562°F). At about 825°C (1517°F) the rate of weight loss is much higher from the upper clay than from the lower clay.

The range of total weight loss (excluding the weight loss above 1000°C) of the clay-additive mixtures which improved the bloating was 5.7 to 7.4 per cent with an average of 6.4 per cent. The range of the mixtures which decreased the bloating was 5.0 to 7.75 per cent with an average of 5.9 per cent. The range of mixtures that produced aggregates of unit weights less than 45.0 lb/cu ft was 5.7 to 7.4 with an average of 6.6 per cent. These figures show that gas evolution alone is not as effective as a combination of gas evolution and fluxing. The sulphur additive gave a weight loss of 7.75 per cent but decreased the bloating.

No definite relationship was evident for comparing the curves showing rate of weight loss of the clay-additive mixtures that improved bloating with those that did not.







Temperature, °F

212 392 572 752 932 1112 1292 1472 1652 1832 2012

Figure 16: Bindarene LA additive

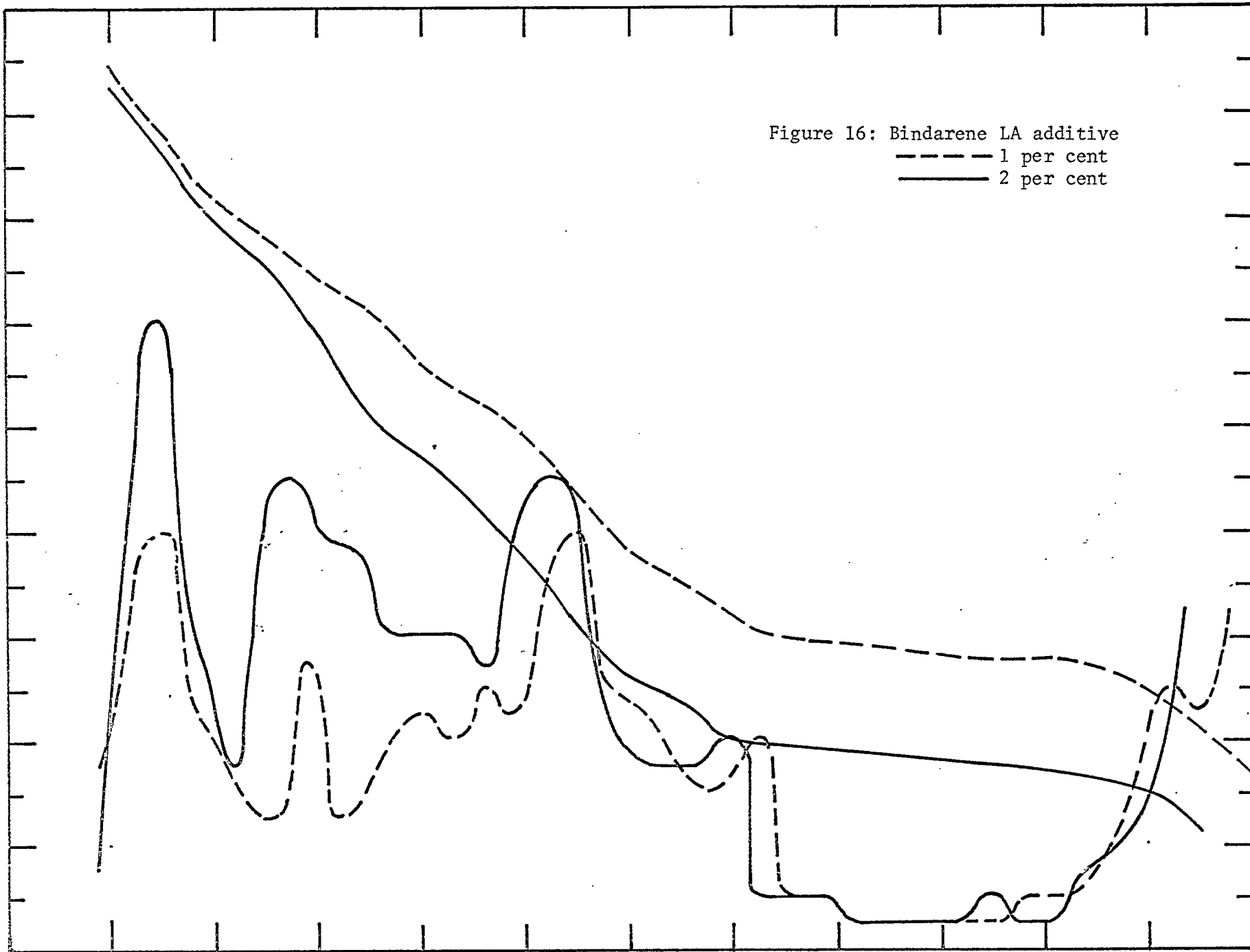
--- 1 per cent
— 2 per cent

Percentage weight loss

1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0

Rate of weight loss (mg/5 min)

16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0



Temperature, °C

100 200 300 400 500 600 700 800 900 1000 1100

Temperature, °F

212 392 572 752 932 1112 1292 1472 1652 1832 2012

Figure 17: Lignosol XD additive

--- 1 per cent
— 2 per cent

Percentage weight loss

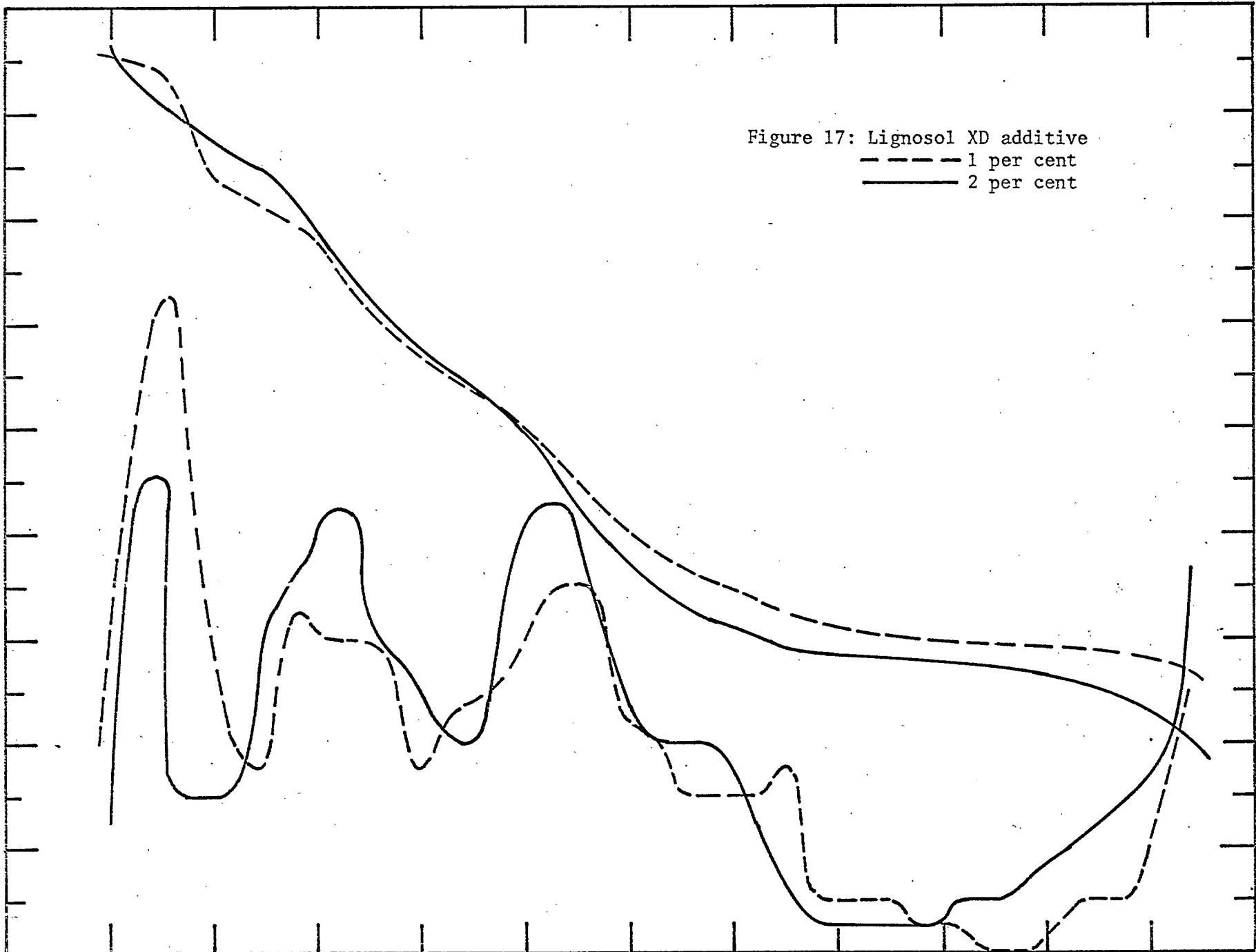
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0

Rate of weight loss (mg/5 min)

16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0

Temperature, °C

100 200 300 400 500 600 700 800 900 1000 1100



Temperature, °F

212

392

572

752

932

1112

1292

1472

1652

1832

2012

Figure 18: Sodium carbonate additive

--- 1 per cent

— 2 per cent

Percentage weight loss

1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0

Rate of weight loss (mg/5 min)

16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0

Temperature, °C

100

200

300

400

500

600

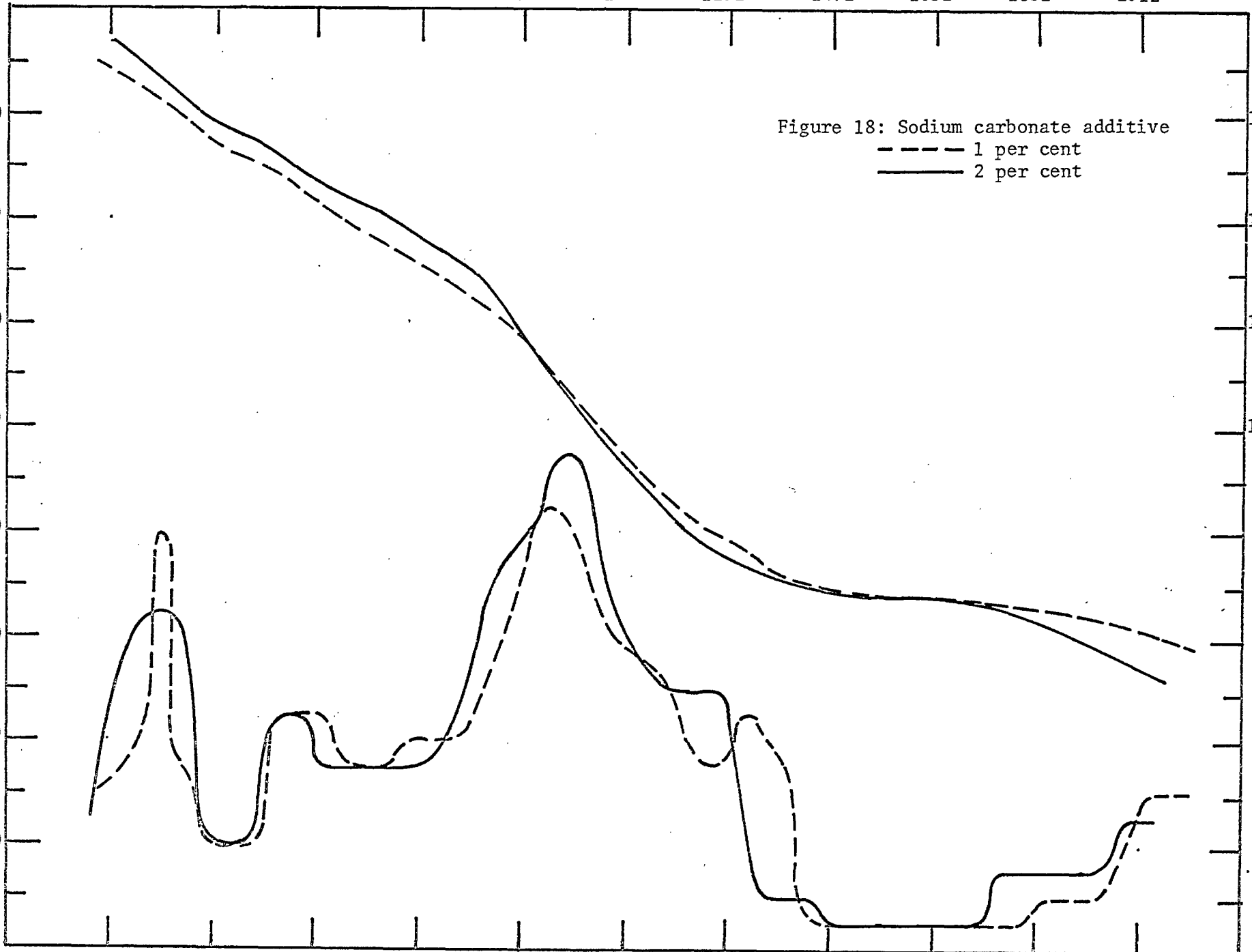
700

800

900

1000

1100



Temperature, °F

212 392 572 752 932 1112 1292 1472 1652 1832 2012

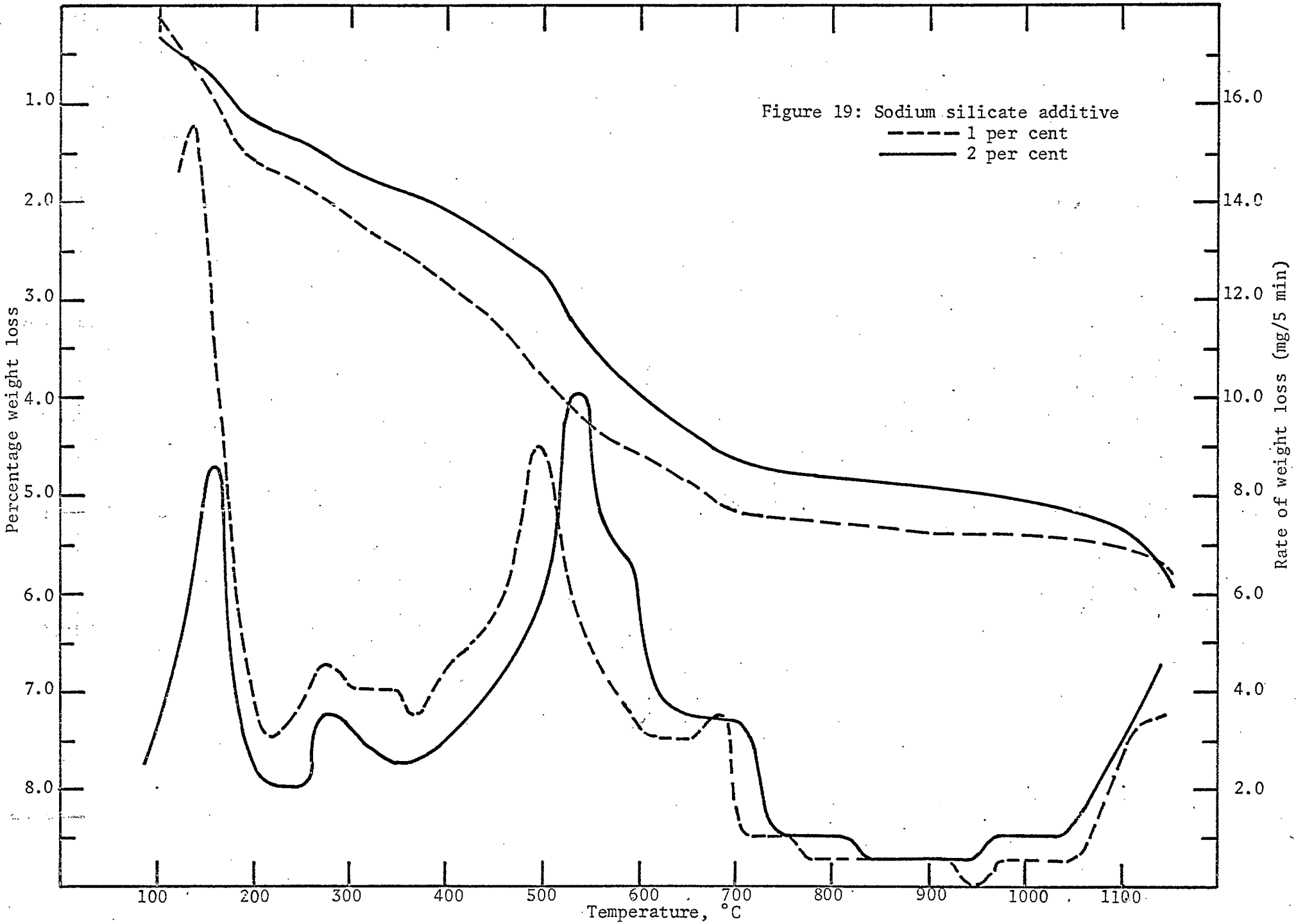
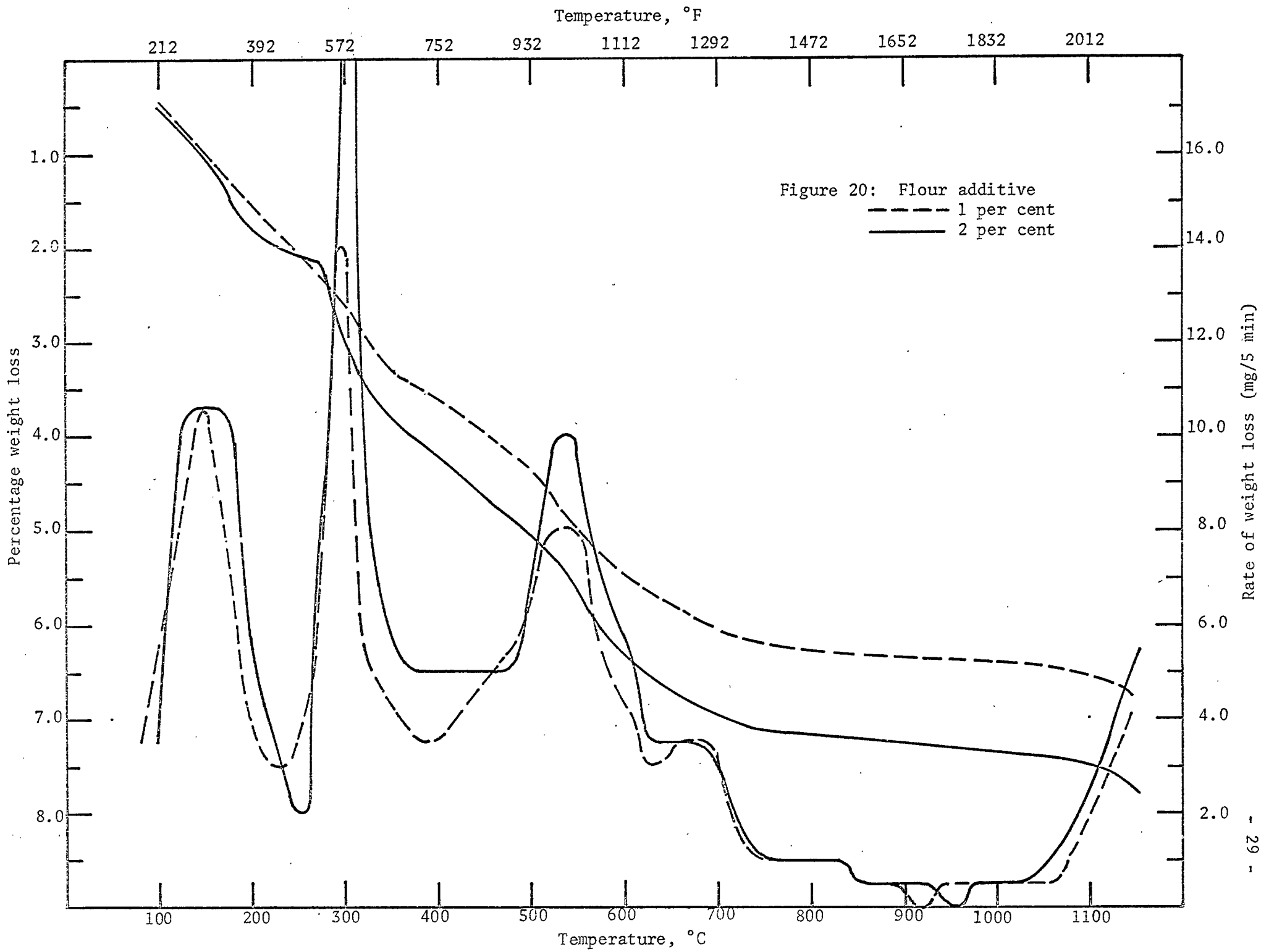
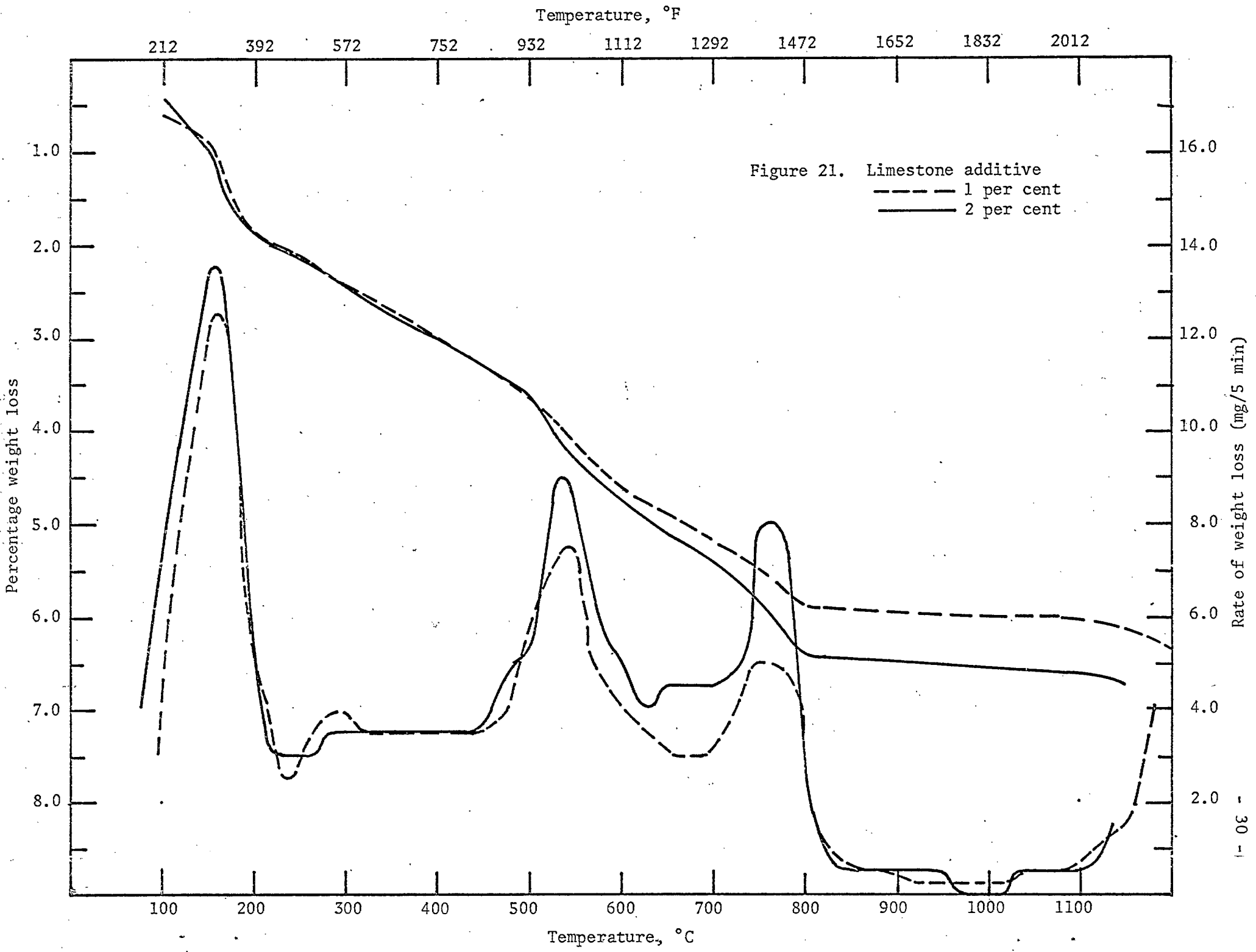
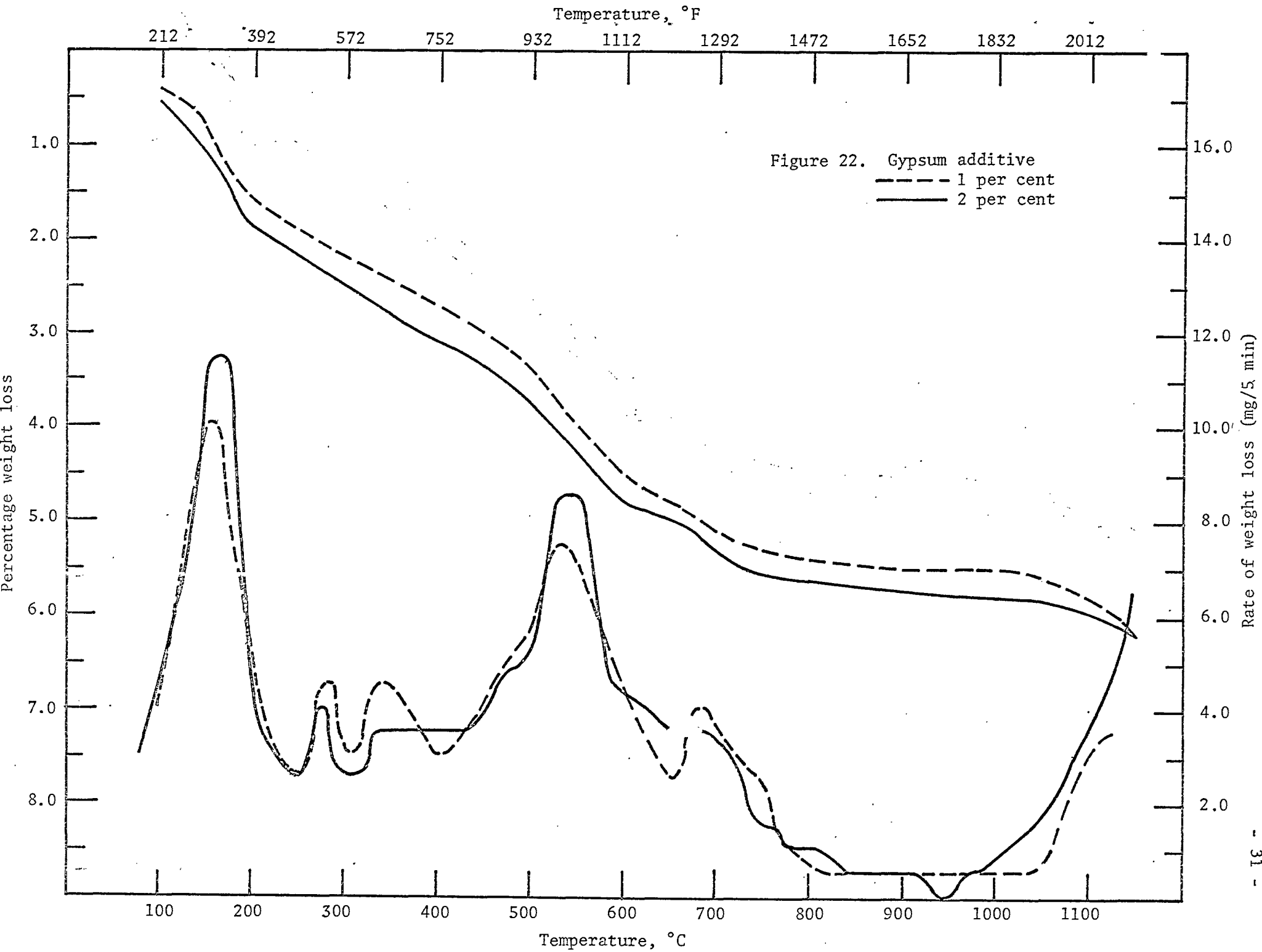


Figure 19: Sodium silicate additive

--- 1 per cent
— 2 per cent

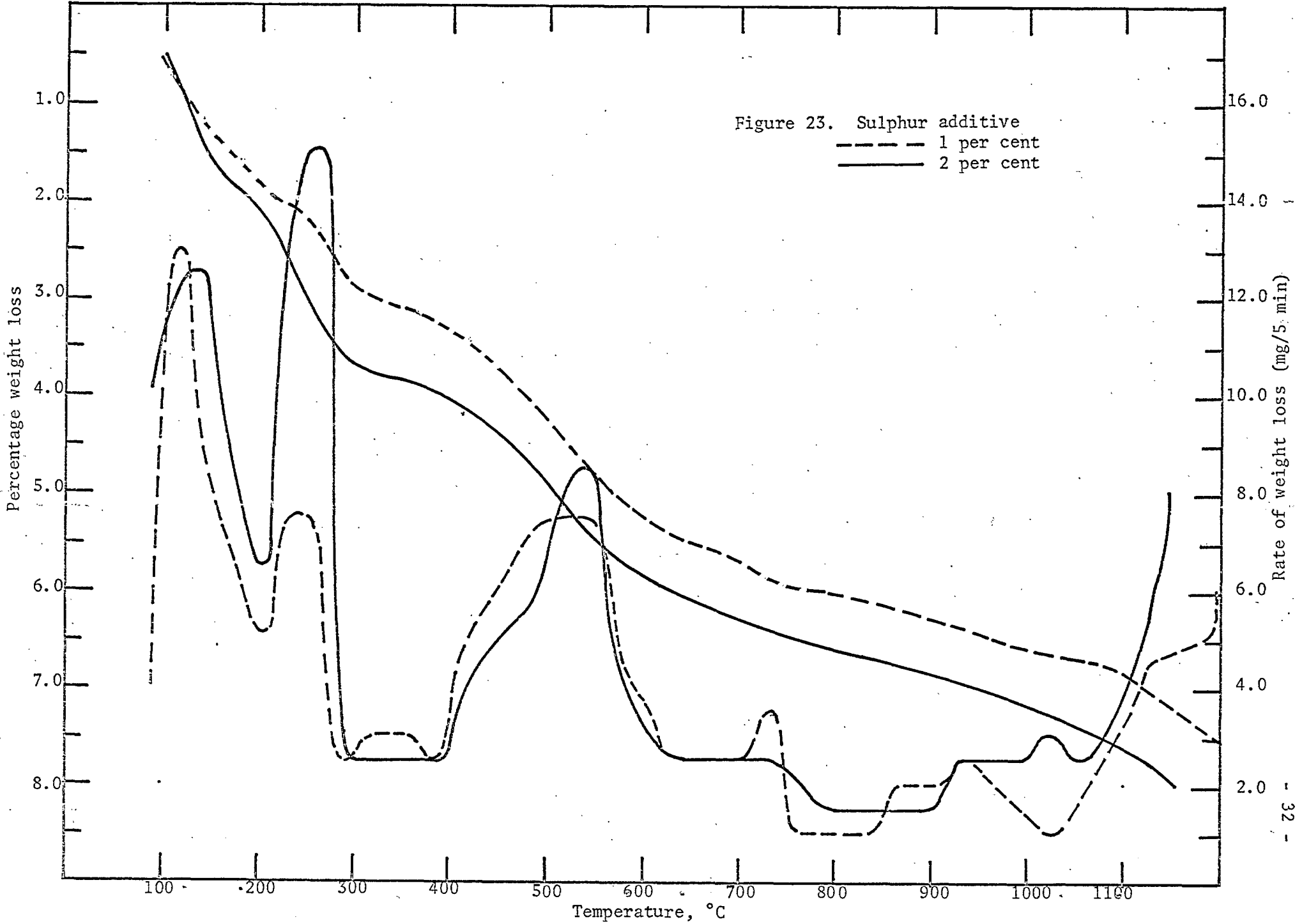






Temperature, °F

212 392 572 752 932 1112 1292 1472 1652 1832 2012



Temperature, °F

212 392 572 752 932 1112 1292 1472 1652 1832 2012

Figure 24: Potash additive

--- 1 per cent
— 2 per cent

Percentage weight loss

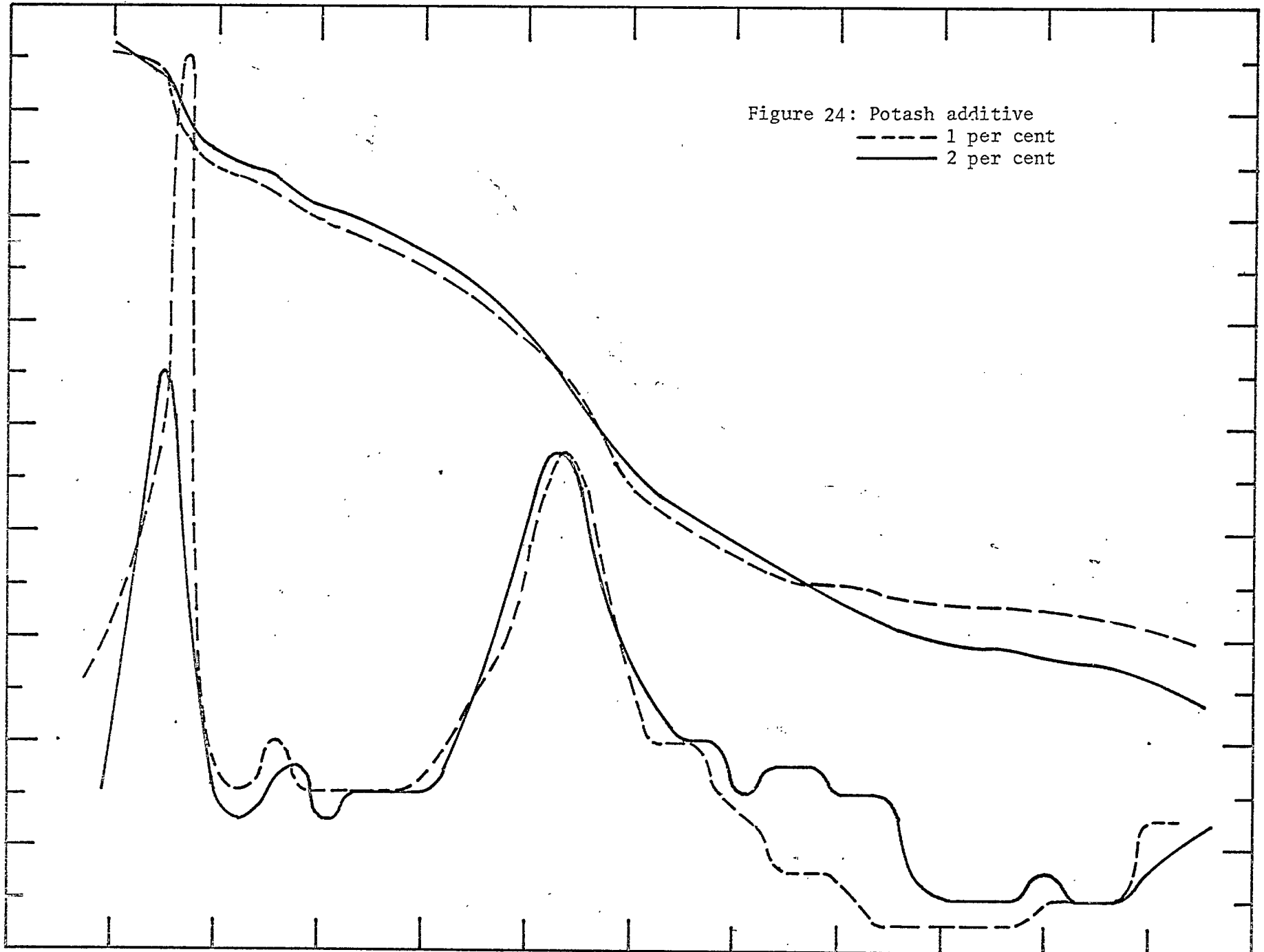
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0

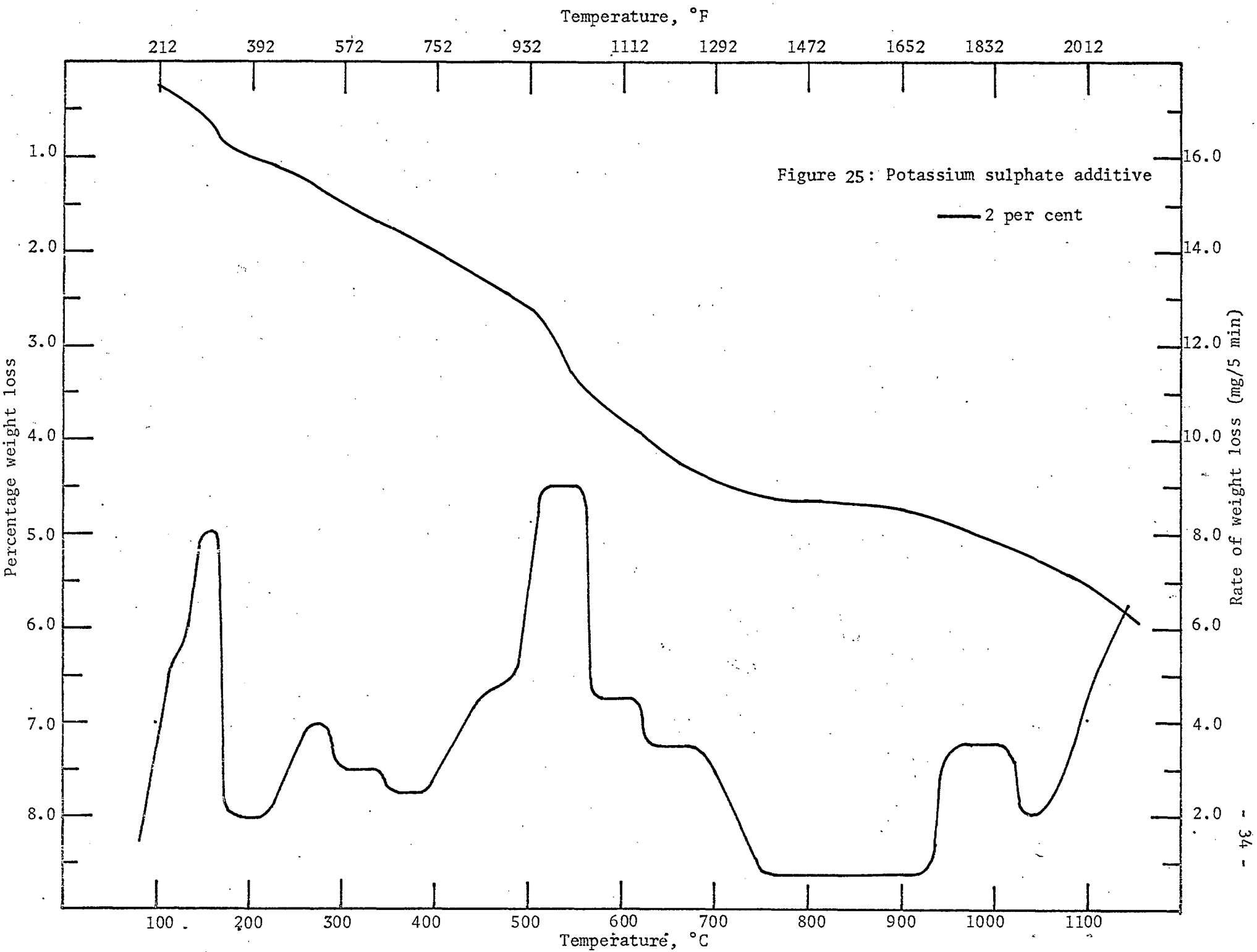
Rate of weight loss (mg/5 min)

16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0

Temperature, °C

100 200 300 400 500 600 700 800 900 1000 1100





CONCLUSIONS

Bloating quality of the "lower" clay is improved to various degrees by additions of: calcium-ammonium lignosulphonate (1 and 2 per cent), calcium lignosulphonate (1 and 2 per cent), sodium lignosulphonate (1 and 2 per cent), sodium carbonate (1 and 2 per cent), flour (1 and 2 per cent), potash (1 per cent) and limestone (2 per cent).

The unit weights of graded coarse lightweight aggregates produced from these clay-additive mixtures in the rotary kiln are between 38.3 and 46.5 lb/cu ft. The crushing strengths of aggregates are comparable to some commercially produced lightweight aggregates.

The higher the percentage of gas-producing material, the better the bloating, provided that the proper fluxes are present to form melts to contain the gases. The rate of gas evolution is not important.

Any future study to be made on this material should include tests to show the effect of using combinations of additives, e.g., materials selected to promote fluxing in combination with additives selected to liberate gases at appropriate temperatures.