

THERMOGRAVIMETRIC ANALYSIS OF CARBONATE AGGREGATE

**FINAL REPORT
IOWA HIGHWAY RESEARCH BOARD
PROJECT HR-336**

FEBRUARY 1994

Highway Division



**Iowa Department
of Transportation**

Final Report for
Iowa Highway Research Board
Research Project HR-336

THERMOGRAVIMETRIC ANALYSIS
OF
CARBONATE AGGREGATE

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February 1994

TECHNICAL REPORT TITLE PAGE

1. REPORT NO.	2. REPORT DATE
HR-336	2-94
3. TITLE AND SUBTITLE	4. TYPE OF REPORT & PERIOD COVERED
Thermogravimetric Analysis of Carbonate Aggregate	Final Report, 3-91 to 2-94
5. AUTHOR(S)	6. PERFORMING ORGANIZATION ADDRESS
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7. ACKNOWLEDGEMENT OF COOPERATING ORGANIZATIONS	
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8. ABSTRACT	
Research has shown that one of the major contributing factors in early joint deterioration of portland cement concrete (PCC) pavement is the quality of the coarse aggregate. Conventional physical and freeze/thaw tests are slow and not satisfactory in evaluating aggregate quality. In the last ten years the Iowa DOT has been evaluating X-ray analysis and other new technologies to predict aggregate durability in PCC pavement.	
The objective of this research is to evaluate thermogravimetric analysis (TGA) of carbonate aggregate. The TGA testing has been conducted with a TA 2950 Thermogravimetric Analyzer. The equipment is controlled by an IBM compatible computer. A TA Hi-Res™ software package allows for rapid testing while retaining high resolution.	
The carbon dioxide is driven off the dolomite fraction between 705°C and 745°C and off the calcite fraction between 905°C and 940°C. The graphical plot of the temperature and weight loss using the same sample size and test procedure demonstrates that the test is very accurate and repeatable. A substantial number of both dolomites and limestones (calcites) have been subjected to TGA testing. The slopes of the weight loss plot prior to the dolomite and calcite transitions does correlate with field performance. The noncarbonate fraction, which correlates to the acid insolubles, can be determined by TGA for most calcites and some dolomites. TGA has provided information that can be used to help predict the quality of carbonate aggregate.	
9. KEY WORDS	10. NO. OF PAGES
Aggregate Aggregate durability Aggregate quality Aggregate testing Thermogravimetric analysis	28

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DISCLAIMER

The contents of this report reflect the views of the author and do not necessarily reflect the official views of the Iowa Department of Transportation. This report does not constitute any standard, specification or regulation.

INTRODUCTION

The primary limitation of longevity of Iowa PCC pavement is premature deterioration near the contraction joints. Iowa Department of Transportation (Iowa DOT) research since the 1960's has identified the quality of the coarse aggregate as the major factor in the length of time until visible joint deterioration occurs in PCC pavement. Progress has been made in identifying aggregates susceptible to early joint deterioration.

Most of the current ASTM aggregate quality and durability tests are relatively old and do not effectively identify nondurable aggregate or durable aggregate for use in PCC pavement. Some durability tests require five months to complete. Quality aggregates are essential to longevity of PCC pavement. Motorists expect and deserve durable pavements.

Modern crushing equipment allows aggregate producers to greatly reduce the time needed between the crushing of the aggregate and its incorporation into asphalt or concrete projects. Adequate quality control of aggregate production requires test methods with short turn-around times.

All new technology should be considered and evaluated for the potential of providing information that may contribute to the determination of the durability of aggregate. In the last ten years, the Iowa DOT has begun using X-ray (1) analysis to predict

the durability of coarse aggregate for PCC pavement. This research has been highly successful in developing an X-ray diffraction method of identifying nondurable Iowa dolomitic aggregates. The durability correlates very well with the d-spacing of the maximum intensity dolomite peak. A d-spacing of less than 2.890 would generally indicate a high quality dolomitic aggregate unless it contained a significant amount of trace element such as manganese. A d-spacing greater than 2.900 would identify a carbonate with poor performance in PCC pavement. X-ray fluorescence has also identified trace elements (in excess of selected levels) that are associated with nondurable aggregate.

Iowa DOT research has shown that relatively high amounts of some trace elements (phosphorus and strontium) are often present in nondurable limestone PCC pavement aggregate (1).

Research is needed to evaluate other new technologies to determine if there are any that could be used as an indicator of PCC aggregate durability.

OBJECTIVE

The objective of this research is to determine if TGA can be used to evaluate carbonate aggregate for use in PCC pavement. The purpose of this paper is to present the findings of the carbonate aggregate TGA study.

EQUIPMENT AND COMPUTER PROGRAM

The Iowa DOT has made arrangements with Iowa State University to use their thermogravimetric equipment for this research. Iowa State University has just recently purchased a TA 2950 Thermogravimetric Analyzer (Figure 1). The system operates on a null-balance principle, using a highly sensitive transducer coupled to a taut-band suspension system to detect minute changes in the weight of a sample. Heating rate and sample temperature are measured by a thermocouple located immediately adjacent to the sample. Finely ground carbonate aggregate samples are placed in platinum sample pans. The operator presses the "Start" key to begin an automatic sequence of events: rotation of the sample platform, automatic sample pickup, raising of the furnace and initiation of the software experimental conditions and procedures. The progress of the test can be monitored on a display screen. The TA 2950 is controlled by an IBM compatible computer.

TA Instruments has developed a Hi-Res™ software package for use with the TA 2950. In the past, it has been necessary to use slow heating rates to improve the identification of transitions and thus increase the resolution of the TGA plot. This required substantial testing time. The Hi-Res™ software makes possible rapid heating during periods when no weight loss occurs. When the computer notes a small weight loss, the computer reduces to a slower heating rate. This yields improved resolution of critical

periods. The software and computer continually record temperature and weight. The Hi-Res™ software substantially reduces testing time and manual monitoring while retaining improved resolution.

TEST PROCEDURE

The current test procedure utilizes a 54 to 57 milligram sample contained on a platinum sample pan that is suspended on a micro scale. After the oven surrounds the sample, carbon dioxide gas flows across the chamber. The oven is rapidly brought to 300°C (Figure 2) where the time and temperature recording begins. The sample is then heated at 40 degrees per minute until weight loss occurs. When weight loss occurs, the heating rate is reduced. Weight loss of the nonargillaceous Iowa carbonates may begin as low as 350°C. Loss of carbon dioxide from the dolomite fraction may begin as low as 570°C but the majority is burned off between 705°C and 745°C (dolomite transition). A pure limestone (no dolomitic fraction) may begin to burn off (lose carbon dioxide) at 700°C. The majority of the limestone (calcite) will burn off between 905°C and 940°C (calcite transition). The temperature and weight changes are internally stored. The data is processed and graphed at a later time. Testing of an individual carbonate sample typically requires from 40 to 90 minutes depending upon the number of transitions encountered.

ANALYSIS OF LIMESTONE AGGREGATES

The Iowa DOT specifications for coarse aggregate for use in concrete include a maximum loss of 6 percent on a water-alcohol freeze/thaw test of the aggregate. This effectively prevents the use of argillaceous aggregates (those containing more than 5% clay). Most of the aggregates (both calcites and dolomites) included in this research contain less than 5% clay.

A typical plot of a very durable limestone (Moberly) is shown in Figure 3. There is very little weight loss prior to the CaCO_3 transition which is greater than 900°C . A durable pure limestone (non-argillaceous calcite) typically loses little weight until it reaches its transition temperature (greater than 905°C). A summary of the limestone TGA test results are given in Table 1.

A nondurable limestone such as Crescent (Figure 4) begins to lose weight at approximately 600°C . This slope prior to the CaCO_3 transition yields the best correlation (of TGA data) with service life of portland cement concrete made with limestone aggregates. The durable PCC aggregates with 40-year service will have little pre-transition slope while those with poor service records will have greater pre-transition slopes. At this time, the slope prior to the CaCO_3 transition yields the best correlation (of TGA data) with field performance. The slope prior to the calcite transition was determined between 130°C and 30°C less than the computer determined calcite transition. Tests on argillaceous

materials show that the slope of the plot is substantially affected by the noncarbonate fraction in the sample.

ANALYSIS OF DOLOMITIC AGGREGATE

As noted in the introduction, the X-ray diffraction peak shift has been effective in identifying some nondurable dolomitic aggregates. Evenly distributed micron sized pyrite and/or manganese substituting for Magnesium are found in dolomitic aggregates associated with poor service record, particularly when deicing salt is used. The manganese-dolomites usually do not show a shift. In Iowa, the coarse grained dolomites are associated with a superior (40 years) service record. The service records associated with quarries operating out of the Hopkinton formation in northeastern Iowa attest to this observation. These coarse grained dolomites score high when tested by all conventional durability tests and also score well on the newer chemical durability tests. The medium grained dolomites are associated with an intermediate (15 to 25 years) service record. Dolomites from the Cedar Valley and Gower units are examples of this type of material. These medium grained dolomites score high on conventional durability tests and score lower on the chemical durability tests but not as low as the service record would indicate. The fine grained dolomites are associated with a poor (10 to 15 year) service record. As would be expected, fine grained, relatively pure dolomites are rare, however, the Otis unit in Linn County is an example of this

material that was used extensively in PCCP. These fine grained dolomites score low on both conventional and chemical durability tests, but not as low as the service records would indicate.

An alternative explanation (as opposed to initial freeze/thaw deterioration) for the relationship of grain size to service record could relate to chemical reactivity prior to freeze/thaw activity. Previous work has shown a strong correlation between grain size and pore size. Fine and medium grained dolomites have an extensive capillary-sized pore system that allows brine to come into contact with the larger surface area of the smaller grains. Other work at the Iowa DOT has shown that most often the chemical reactivity is most severe at the grain boundaries, including the area where the grains interlock. The distance of the interlock from one pore (brine filled?) to another is directly related to the grain size of the aggregate particle, therefore, small grains will become detached quicker than large grains when chemical reactivity occurs even if the bulk chemistry of the two is identical.

Scott Schlorholtz's research (3) using thermogravimetric analysis has shown that grains are subdivided into crystallites. As would be expected, chemical reactivity appears to relate to the number and size of these crystallites. TGA pre-transition slopes relate to crystallite size and can be used in the main formula used to calculate overall carbonate aggregate chemical quality. A summary of the dolomitic TGA test results is given in Table 2.

In general, the TGA of a durable dolomitic aggregate has shown that the carbon dioxide begins to exit at around 570°C (Figure 2). It continues to lose weight with a steeper sloping plot until it reaches a temperature greater than 705°C where the balance of the magnesium carbonate is changed to magnesium oxide. The carbon dioxide from the calcium carbonate is driven off at greater than 905°C.

With the poor durability dolomitic aggregate (Figure 5), there is very little weight loss until above 700°C where the carbon dioxide (from the magnesium carbonate) is driven off. Again the carbon dioxide (from the calcium carbonate) is driven off at greater than 905°C. For a dolomitic aggregate (greater than 5% MgCO₃) the slope prior to the MgCO₃ transition yields the best correlation (using TGA data) with field performance. The slope prior to the dolomite transition was determined between 100°C and 50°C less than the computer determined dolomite transition.

DETERMINATION OF NONCARBONATE FRACTION

The TGA test procedure can be used to determine the percent of dolomite, calcite and noncarbonate in many carbonate aggregates. The noncarbonate fraction should equate to acid insolubles. Some states (Minnesota and Oklahoma) use the percent insolubles as a factor in determining aggregate quality for asphalt as well as PCC pavements. TGA may provide a more rapid method of determining equivalent insolubles. Using atomic weight

conversion factors, the following formula has been developed for the Iowa DOT TGA test method to determine calcite, dolomite and noncarbonate:

$$\text{Percent Dolomite (D)} = 4.19 (100 - R_D)$$

$$\text{Percent Calcite (C)} = 2.27 (R_D - R_C) - 0.56D$$

$$\text{Percent noncarbonate} = 100 - (D+C)$$

R_D - Residue at the bottom of the dolomite transition.

R_C - Residue at the bottom of the calcite transition.

There are some large grained dolomites that lose material over the edge of the sample pan due to micro explosions. This formula will not work for those aggregates unless the dolomite is ground extremely fine so that it will stay in the sample pan during heating. In addition, some carbonates may contain hydrous minerals such as gypsum. Weight loss associated with these hydrous minerals would need to be considered when determining percentages of the other compounds.

CORRELATION OF TGA DATA WITH FIELD PERFORMANCE

This research, along with the X-ray analysis, is an effort by the Iowa DOT to relate results of rapid new testing technology to field performance. TGA has been conducted on over 800 carbonate samples. A corresponding field performance is available for 49 of these samples. The TGA data for these samples are given in

Tables 1 and 2. The tables are tabulated in order of field performance from 40 years first to 10 years last. Initially, the values were checked to see if the transition temperatures would relate to field performance. A visual review shows no relationship.

The pre-transition slopes do relate to field performance. In general, a flat slope ahead of the calcite transition relates to good performance of the aggregate in PCC pavement. Conversely, a steeper slope relates to poorer performance. A correlation of 49 TGA slopes prior to the calcite transition yielded a coefficient of determination of $r^2 = 0.45$. If only those carbonates that are 95% or more calcite are included ($N=22$), a curvilinear correlation (Figure 6) yields a coefficient of determination, r^2 of 0.83 which shows a good correlation.

If the slope prior to the dolomite transition is used for carbonates that are 95% or more dolomite (Figure 7), a coefficient of determination, r^2 for a curvilinear fit is 0.82, which again shows a good correlation.

These correlations are good enough to be an indication of how a carbonate will perform in PCC pavement. It is the intent of the Iowa DOT to use this data along with pore size distribution data and X-ray analysis data to predict the durability of carbonates in PCC pavement.

PRE-CALCITE TRANSITION SLOPE RELATIONSHIP TO NONCARBONATE FRACTION

The calcite pre-transition slope correlates with the noncarbonate fraction. This is true for both calcites and dolomites. The noncarbonate fractions were determined by both TGA and X-ray fluorescence (XRF).

The correlations (r Spearman) are as follows:

SAMPLES CONTAINING 90% OR MORE DOLOMITE

	<u>N</u>	<u>r^2</u>
XRF noncarbonates vs. TGA noncarbonates.....	291	0.10
TGA calcite pre-transition slope vs. XRF noncarbonate fraction..	291	0.53
TGA calcite pre-transition slope vs. TGA noncarbonate fraction..	291	0.21
PCCP service record vs. XRF noncarbonate fraction.....	525	0.01

SAMPLES CONTAINING 90% OR MORE DOLOMITE (EXCLUDING COARSE GRAINED)

XRF noncarbonates vs. TGA noncarbonates.....	153	0.29
TGA calcite pre-transition slope vs. XRF noncarbonate fraction..	153	0.66
TGA calcite pre-transition slope vs. TGA noncarbonate fraction..	153	0.31

SAMPLES CONTAINING 90% OR MORE CALCITE

XRF noncarbonates vs. TGA noncarbonates.....	208	0.83
TGA calcite pre-transition slope vs. XRF noncarbonate fraction..	208	0.85
TGA calcite pre-transition slope vs. TGA noncarbonate fraction..	208	0.72
PCCP service record vs. XRF noncarbonate fraction.....	433	0.48

From the above correlations it can be seen that: 1) The calcite pre-transition slope does correlate with the noncarbonate fraction (acid insolubles), however, the correlation is much better for calcites than dolomites. 2) Some of the coarse grained dolomites lose some of their material over the edge of the sample pan during heating but even when the coarse grained dolomites are excluded, they do not correlate to the noncarbonate fraction as well as the calcites. 3) In all cases, the calcite

pre-transition slope correlated better with XRD noncarbonate fraction than the TGA noncarbonate fraction. This is to be expected since XRF calculations are considered much more accurate.

DISCUSSION OF TGA

TGA analysis is not new. Much of the earlier work used differential thermal analysis (DTA). Apparently there has been minimal use of TGA to analyze carbonate aggregate (2). Earlier TGA equipment was difficult to operate and not suited for production type of work. Improved TGA equipment can handle a large number of samples at a reasonable cost per sample with a high degree of accuracy.

Test methodology has an influence on the weight loss and temperatures obtained from the analysis. If the sample size, rate of heating and test method are held constant, the analysis is very accurate and repeatable (Table 3).

Iowa State University, under the direction of Scott Schlorholtz, conducted a related study of carbonate aggregate which includes TGA (3). X-ray analysis was used in conjunction with TGA and scanning electron microscopy in an effort to explain the relationships between chemistry, crystallite size, and TGA test results of carbonate aggregates.

CONCLUSIONS

This research on TGA of carbonate aggregate supports the following conclusions:

1. Current TGA equipment is capable of rapid, accurate, and repeatable analysis of carbonate aggregate.
2. A standardized test method is necessary to obtain repeatable results.
3. The TGA slopes prior to the calcite and dolomite transition yield a good correlation with field performance of carbonates in PCC pavement.
4. The noncarbonate fraction correlates with PCCP service record for calcites but not dolomites. The noncarbonate fraction as calculated by XRF is more accurate, and when available, should be used rather than the TGA calculation.
5. The calcite pre-transition slope correlates to the noncarbonate fraction for both dolomites and calcites, however, the calcite correlation is much stronger.
6. When evaluating coarse grained dolomites by TGA, weight losses larger than expected from the loss of carbon dioxide gas may occur due to microexplosions that cause some of the sample to be lost over the edge of the sample pan.
7. TGA exhibits a potential for characterization of carbonate aggregate. The dolomite pre-transition slope can be used for characterizing dolomites. The calcite pre-transition slope and/or percent noncarbonate can be used to characterize the calcites. TGA test results combined with XRF, XRD and pore index test results can give a rapid evaluation of carbonate aggregate intended for use in PCCP. For routine daily quality control, only one of the tests would need be run once the parameters of the working ledge were established. Test results could be available in less than one day when using TGA, XRF, XRD and pore index equipment.

ACKNOWLEDGEMENTS

This research was conducted and funded as Highway Research Advisory Board project HR-336. The authors wish to express their

appreciation to Turgut Demirel, Scott Schlorholtz, Jerry Amenson and Ken Bergeson of Iowa State University for their assistance in the TGA of carbonate aggregate and in the interpretation of the data.

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TABLE TITLES

1. TGA Test Result Summary for Limestone Aggregates
2. TGA Test Result Summary for Dolomitic Aggregate
3. Repetitive Analysis of Lisbon Carbonate

TABLE 1
TGA Test Result Summary for Limestone Aggregates

<u>Sample Identification</u>	<u>Years to Visible Deterioration</u>	<u>Calcite Loss Temperature °C</u>	<u>Residue at 940°C Percent</u>	<u>Slope Prior to Calcite Transition \$/100°C</u>
Alden 506	40	926.74	56.13	0.175
Ft. Dodge 2258	40	927.40	56.05	0.150
Gilmore City 98	40	928.79	56.13	0.110
Hodges 420	40	931.18	56.10	0.135
LeGrand 965	40	919.34	56.62	0.102
Moberly 649	40	929.24	56.11	0.076
Montour 349	40	916.42	56.63	0.121
Waucoma 1287	40	929.99	56.30	0.235
Gilmore City 3378	30	937.26	56.22	0.112
Klein 110	30	925.44	56.35	0.140
Linwood 890	30	924.85	56.43	0.242
Quimby 1466	30	923.51	56.01	0.190
Stevens 440	30	928.83	56.32	0.233
Sugar Beet 3096	30	927.82	56.40	0.251
Conklin 224	25	908.93	56.86	0.318
Hovey 1301	25	919.63	56.65	0.532
Stewartville 316	20	917.97	57.08	0.658
Weeping Water 799	20	923.00	57.25	0.720
Crescent 1315	10	921.18	57.40	1.396
Early Chapel 878	10	922.48	57.61	1.278
Stanzel 716	10	927.51	56.84	1.397
Ullin, IL 1580	10	922.86	57.45	0.631

TABLE 2A
TGA Test Result Summary for Dolomitic Aggregate

<u>Sample Identification</u>	<u>Years to Visible Deterioration</u>	<u>Dolomite Loss Temperature °C</u>	<u>Residue at 745°C Percent</u>	<u>Slope Prior to Dolomite Transition %/100°C</u>	<u>Calcite Loss Temperature °C</u>	<u>Residue at 940°C Percent</u>	<u>Slope Prior to Calcite Transition %/100°C</u>
Aurora 793	40	730.37	78.21	3.153	919.04	53.59	0.817
Gassman 2471	40	709.42	76.27	5.836	902.03	53.04	1.024
Lamont 983	40	726.74	74.57	4.124	920.13	51.28	0.641
Lisbon 214	40	719.90	76.65	5.294	918.56	52.02	0.478
Maryville 875	40	721.64	75.67	4.628	919.85	51.96	0.607
McCausland 3026	40	731.29	74.36	6.103	917.72	50.53	0.400
McGuire 3098	40	722.72	76.55	3.819	917.52	52.01	0.448
New Liberty 3286	40	717.22	74.92	4.578	903.57	50.86	0.452
S. Cedar Rapids 2385	40	716.40	76.40	6.158	916.51	51.96	0.489
Sedgewick 1843	40	719.74	76.99	4.922	916.50	52.37	0.440
Brown 2611	30	722.89	74.77	3.309	901.06	52.48	1.019
McCausland 3021	25	735.51	78.22	2.238	919.79	52.97	0.707
LeClaire 2579	20	741.79	77.07	2.334	918.43	52.58	1.104

TABLE 2B
TGA Test Result Summary for Dolomitic Aggregate

<u>Sample Identification</u>	<u>Years to Visible Deterioration</u>	Dolomite Loss Temperature °C	Residue at 745°C Percent	Slope Prior to Dolomite Transition %/100°C	Calcite Loss Temperature °C	Residue at 940°C Percent	Slope Prior to Calcite Transition %/100°C
Little River 1232	20	733.10	77.19	1.464	918.71	54.74	1.243
Peske 839	20	738.27	81.08	1.703	914.79	54.39	1.403
Portland 1405	20	717.22	79.17	1.275	901.79	53.93	1.920
Stevens 436	20	738.53	81.09	1.908	915.85	53.86	1.126
Bryan 1228	15	719.52	77.03	2.056	916.48	54.99	1.765
Elkader 1092	15	738.61	81.63	1.817	914.10	56.11	1.525
Grand Meadow 282	15	727.87	80.38	1.390	901.53	55.24	1.107
Jabens 944	15	736.92	80.10	2.107	915.82	52.90	1.483
LeGrand 958	15	730.77	82.94	0.890	905.98	54.03	0.628
Osterdock 1497	15	744.25	82.60	1.553	920.08	54.79	0.965
Paralta 3472	15	723.40	77.83	2.125	905.08	53.10	0.588
Pints 719	15	740.28	79.27	1.613	915.82	52.80	0.726
Smith	15	735.86	80.50	1.445	917.87	53.80	0.944
Ames 53	10	726.22	84.43	0.644	912.71	58.73	2.566
Stanzel 712	10	730.11	96.37	0.166	920.56	56.33	1.397

TABLE 3
Repetitive Analysis of Lisbon Carbonate

Run No.	Sample Size mg	Dolomite Loss Temperature °C	Residue at 800°C mg	Calcite Loss Temperature °C	Residue at 950°C mg
1	20.591	714.53	75.92	920.64	51.49
2	20.431	713.86	76.05	920.57	51.65
3	20.127	714.17	75.60	920.38	50.77
4	20.457	714.40	76.02	920.27	51.63
5	20.466	<u>714.53</u>	<u>76.28</u>	<u>920.23</u>	<u>51.78</u>
Standard Deviation		0.29	0.25	0.18	0.40
1	55.812	720.00	76.70	919.89	52.07
2	55.195	717.74	76.74	920.12	52.15
3	55.314	718.48	76.75	919.91	52.12
4	54.892	719.14	76.55	919.67	51.95
5	55.304	719.20	76.57	919.52	51.82
6	55.351	717.33	76.58	918.98	51.98
7	55.612	<u>719.88</u>	<u>76.65</u>	<u>918.55</u>	<u>52.02</u>
Standard Deviation		1.02	0.08	0.56	0.11

FIGURE CAPTIONS

1. Schematic Drawing of the TA Instruments 2950 Thermogravimetric Analyzer
2. Computer Plot of a Good Quality Lisbon Dolomitic Aggregate
3. Computer Plot of a Good Quality Moberly Limestone (Calcite) Aggregate
4. Computer Plot of a Poor Quality Crescent Limestone Aggregate
5. Computer Plot of a Poor Quality LeGrand Dolomitic Aggregate
6. Limestone Service Life Vs. Slope Prior to the Calcite Transition
7. Dolomite Service Life Vs. Slope Prior to the Dolomite Transition
8. Computer Plot of a Landis Dolomite

Figure 1 – Schematic Drawing of the TA Instruments 2950 Thermogravimetric Analyzer

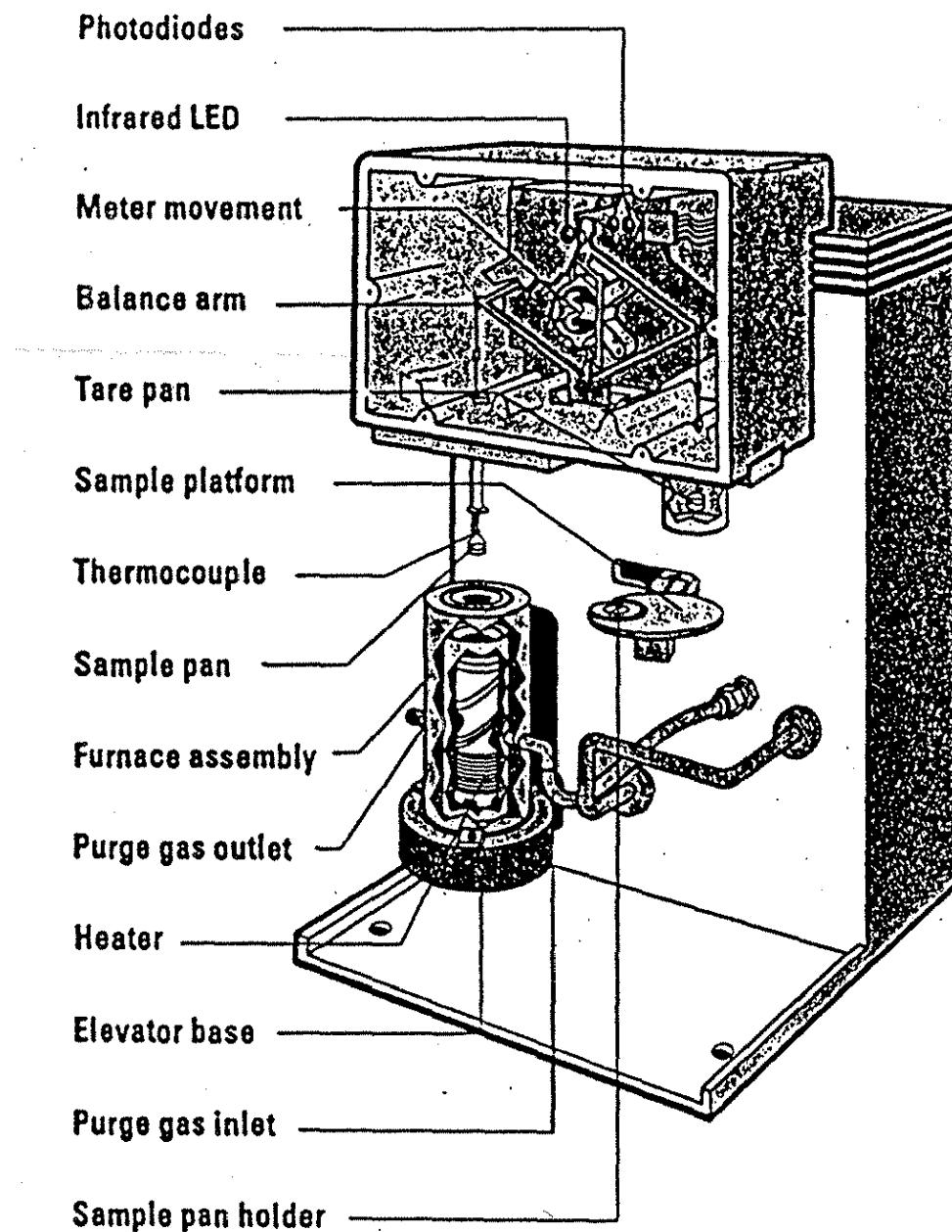
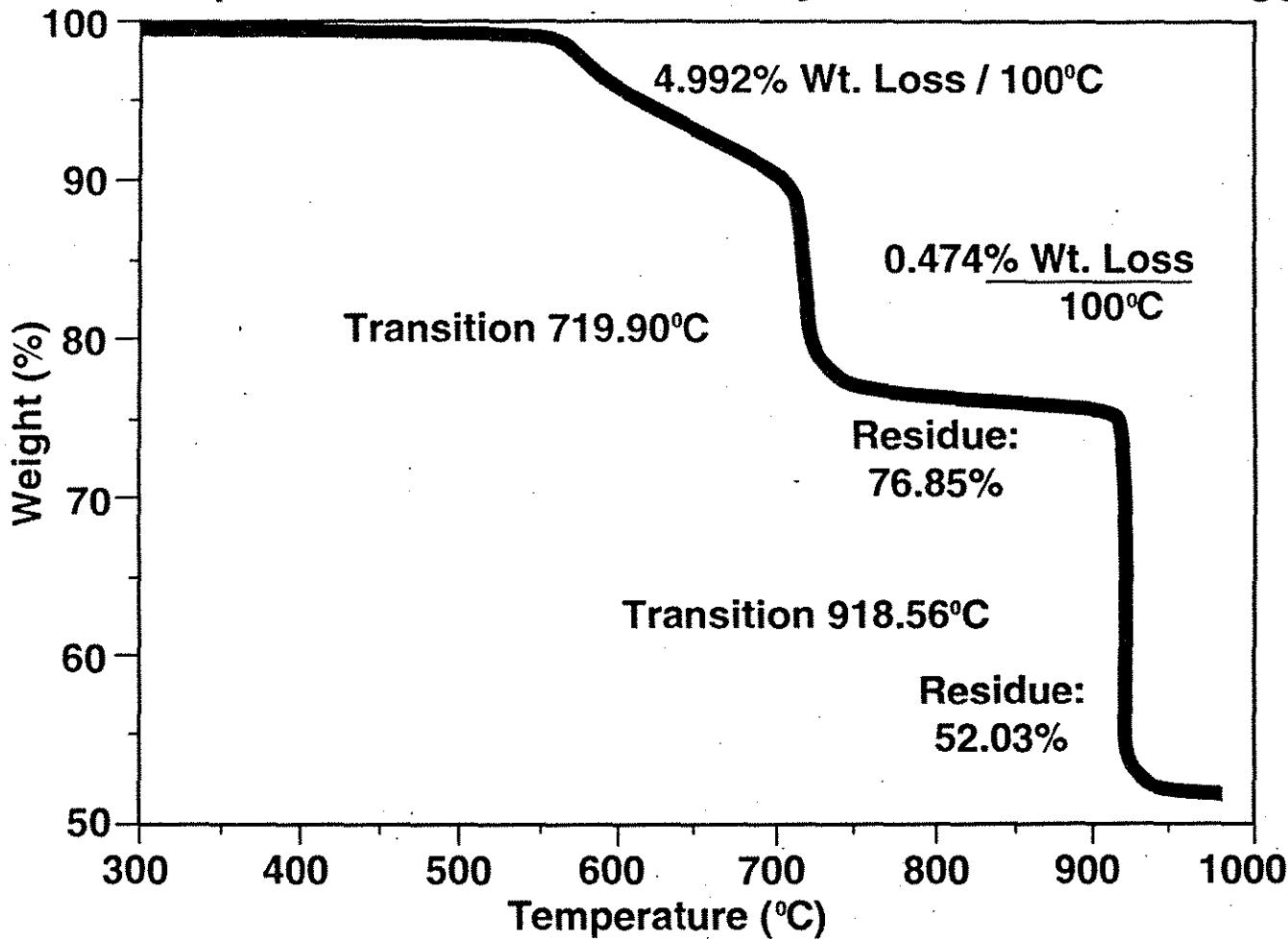


Figure 2 Computer Plot of a Good Quality Lisbon Dolomitic Aggregate



**Figure 3 Computer Plot of a Good Quality Moberly Limestone
(Calcite) Aggregate**

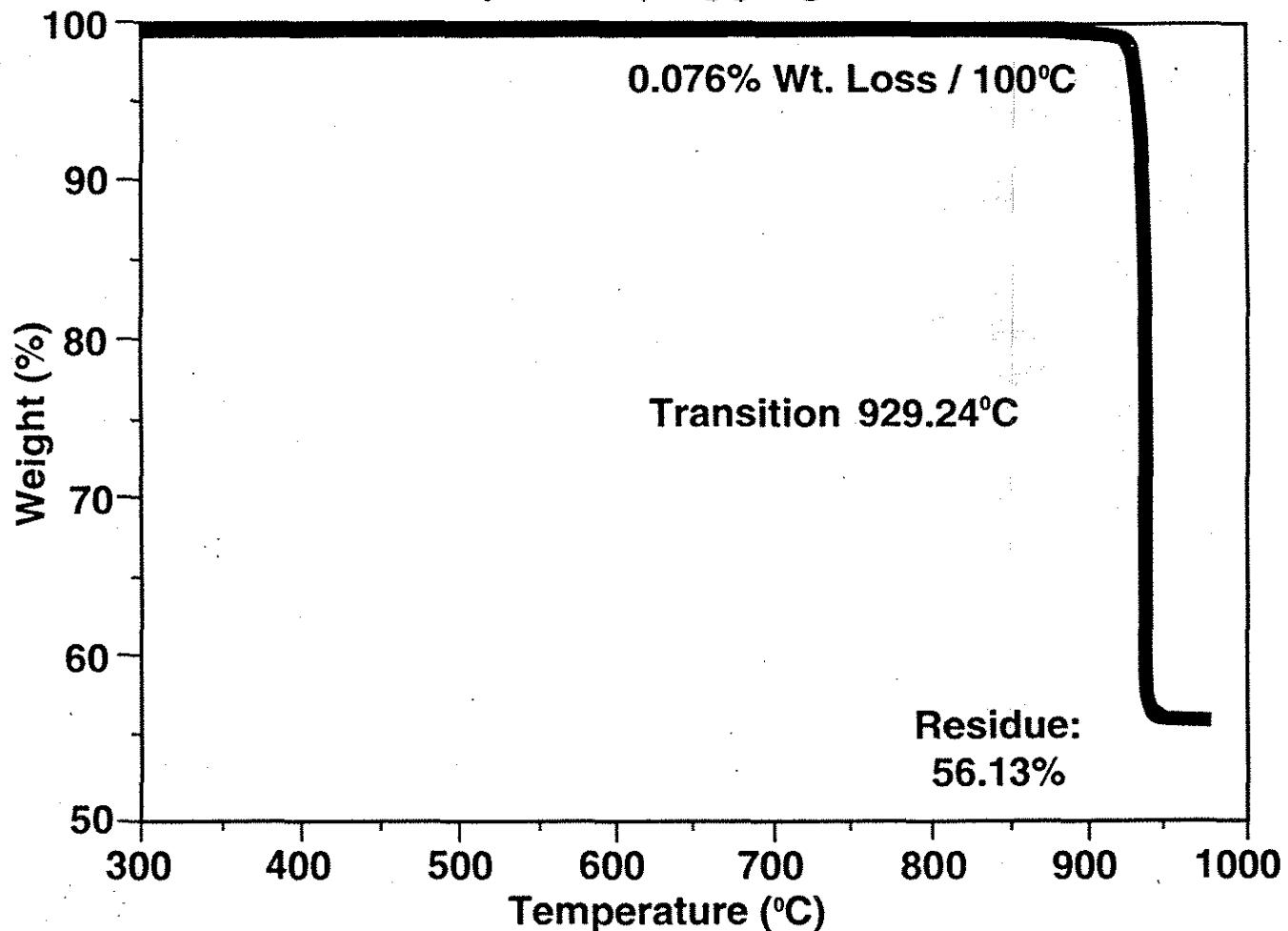


Figure 4 Computer Plot of a Poor Quality Crescent Limestone Aggregate

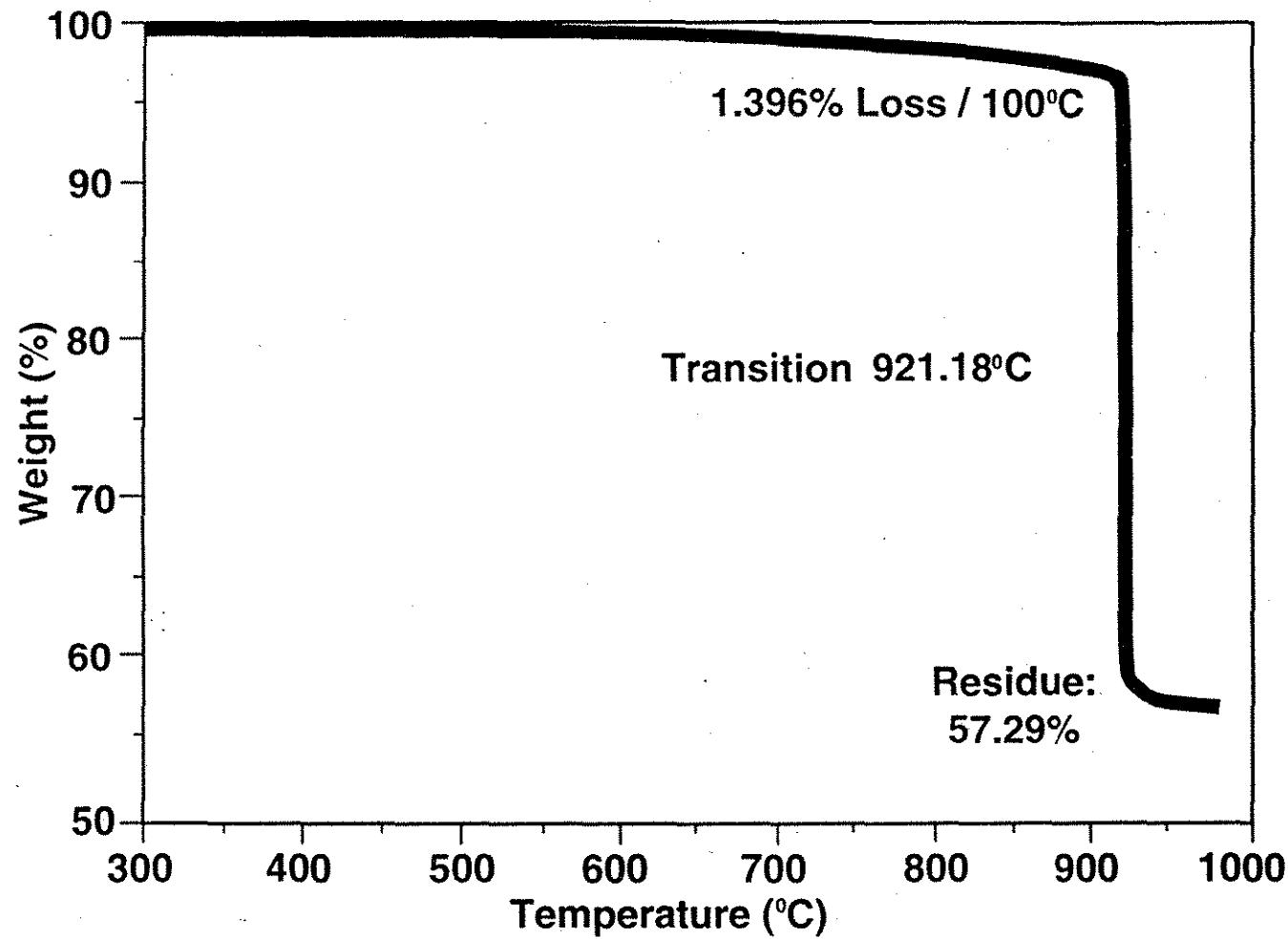
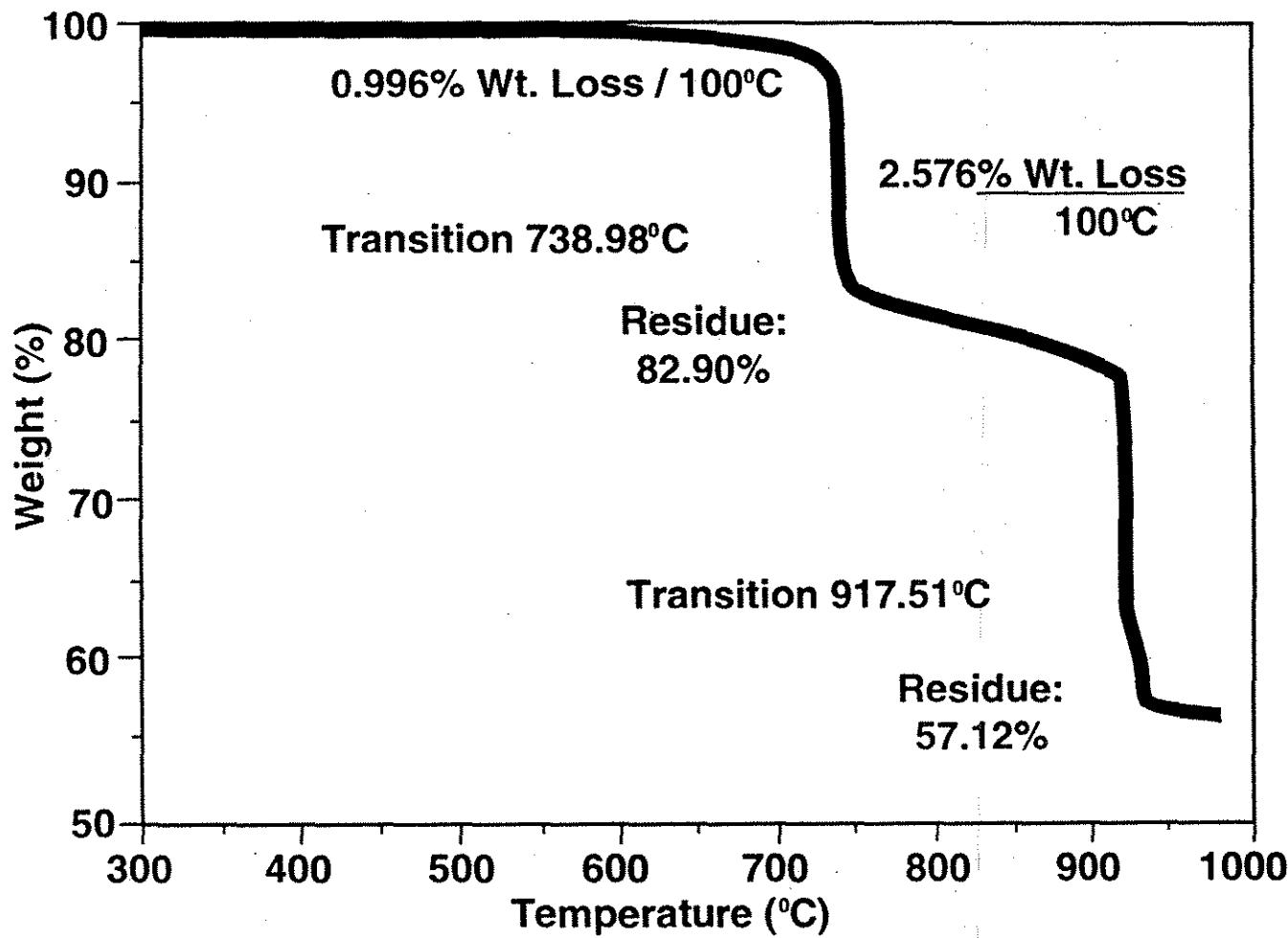
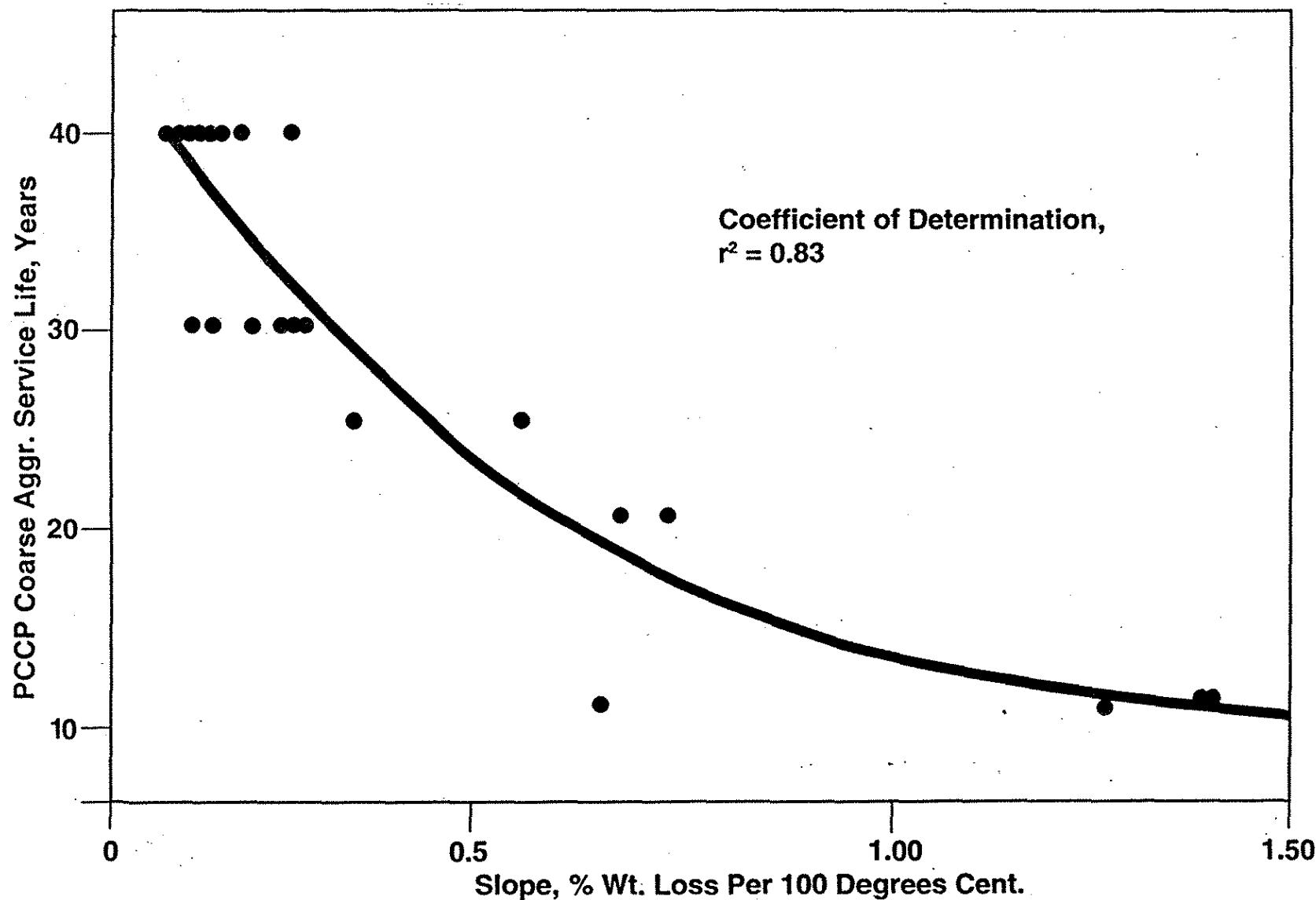


Figure 5 Computer Plot of a Poor Quality Legrand Dolomitic Aggregate



**Figure 6 Limestone Service Life vs. Slope
Prior to the Calcite Transition**



**Figure 7 Dolomite Service Life Vs. Slope
Prior to the Dolomite Transition**

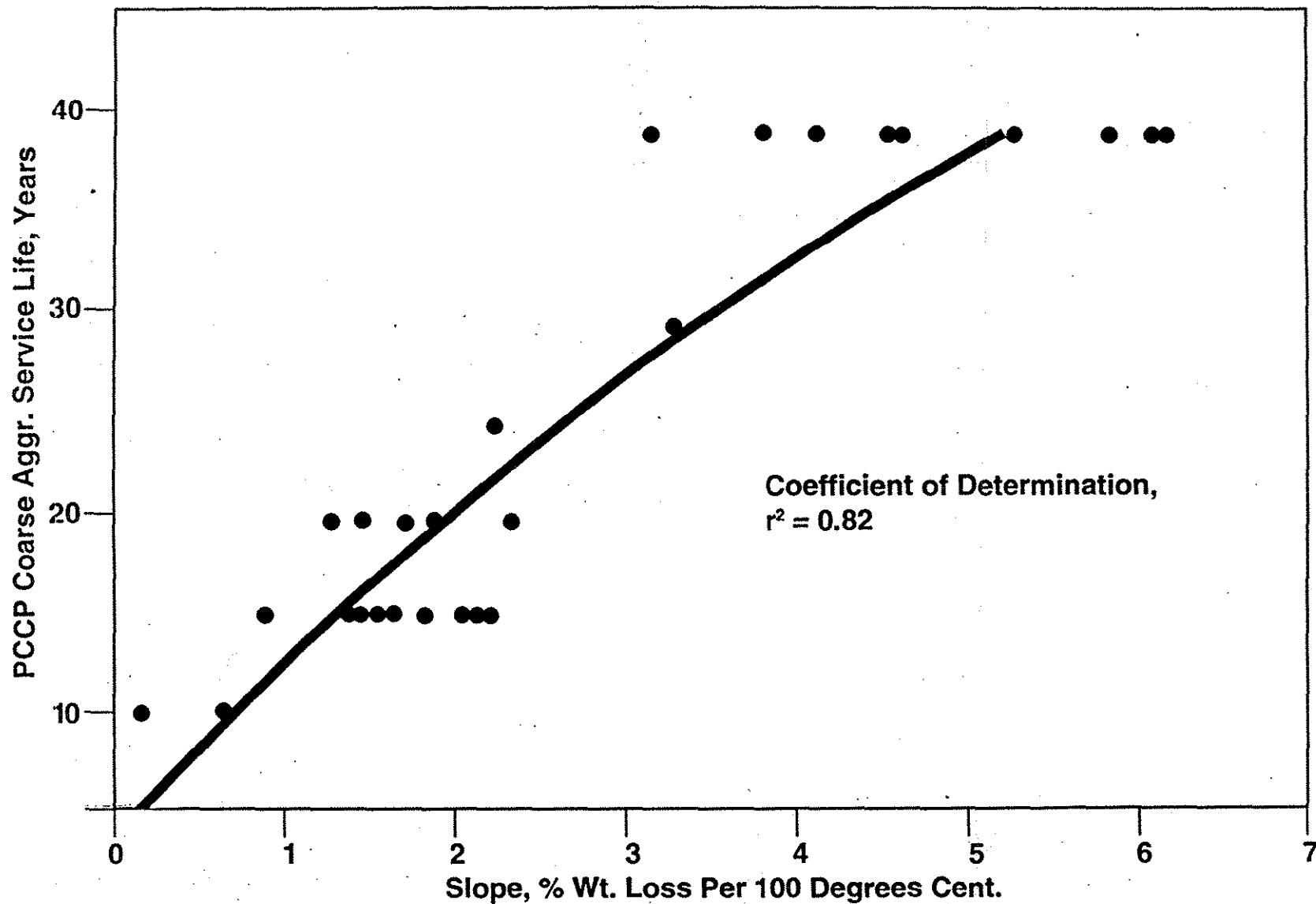


Figure 8 Computer Plot of a Landis Dolomite

