Final Report
for
Iowa Highway Research Board
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THE RELATIONSHIP OF

FERROAN DOLOMITE AGGREGATE
TO
RAPID CONCRETE DETERIORATION

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ABSTRACT

Some of Iowa's 13,200 miles of portland cement concrete (pcc) pavement have remained structurally sound for over 50 years while others have suffered premature deterioration. Research has shown that the type of coarse aggregate used in the pcc is the major cause of this premature deterioration. Some coarse aggregates for concrete exhibit a nonuniform performance history. They contribute to premature deterioration on heavily salted primary roadways while providing long maintenance-free life on unsalted secondary pavements. This inconsistency supports the premise that there are at least two mechanisms that contribute to the deterioration. Previous research has shown that one of these mechanisms is a bad pore system. The other is apparently a chemical reaction.

The objective of this research is to develop simple rapid test methods to predict the durability of carbonate aggregate in pcc pavement.

X-ray diffraction analyses of aggregate samples have been conducted on various beds from numerous quarries producing diffraction plots for more than 200 samples of dolomitic or dolomite aggregates. The crystalline structures of these dolomitic aggregates show maximum-intensity dolomite/ankerite peaks ranging from a d-spacing of 2.884 angstroms for good aggregates to a d-spacing of 2.914 angstroms for nondurable aggregates. If coarse aggregates with known bad pore systems are removed from this summary, the d-spacing values of the remaining aggregates correlate very well with expected service life. This may indicate that the iron substitution for magnesium in the dolomite crystal is associated with the instability of the ferroan dolomite aggregates in pcc pavement.

INTRODUCTION

Of the 13,200 miles of pcc pavement in Iowa, some have remained structurally sound for over 50 years while others have suffered premature deterioration. For years, Iowa has had a quality assurance program based upon frequent testing of the cement and aggregate used in pcc. The oldest of these quality control tests is a 16 cycle water/alcohol freeze and thaw test for the coarse aggregate. This test effectively excludes argillaceous (shaley) materials and has been a part of the standard specifications for 40 years.

In general, Iowa has had good performance from pcc pavement. One of the major problems in Iowa with some pcc pavements has been a rapid deterioration usually beginning at the joints and referred to as D-cracking (1). D-cracking (Figure 1) has severely reduced the life of these pcc pavements in Iowa, necessitating extensive full depth patching. Research in the 1960's identified the major factor in D-cracking as nondurable coarse aggregate. This research further revealed that the mechanism causing the cracking was freezing and thawing of water within the concrete. Iowa began using an ASTM C666, Method B (freezing in air, thawing in water) test in 1962 to further identify nondurable coarse aggregates. A 90-day moist-cure preparation prior to the ASTM C666, Method B testing produced results that correlated relatively well with the performance history of pcc pavements in Iowa. Research beginning in 1978 revealed a relationship between the freeze/thaw failure of carbonate aggregates and their pore systems (2, 3). The Iowa Pore Index was adopted and again was relatively reliable in identifying nondurable carbonate coarse aggregate.

Unfortunately, there were still notable exceptions in trying to categorize all aggregates which result in rapid deterioration as being caused by adverse pore systems (substantial pores in the 0.02 to 0.2 micron diameter range). Some nondurable aggregates with very good pore systems (pore sizes predominately larger than 0.2 micron diameter) contribute to rapid deterioration in a concrete system. This would indicate that there is another mechanism of failure. Dr. John Lemish of Iowa State University conducted substantial research on carbonate rocks as concrete aggregates. In June 1962 research, he identified rim reactions with particular nondurable aggregates and noted that the aggregate "had reacted chemically with its concrete environment" (4).

Dr. D. L. Biggs of Iowa State University in July 1967 referred to the ferroan nature of a D-cracking Iowa coarse aggregate (5). This research on compositional and mechanical properties of carbonate rocks was some of the earliest Iowa work with x-ray diffraction analysis of carbonates. The mineralogical characteristics of carbonate rocks from 13 different Iowa quarries were analyzed. Acetic acid was used to remove the calcite for a better diffraction evaluation of the crystallinity of the dolomite aggregates. Using Norelco x-ray diffraction equipment, a maximum-intensity dolomite peak was determined for a sample from Pints Quarry, bed 5B (Coralville) as having a d-spacing of 2.895 angstroms. Dr. Biggs noted that some dolomite aggregates "contain an excess of magnesium and probably iron in solution". While no further pursuit of that portion of the research was conducted, current research shows this finding to be relevant to the performance of dolomitic aggregates.

ENVIRONMENTAL EFFECT ON RATE OF PAVEMENT DETERIORATION

Office of Materials geologists evaluate pcc pavements to identify aggregate related failures. Continued observation of pcc pavement would indicate that the rate of deterioration is quite often dependent on environmental conditions. In some instances, this varied rate would relate to the pore system of the coarse aggregate contained in the concrete (1). Nondurable aggregates with an undesirable pore system seem to be affected by the availability of water. Between 15 and 20 years of age, some pcc pavements will exhibit moderate joint deterioration throughout the length of the project but accelerated joint deterioration is often found where perched water tables are intercepted through cut areas. It also appears that the permeability of the base does affect the rate of D-cracking deterioration with an increased rate of deterioration occurring in pcc placed on relatively impermeable bases.

Joint seal research was incorporated into a 1966 project (Montgomery Co. Rd. H-54 at M-63) to compare preformed neoprene seals with the standard (in 1966) low-grade bituminous sealing material. The low-grade bituminous sealing material failed very quickly and water was allowed into the bituminous sealed joints. The preformed neoprene seals functioned relatively well, and even though there was some loss of seal, they functioned much better than the bituminous sealed joints. The bituminous sealed joints exhibited rapid joint deterioration much earlier and the deterioration progressed more rapidly than did the joints sealed with preformed neoprene. To date, joint deterioration at the bituminous sealed joints is much more severe than at the neoprene sealed joints.

Substantial D-cracking deterioration (aggregate with a bad pore system) was identified on a section of pcc pavement at an interstate interchange (I-80 & US 71) that remained closed and received no deicing salt applications for a number of years. The section with no traffic exhibited approximately the same rate of D-cracking deterioration as that open to traffic. This D-cracking was caused by an aggregate with a bad pore system.

Primary roadway concrete has a higher cement content than the secondary roadway concrete. Laboratory research on the freeze/thaw durability of concrete has shown that with the use of the same materials the higher cement content produces a concrete with better durability than a concrete with a lower cement content.

There is one additional environmental difference between primary and secondary concrete pavements. There are some carbonate aggregates that have a very good pore system and yet exhibit rapid D-cracking on primary roadways. However, these same aggregates cause little or no D-cracking on secondary roadways which receive limited or no applications of deicing salts. A pavement containing a Pennsylvanian crushed limestone from the Lemley-Exline quarry was incorporated into a pavement on primary highway US 34 in southern Iowa in 1962. Today, it exhibits severe D-cracking. A secondary road containing the same Lemley-Exline aggregate constructed in 1964 exhibits only staining and no visible D-cracking deterioration. A D-cracking susceptible gravel containing 70% carbonate was used in primary highway US 30 in central Iowa in 1965. Severe D-cracking of this pcc pavement has made it necessary to resurface a section with asphalt concrete. A secondary road containing the same gravel aggregate was constructed in 1966 and exhibits no indication of D-cracking today. In general, the secondary pavements do not receive

applications of deicing salt for wintertime maintenance. The primary roadways receive substantial amounts of sodium chloride deicing salts. Many secondary highway departments use selective deicing salt applications at stop signs, on bridges and at railroad crossing. This, in conjunction with salt brine being tracked from primary roadways, results in substantial salting on the secondary roadways at primary road intersections. The serious joint deterioration prevalent on Story County road E-29 at the intersection with US 65 decreases rapidly to no deterioration a short distance from US 65. This inconsistency in the performance of carbonate aggregates in pcc pavement was one of the factors that led to the current research on the instability of ferroan dolomites.

Another situation where nonuniformity in the rate of joint deterioration may occur is where a pcc roadway constructed with a nondurable aggregate passes under a bridge. There are cases where joint deterioration is extensive except where the road passes under the bridge (US 30 under I-35 and US 34 beneath city street overpasses in Burlington). The joints that are essentially beneath the overpass bridges will exhibit little or no joint deterioration. This variation could result from fewer freeze/thaw cycles or from being protected from the effects of the sun (temperature and/or evaporation) or rainfall. Despite much speculation, the mechanism that causes this variation has not yet been identified.

OBJECTIVE

The objective of this research is to develop simple, rapid test methods to predict the durability of carbonate aggregate in pcc pavement. The test methods include analyses of the pore systems and chemical compositions of aggregate sources.

DETERIORATION MECHANISMS

Past research and performance histories have provided conclusive evidence that the major property of one type of nondurable aggregate is an undesirable pore system. The mercury porosimeter, the Iowa Pore Index test and a recently developed ice porosimeter will very adequately identify aggregates containing undesirable pore systems. Other deterioration mechanisms would apparently be chemically related. A sodium chloride salt treatment (6) prior to ASTM C666, Method B, freeze/thaw testing causes rapid loss of durability with some aggregates while exhibiting very little effect on other aggregates. Research regarding the NaCl effects on aggregate seems to point toward an alteration of the crystalline structure. This alteration weakens the aggregate, rendering the concrete susceptible to deterioration by freeze/thaw cycles.

Another relatively simple test was used in an effort to determine the chemical reaction that was taking place with the carbonate aggregates. A slice of rock was sawed from a piece of Garrison ferroan dolomite. One side of this slice was polished. The polished slice was broken into three pieces which were boiled in three different liquids for two hours. One liquid was distilled water, the second was a calcium chloride solution and the third was a sodium chloride solution. Scanning electron microscope photomicrographs were taken of each sample (Figures 2, 3 & 4). The piece boiled in distilled water showed no appreciable change. The section boiled in calcium chloride solution exhibited only minor etching and discoloration. The section that was boiled in sodium chloride showed substantial etching and discoloration (an apparent disruption of the crystal surface). X-ray diffraction analysis of material boiled in the calcium chloride solution showed changes that were not visually evident (Figure 5). Prior to the boiling of the material in the

calcium chloride solution, there was no maximum-intensity calcite peak. There was a strong dolomite maximum-intensity peak at 2.904 angstroms. After boiling in calcium chloride there was still a strong maximum-intensity dolomite peak at 2.904 angstroms but there was also a maximum-intensity calcite peak at a d-spacing of 3.039 angstroms. This would indicate that some of the iron and/or magnesium was replaced by calcium, but this apparently did not change the appearance of the polished surface to any extensive degree. The diffraction plots of the Garrison dolomite after boiling in water, sodium chloride or magnesium chloride showed no significant change (Figures 6, 7 & 8). Boiling of a Maryville non-ferroan dolomite in calcium chloride also produced a calcite peak (Figure 9). While this study does not prove the aggregate is weakened by deicing salts, it does suggest that deicing salts produce chemical and crystallographic changes within the aggregate that may lead to deterioration.

X-RAY DIFFRACTION STUDY OF THE DOLOMITE/ANKERITE PEAK
A Siemens D-500 computer-controlled X-ray diffractometer was used for the analysis of all crushed carbonates. The diffractometer is controlled with a PDP-11-03 microcomputer. The sample, pulverized to pass a No. 300 mesh sieve, is rotated during analysis while scanning through a d-spacing range from 15 to 70.

The mineral and elemental compositions have been determined for many carbonates using X-ray fluorescence and diffraction. The compositions of a selected few of the carbonates used in this report are given in Table 1.

An X-ray diffraction analysis of the maximum-intensity dolomite peak d-spacing has been conducted on various beds from numerous quarries within Iowa (Table 2). Only dolomites and dolomite-limestone mixtures are shown in Table 1 which includes 57 of the over 200 samples that have been analyzed. X-ray diffraction plots have been produced for a number of these dolomitic or dolomite aggregates. The maximum-intensity dolomite/ankerite peak of these aggregates shifts from a d-spacing of 2.884 angstroms for a dolomite to 2.914 angstroms for a ferroan dolomite containing iron in the crystalline structure. Iron can become a part of the dolomite crystalline structure (7) yielding a ferroan dolomite or ankerite ($Ca (Mg, Fe) (CO_3)_2$).

In Iowa, non-ferroan dolomites have generally given excellent durability in portland cement concrete. Many of these non-ferroan dolomites are of the Silurian age and have performance histories of 30 years or more. Aggregates produced at the Garrison Quarry from the Coralville Member of the Devonian Cedar Valley Formation are nonargillaceous, ferroan dolomites. Garrison aggregate does not contain floating dolomite "rhombs" in a fine-grained argillaceous matrix. There is a predominance of pore diameters larger than 0.2 microns. It would be generally accepted that the Garrison aggregate contains a very good pore system and when tested according to ASTM C666, Method B freezing and thawing test, it will yield durability factors above 90. Based upon all of these characteristics, a very good durability in pcc pavement would have been expected from this Garrison dolomite prior to this research. Unfortunately, when this coarse aggregate is used in portland cement concrete, joint deterioration can be identified in 12-13 years, and it requires patching before 20 years.

The Garrison dolomite exhibits one of the greatest shifts in the maximum- intensity dolomite/ankerite peak. A comparison of the Garrison dolomite and the Maryville dolomite is given in Figure 10. The maximum-intensity dolomite peaks do not coincide for the Garrison dolomite and the Maryville dolomite. The d-spacing for the Garrison ferroan dolomite is 2.904 angstroms while the Maryville, a non-ferroan dolomite, has a maximum-intensity dolomite d-spacing of 2.884. The maximum-intensity dolomite peak of the dolomite standard is 2.886. The maximum-intensity peak for ankerite standard, a ferroan dolomite, has a d-spacing of 2.899 angstroms. The Garrison dolomite crystal structure contains sufficient iron to cause the maximum-intensity peak for this aggregate to shift to a d-spacing of 2.904 angstroms. This shift apparently relates directly to the amount of iron present in the crystalline dolomite. If the aggregates that have bad pore systems are excluded, (they are noted with an asterisk in Table 2) the maximum-intensity dolomite/ankerite d-spacing values correlate very well with performance histories. This correlation is shown in Figure 11 and exhibits a coefficient of correlation (r) of -0.91 and an r^2 of 0.82. The Maryville dolomite for example is a very good aggregate with an expected service life of 40 years while the Garrison dolomite has a very poor service record with a predicted service life of 15 years. The shift of the maximum-intensity dolomite/ankerite peak does correlate very well with expected service life and, therefore, may be an excellent indicator of the potential durability of that aggregate in pcc pavement.

An X-ray diffraction analysis was conducted on a sample made by combining 50% of the Garrison ferroan dolomite with 50% of an Aurora (Silurian age very similar to Maryville) non-ferroan dolomite (Figure 12). It did not result in

one maximum-intensity dolomite peak but yielded two peaks of approximately one half the intensity of the individual samples.

Most researchers attribute the deterioration mechanism of the Kingston, Canada, dolomitic-limestone aggregate to alkali-carbonate reactivity. The dolomite in the Kingston aggregate produces a maximum-intensity dolomite/ankerite peak at a d-spacing of 2.908 angstroms.

It is interesting to note that the Pints Coralville maximum-intensity dolomite/ankerite peak occurs at a d-spacing of 2.895 angstroms which exactly duplicates the d-spacing determined by Dr. Biggs in the 1967 research.

The Stanzel aggregate is predominantly a limestone but does have a maximum-intensity dolomite/ankerite peak occurring at 2.907 angstroms. The Stanzel aggregate has a good pore system but has a very poor service record. Based on the correlation of performance histories to dolomite peak shifts, carbonates (dolomites and dolomitic limestones) having greater maximum-intensity dolomite/ankerite d-spacings would be expected to exhibit poorer performance in pcc pavement than those dolomites with smaller d-spacings. This x-ray diffraction study would indicate that iron in carbonate aggregates could result in instability causing premature deterioration when used in pcc pavements.

CONCLUSIONS

This research on the instability of some carbonate aggregates in pcc pavement supports the following conclusions:

1. Iron substituting for magnesium in the dolomite crystalline structure of coarse aggregate correlates with performance history in Iowa pcc pavement, especially when NaCl deicing salts are used.

2. For carbonate aggregates with good pore systems, a low maximum-intensity x-ray diffraction dolomite d-spacing would indicate a durable aggregate while a high maximum-intensity dolomite/ankerite x-ray diffraction d-spacing would indicate a nondurable aggregate.

3. The determination of the d-spacing of the maximum-intensity dolomite/ankerite peak for dolomite or dolomitic limestone aggregates is a good screening test to predict dolomite-aggregate durability in pcc pavement.

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TABLE TITLES

1. XRF Chemical Analyses of Sources (By Percent)

2. d-Spacing of the Maximum-Intensity Dolomite/Ankerite Peak

TABLE 1   XRF CHEMICAL ANALYSES OF SOURCES (BY PERCENT)

ELEMENTS & THEORETICAL COMPOUNDS	AURORA DOLOMITE	GARRISON DOLOMITE	KINGSTON DOLOMITIC LIMESTONE	MARYVILLE DOLOMITE	PINTS DOLOMITE	STANZEL LIME-STONE
Sr		0.02	0.04			0.05
SrO		0.03	0.05			0.06
Mg	11.80	10.71	5.38	12.68	11.61	0.69
MgO	19.56	17.76	8.92	21.03	19.25	1.13
$MgCO_3$	40.91	37.15	18.65	43.99	40.26	2.36
Ca	22.25	23.61	27.14	21.89	23.62	38.10
CaO	31.12	33.03	37.98	30.62	33.05	53.30
$CaCO_3$	55.55	58.96	67.79	54.66	58.99	95.14
Fe	0.18	1.07	0.82	0.23	0.40	0.60
Fe_2O_3	0.25	1.52	1.17	0.32	0.57	0.86
S	0.02	0.65	0.14	0.02	0.10	0.06
SO_3	0.05	1.63	0.34	0.05	0.24	0.16
Ti		0.01	0.06	0.01	0.01	0.01
TiO_2		0.02	0.11	0.02	0.02	0.02
Mn		0.06	0.02			0.03
MnO		0.07	0.03			0.04
Si	1.79	0.71	4.21	0.34	0.22	0.86
SiO_2	3.84	1.53	9.00	0.74	0.47	1.84
K	0.12	0.09	0.67	0.04	0.02	0.04
K_2O	0.14	0.11	0.80	0.05	0.03	0.05
P		0.01	0.01	0.01	0.01	0.02
P_2O_5		0.02	0.02	0.02	0.02	0.04
Al	0.26	0.16	1.31	0.13	0.06	0.11
Al_2O_3	0.50	0.30	2.48	0.24	0.11	0.22

TABLE 2   d SPACING OF THE MAXIMUM INTENSITY DOLOMITE/ANKERITE PEAK

SOURCE NAME	PEAK	YRS	SOURCE NAME	PEAK	YRS	SOURCE NAME	PEAK	YRS
MARYVILLE...........	2.884	40	LAMONT.............	2.891	40	MONTOUR EAGLE CITY.	2.902	20
AURORA..............	2.887	40	RHEIM..............	2.891	35	NELSON CORALVILLE..	2.902	15
SHAFFTON...........	2.887	30	SKYLINE............	2.891	30	LOGAN..............	2.902	15*
DALEY..............	2.887	30	FRANTA.............	2.891	30	VIRGINIA 01........	2.902	15
HAWARDEN GRAVEL.....	2.887	20*	DOTZLER............	2.892	25	GLORY RAPID........	2.903	10
GILMORE CITY........	2.888	30	WLOO SOUTH SOLON...	2.892	20*	CONKLIN RAPID......	2.903	10
BOWSER..............	2.888	30	KANKAKEE ILLINOIS..	2.893	40	VIRGINIA 12........	2.903	15
LISBON..............	2.889	40	PINTS SOLON........	2.893	20*	JABENS CORALVILLE..	2.904	15
TRIPOLI.............	2.889	40	FERGUSON...........	2.894	25	SULLIVAN SLOUGH....	2.904	10*
BROWN..............	2.889	30	PINTS CORALVILLE..	2.895	20*	THAYER.............	2.904	05*
KENDALLVILLE........	2.889	30	NASHUA.............	2.896	15*	GARRISON CORALVILLE	2.904	15
JOHNSON.............	2.890	30	AMES MINE..........	2.897	25	STENNETT...........	2.905	05
ELKADER.............	2.890	15*	PORTLAND WEST......	2.897	25	STANZEL............	2.907	05
WLOO SOUTH C'VILLE..	2.890	10*	PINTS RAPID........	2.899	15	JEFFERSON..........	2.907	05*
SEDGEWICK...........	2.890	40	BIRDS HILL GRAVEL..	2.900	15*	EARLY CHAPEL.......	2.908	15*
ANAMOSA.............	2.890	35	LEGRAND............	2.900	20	KINGSTON...........	2.908	10
WYOMING.............	2.890	40	SMITH..............	2.901	15	PENN-DIXIE.........	2.911	05*
ULLIN ILLINOIS......	2.890	10*	MALCOM MINE........	2.901	15	PLANO..............	2.912	15*
AHLES	2.891	30	GRAND MEADOW MINN..	2.901	20*	LEMLEY.............	2.914	15*

YRS   VISUAL DETERIORATION-FREE LIFE

*   AGGREGATE PORE SYSTEM CONTRIBUTES TO FREEZE-THAW DETERIORATION

FIGURE CAPTIONS

1. A close-up of D-cracking at the intersection of the transverse and longi-
    tudinal joints.
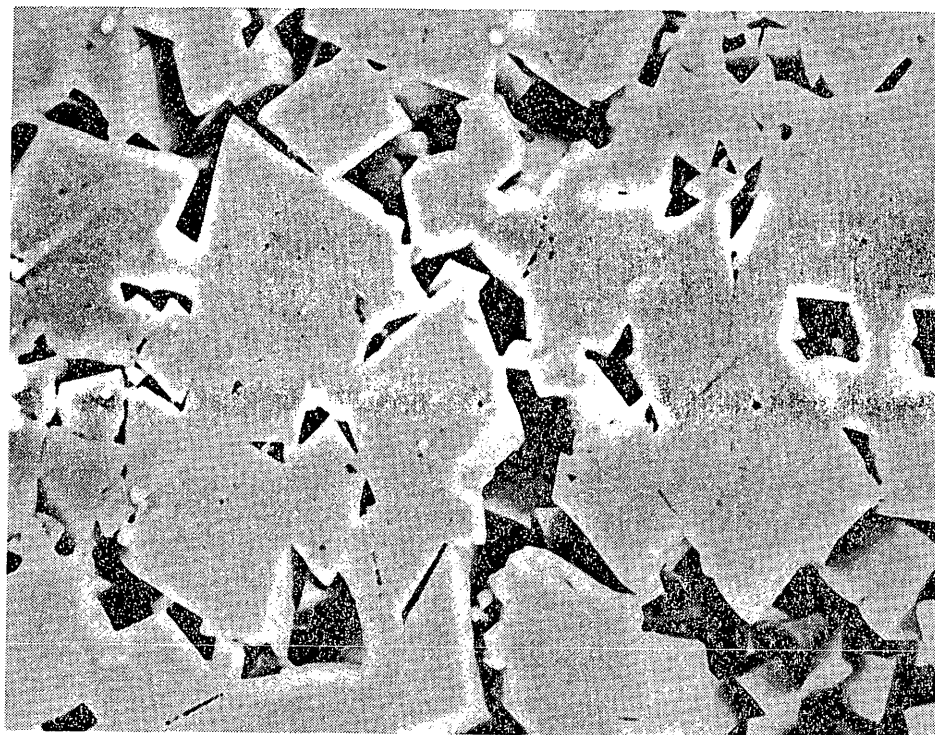
2. SEM photomigrograph of a polished Garrison dolomite surface after boiling
    in distilled water.

3. SEM photomigrograph of a polished Garrison dolomite surface after boiling
    in a calcium chloride solution.

4. SEM photomigrograph of a polished Garrison dolomite surface after boiling
    in a sodium chloride solution.

5. Garrison X-ray diffractogram - no treatment and boiling in a calcium
    chloride solution.

6. Garrison X-ray diffractogram - no treatment and boiling in distilled wa-
    ter.

7. Garrison X-ray diffractogram - no treatment and boiling in a sodium
    chloride solution.

8. Garrison X-ray diffractogram - no treatment and boiling in a magnesium
    chloride solution.

9. Maryville X-ray diffractogram - no treatment and boiling in a calcium
    chloride solution.

10. X-ray diffraction plots of Maryville and Garrison dolomites.

11. Graph of d-spacing of maximum-intensity dolomite peak vs years to visible
    deterioration of the concrete.

12. X-ray diffraction plots of Garrison and Aurora dolomite and a 50%
    Garrison - 50% Aurora mixture.

Figure 1   A close-up of D-cracking at the intersection of the
transverse and longitudinal joints

Figure 2   SEM photomigrograph of a polished Garrison dolomite
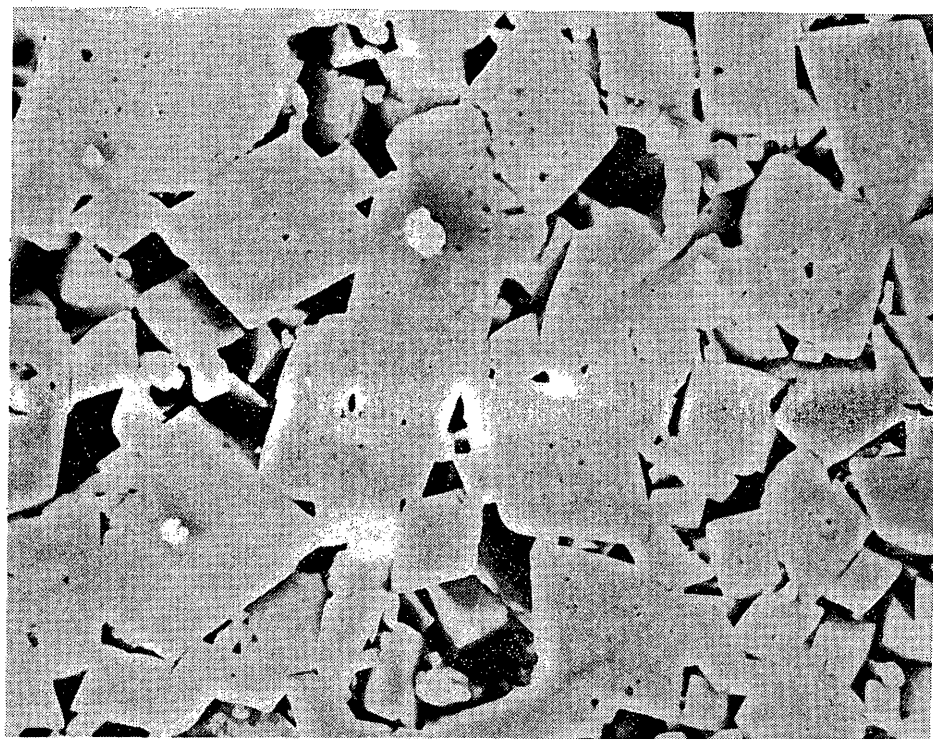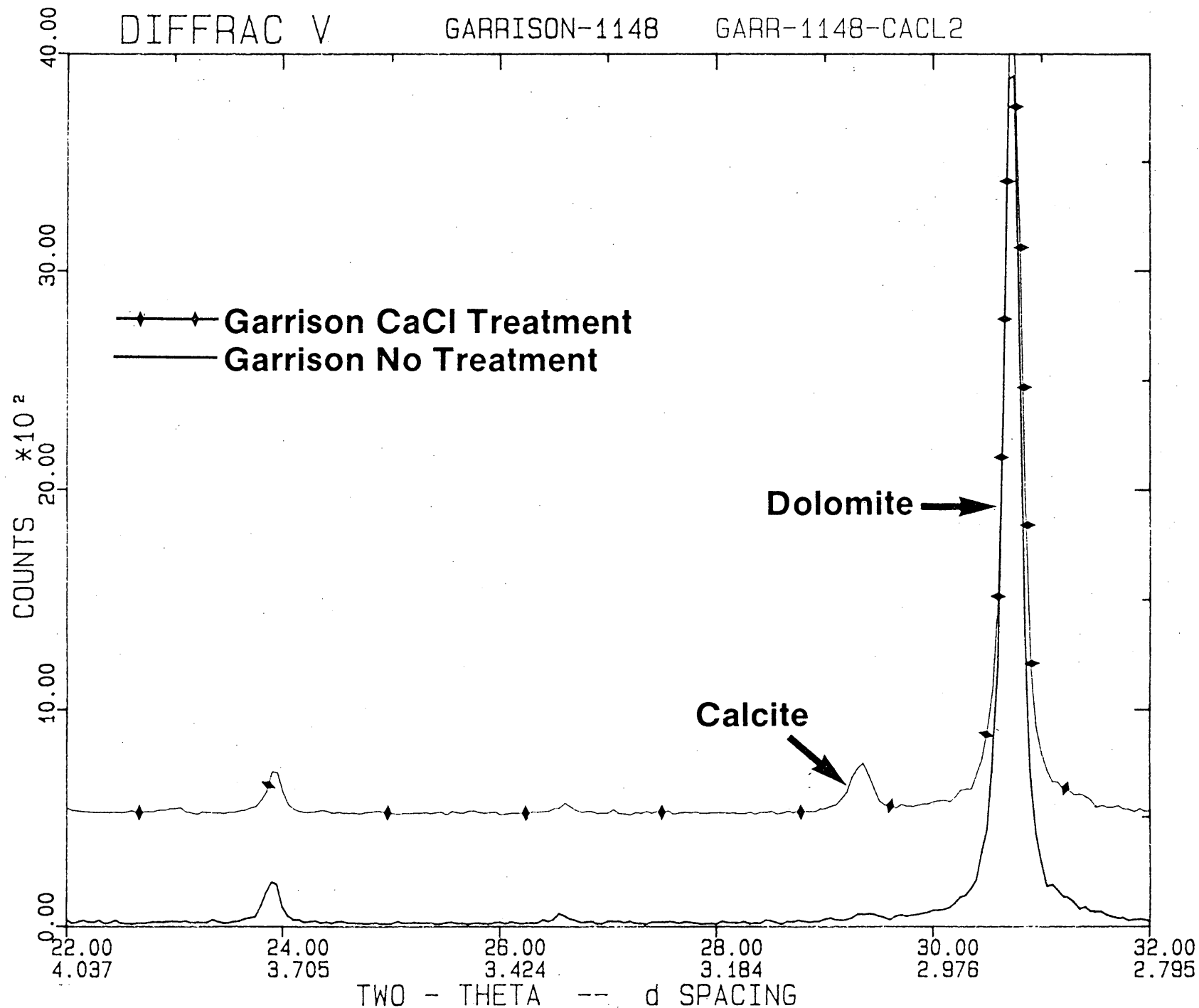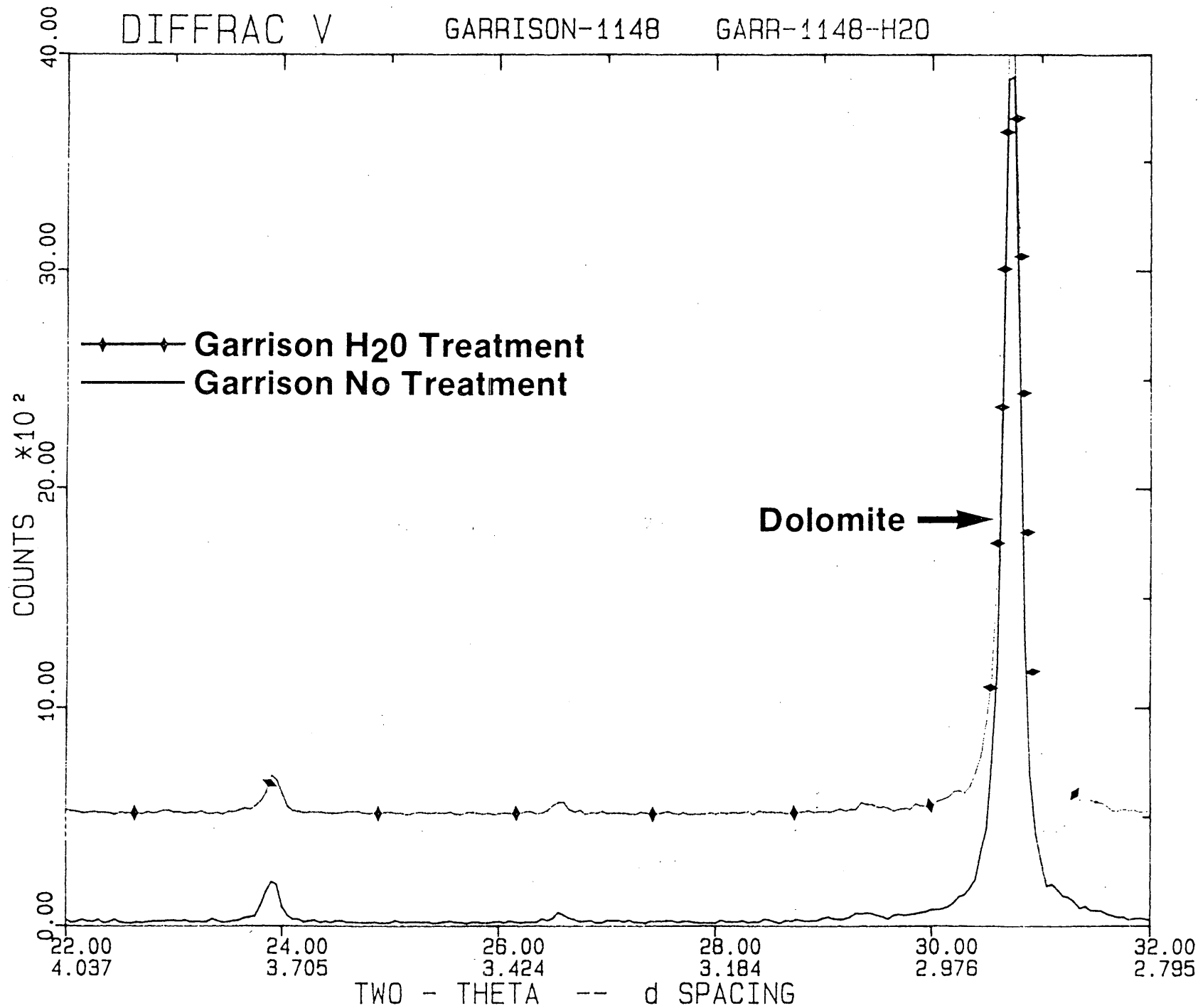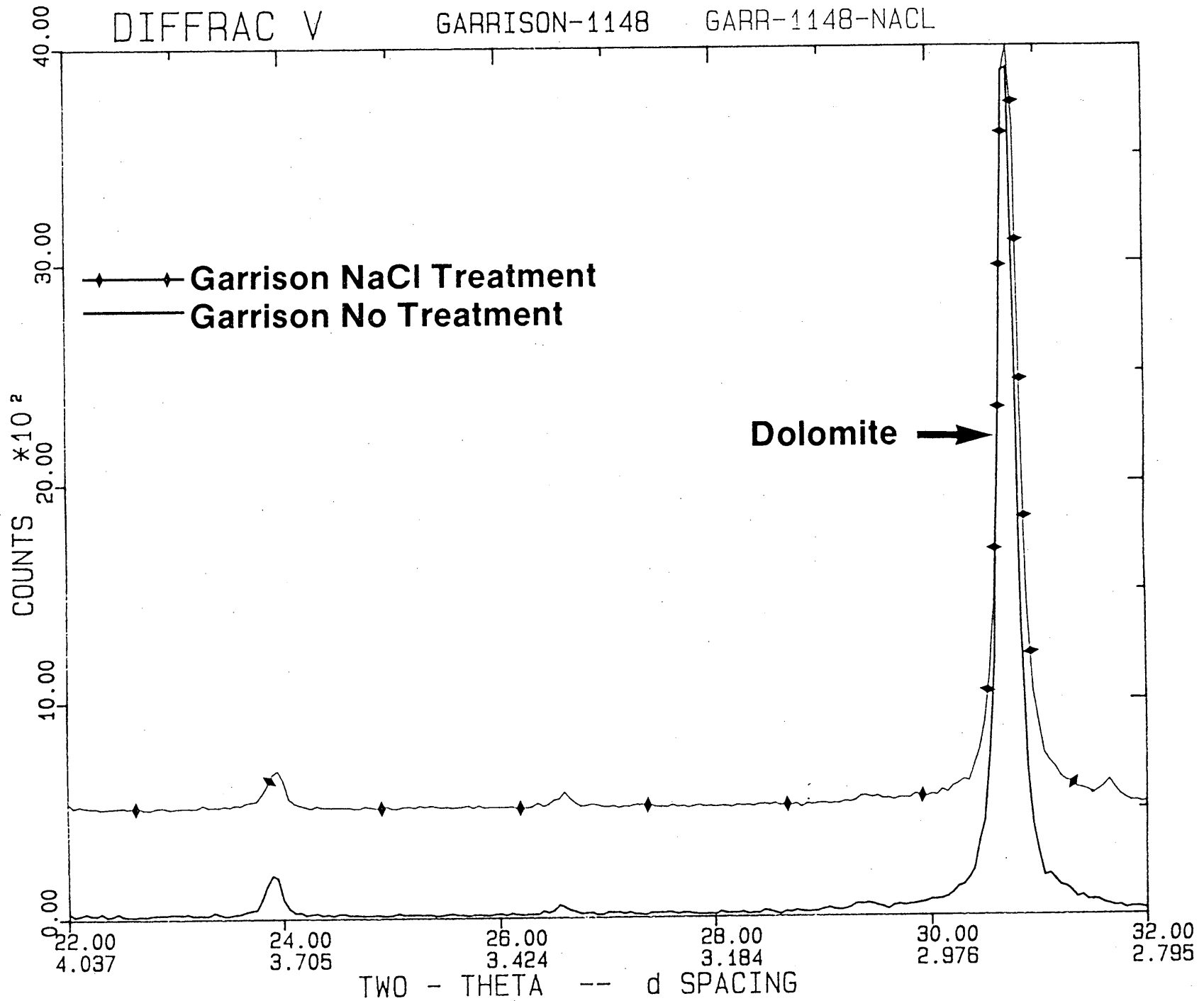surface after boiling in distilled water

Figure 3   SEM photomigrograph of a polished Garrison dolomite
           surface after boiling in a calcium chloride solution

Figure 4   SEM photomigrograph of a polished Garrison dolomite
           surface after boiling in a sodium chloride solution

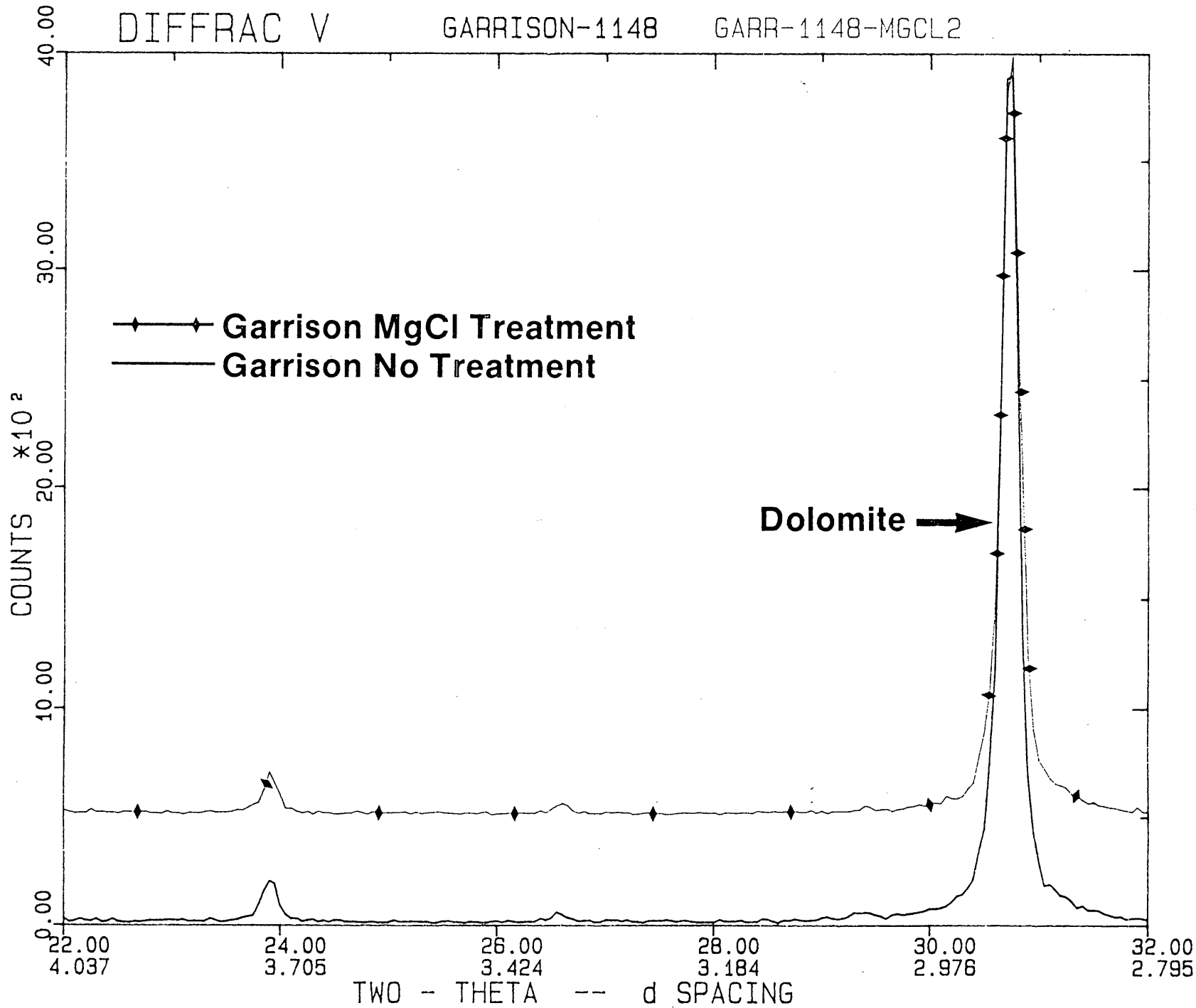FIGURE 5   GARRISON X-RAY DIFFRACTOGRAM. - NO TREATMENT AND BOILING IN CALCIUM CHLORIDE SOLUTION

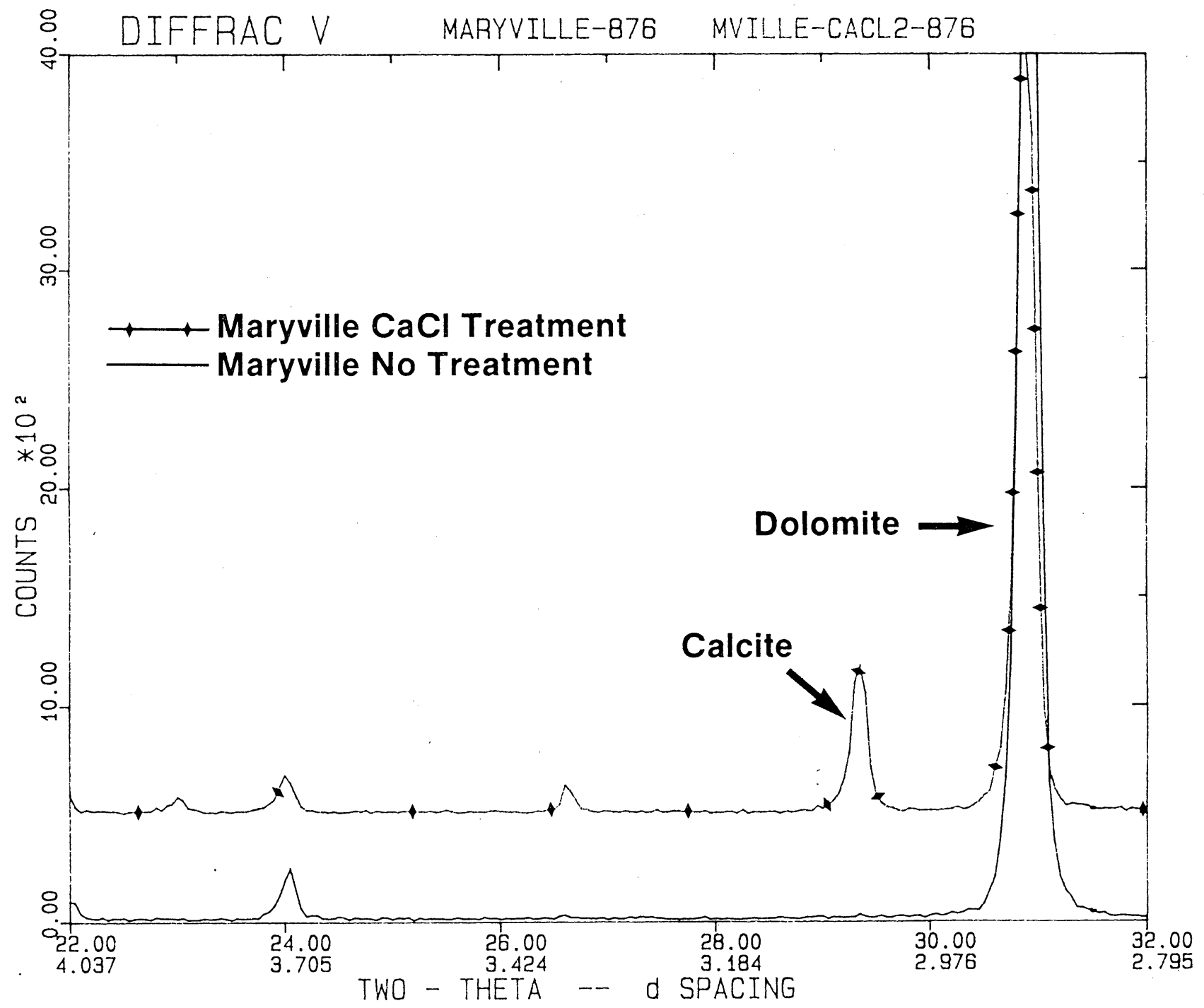FIGURE 6 GARRISON X-RAY DIFFRACTOGRAM – NO TREATMENT AND BOILING IN DISTILLED WATER

DIFFRAC V   GARRISON-1148   GARR-1148-NACL

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Legend:
- →——→ **Garrison NaCl Treatment**
- —— **Garrison No Treatment**

Dolomite ——→

COUNTS *10²

TWO – THETA -- d SPACING

| TWO-THETA | 22.00 | 24.00 | 26.00 | 28.00 | 30.00 | 32.00 |
| d SPACING | 4.037 | 3.705 | 3.424 | 3.184 | 2.976 | 2.795 |

FIGURE 8   GARRISON X-RAY DIFFRACTOGRAM – NO TREATMENT AND BOILING IN A MAGNESIUM CHLORIDE SOLUTION

FIGURE 9   MARYVILLE DOLOMITE X-RAY DIFFRACTOGRAM - NO TREATMENT AND BOILING IN A CALCIUM CHLORIDE SOLUTION

DIFFRAC V   MARYVILLE-876   MVILLE-CACL2-876

Maryville CaCl Treatment
Maryville No Treatment

Dolomite →

Calcite

TWO - THETA -- d SPACING

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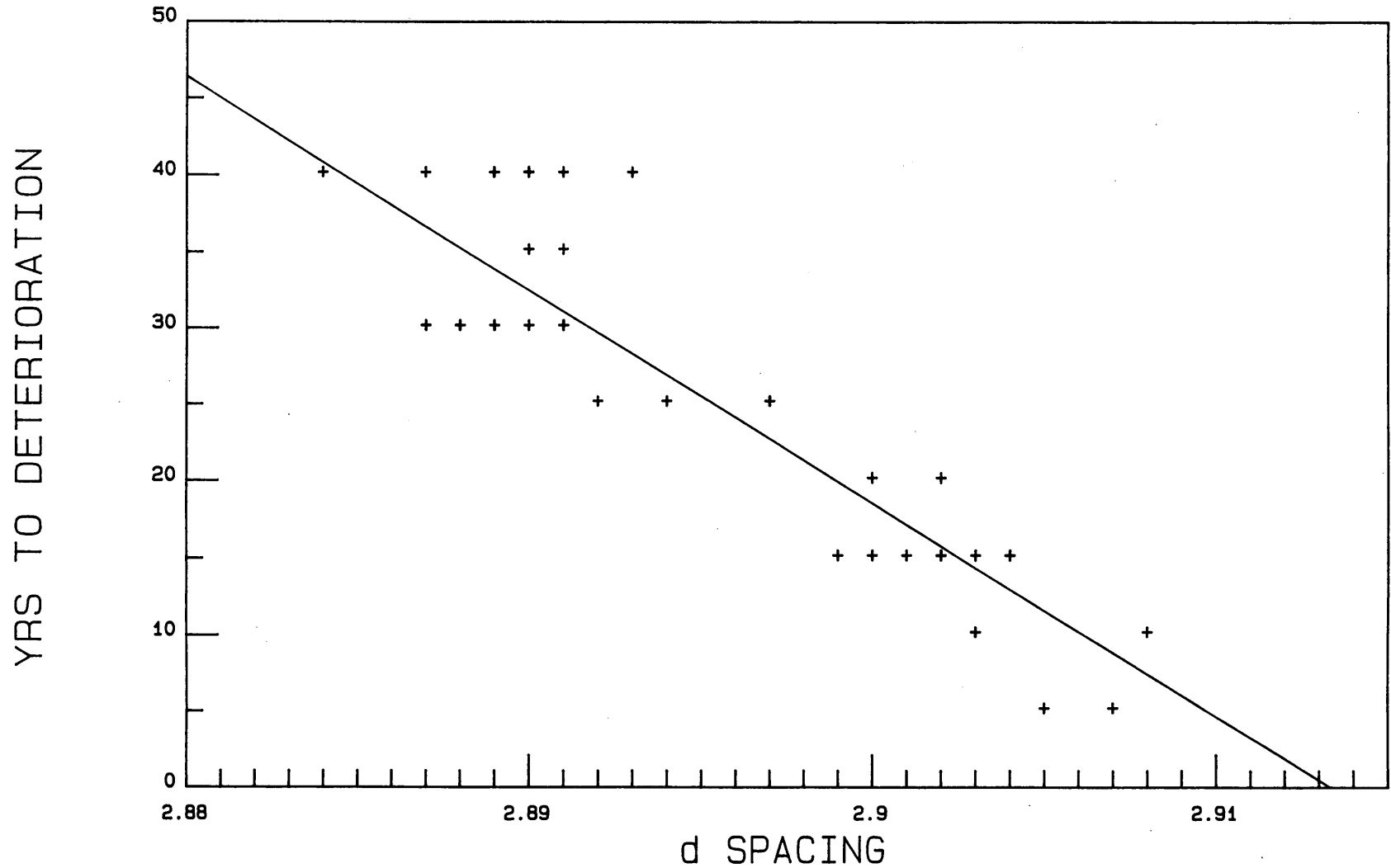FIGURE 10   X-RAY DIFFRACTION PLOTS OF MARYVILLE AND GARRISON DOLOMITES

d SPACING OF MAXIMUM INTENSITY DOLOMITE PEAK
VS
YEARS TO VISIBLE DETERIORATION OF THE CONCRETE

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FIGURE 12   X-RAY DIFFRACTION PLOTS OF GARRISON AND AURORA DOLOMITE AND A 50% GARRISON – 50% AURORA MIXTURE