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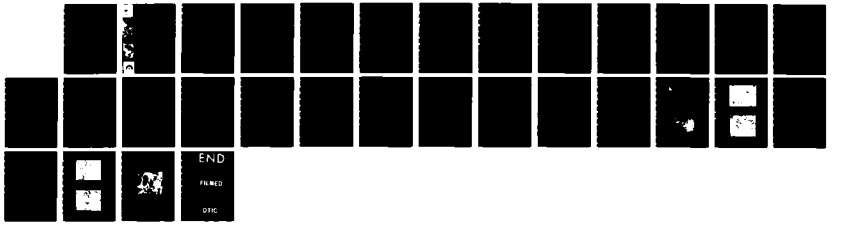

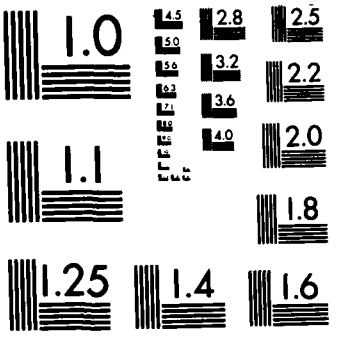

US Army Corps of Engineers

STRUCTURES LABORATORY

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CHARACTERIZATION OF SEVERAL PLASTERS AND ONE RETARDER FOR REPOSITORY SEALING MIXTURES

by

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Structures Laboratory

DEPARTMENT OF THE ARMY
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Plaster
Plaster of paris
Plaster retarder
Repository sealing

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Samples of plasters (i.e., calcium sulfate hemihydrate, $CaSO_4 \cdot 1/2H_2O$) from six sources and one plaster retarder from a different source were obtained and characterized by a combination of chemical and physical testing and by petrographic examination. The petrographic examination included X-ray diffraction (XRD), optical microscopy, differential thermal analysis (DTA), and scanning electron microscopy (SEM) procedures.

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20. ABSTRACT (Continued).

The intent of this work was twofold. One purpose was to determine if plaster per se could be used as an ingredient for cementitious mixtures intended for repository sealing applications. Previous experience had been with use of a proprietary admixture that contained plaster. The second purpose was to determine one or more methods of distinguishing between the alpha and beta forms of calcium sulfate hemihydrate.

It was concluded that commercially available plaster of paris rather than a proprietary admixture could be used as an ingredient for the proposed cementitious mixtures. The other finding was that, as expected, the beta form of $CaSO_4 \cdot 1/2H_2O$ is finer and thus requires more water for mixing so the coarser alpha form is preferable for the described use. While the two forms can be distinguished by physical tests, by DTA, by SEM, and by optical microscopy, the latter procedure is the simplest and the quickest. All that is required is examination of a plaster as an immersion mount with a polarizing microscope to determine particle size and shape.

Four of the six plasters were the alpha form while two were the beta form. The retarder contained both forms of plaster along with organic material (thought to be keratin).

Keywords: [illegible] water; plaster of paris; tables (data).

Preface

This report was prepared for the US Department of Energy (DOE) under continuing contract DE-AI97-81ET 46633. It was a milestone for FY 84 which was prepared as a draft report that was submitted in May 1984.

Mr. Steve Webster of the DOE in Columbus, Ohio, was Project Manager when this report was prepared for publication.

This report was prepared in the Concrete Technology Division (CTD) of the Structures Laboratory (SL), USAE Waterways Experiment Station (WES), by Messrs. A. D. Buck, J. P. Burkes, and Ronald E. Reinhold (retired), under the direction of Mr. J. M. Scanlon, Chief, CTD, and Mr. Bryant Mather, Chief, SL. Mr. Buck was Project Leader in the CTD.

Commanders and Directors of WES during the preparation of this report were COL Tilford C. Creel, CE, and COL Robert C. Lee, CE; Technical Director was Mr. Fred R. Brown. During the publication of this report, COL Allen F. Grum, USA, was Director of WES; Dr. Robert W. Whalin was Technical Director.

Contents

Conversion Factors, Non-SI to SI (Metric) Units of Measurement

Non-SI units of measurement used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
inches	25.4	millimetres
pounds (mass)	0.4535924	kilograms
angstroms	0.1	nanometres

CHARACTERIZATION OF SEVERAL PLASTERS AND ONE RETARDER FOR REPOSITORY SEALING MIXTURES

Background

1. Earlier work (approximately 1981) in the repository sealing program on expansive hydraulic cementitious systems used proprietary commercial forms of calcium sulfate hemihydrate (plaster of paris), usually with additives to induce expansion. This project was undertaken to determine whether nonproprietary forms of plaster of Paris (calcium sulfate hemihydrate, $CaSO_4 \cdot 1/2H_2O$), hereafter called "plaster," could be used. In addition, the existence of two forms of plaster and means to discriminate between them were to be examined.

2. Accordingly, seven plaster samples from six sources (one set of two samples) representing four producers were obtained. A plaster retarder was obtained from a different source. These materials were characterized chemically, physically, and petrographically. Since two of the plasters were from one source, only one of these received the full testing.

Samples

3. The following samples were examined:

Structures Laboratory (SL) Serial No.	Other Information
AD-626	Two 100-lb bags of expansive additive received 13 February 1980
	Same as above but no SL serial number. Identified as D-53, two bags received 1 May 1979
AD-654	One 100-lb bag of plaster received October 1980
AD-656	50 lb molding plaster (beta form, no retarder) received 25 March 1981
AD-657	Three bags "Gypsum" received 21 February 1981
AD-660	49 lb plaster (alpha form, no retarder) received 3 April 1981
AD-661	50 lb plaster (alpha form, no retarder) received 3 April 1981

Structures Laboratory (SL) Serial No.	Other Information
AD-661(2)	Two 50-lb cardboard containers received 11 August 1981
AD-661(3)	Three 100-lb bags received 21 August 1981
AD-664	Eight 24-oz bags of plaster retarder received May 1981

Test procedure

4. Tests of the plasters were made in accordance with applicable portions of ASTM: C 471-76, "Methods for Chemical Analysis of Gypsum and Gypsum Products," and ASTM: C 472-79, "Methods for Physical Testing of Gypsum Plasters and Gypsum Concrete." The results were compared with ASTM: C 59-76, "Specification for Gypsum Casting and Molding Plaster." Other tests were used as needed and are indicated in the test data tabulations. Testing of the retarder (AD-664) was limited to petrographic procedures.

5. A portion of each as-received sample except D-53 was examined by X-ray diffraction (XRD) as a backpacked powder. The backpacking was done to minimize preferred orientation. D-53 material had been examined by XRD as part of an earlier examination.(1) Portions of the water-insoluble residues of six plasters (D-53, AD-654, AD-656, AD-657, AD-660, and AD-661) were sprinkled on double-coated tape and examined by XRD; these residues had been prepared by dehydrating the six plasters to anhydrite by heating and then dissolving the anhydrite in distilled water.

6. Since the insoluble residues appeared to contain clay minerals, a water slurry of each was placed on a 1- by 1-in. (25- by 25-mm) glass slide and allowed to dry; these dried films were then examined by XRD. Five of these slides (D-53, AD-656, AD-657, AD-660, and AD-661) were treated with glycerol for 24 hr and then examined again by XRD. Four of these slides (D-53, AD-656, AD-660, and AD-661) were heated for 1 hr at 350° C and then examined by XRD in an atmosphere of static nitrogen. The heated film of AD-661 on a glass slide was also examined by X-ray emission spectroscopy (XRE).

7. XRD samples were examined with an X-ray diffractometer using nickel-filtered copper radiation.

8. Samples of three as-received plasters (AD-626, AD-656, AD-661) and the retarder (AD-664) were prepared as pressed pellets and examined by XRE to determine identifiable elements. These emission patterns and the one of the AD-661 slide were made using chromium radiation and a pentaerythritol (PET) analyzing crystal.

9. Immersion oil with a refractive index of 1.544 was used to make immersion mounts of as-received material from each sample and of the insoluble residues from plaster samples D-53, AD-654, AD-656, AD-657, AD-660, and AD-661. Each mount was examined with a polarizing microscope.

Photomicrographs of plasters AD-656 and AD-661 were made in plane polarized light and with crossed polarizers to illustrate the differences in the appearance of alpha and beta type plaster grains.

10. A portion of each as-received sample was examined with a differential thermal analyzer (DTA) to a temperature of about 1050° C in a nitrogen atmosphere using a heating rate of 10° C per minute. Other portions of samples AD-656, AD-657, AD-660, and AD-661 were given pretreatment over magnesium nitrate solution before DTA examination as before. To assist in the recognition of thermal peaks in the DTA patterns that might be due to the presence of an organic additive, a known organic additive (D-65)[1]* was added to a larger amount of plaster AD-660 and a DTA pattern was made. Since it was known that AD-660 did not contain an organic additive, any new peaks in the DTA pattern of the mixture would be due to the added material.

11. Plasters AD-656 and AD-661 and retarder AD-664 were examined with a scanning electron microscope (SEM). The plasters (AD-656, AD-661) were prepared by stirring about 0.25 g of sample in 50 ml of ethel alcohol to keep the particles suspended. Some of these suspensions were placed on aluminum sample stubs and allowed to dry. The retarder (AD-664) was prepared by placing double-coated tape on a SEM stub and sprinkling a small amount of the powder on the sticky top surface. These three sample-mounted stubs were then examined with an SEM without coating the samples since they were hydrates that might be sensitive to normal SEM preparation procedure. This avoided the vacuum drying usually done during coating of samples. In addition, the examination was done at a low accelerating voltage (6 kv) to minimize heat-induced changes and charging in the samples. After this examination, the samples were coated in a vacuum evaporater with a layer of carbon approximately 50 Å thick and a layer approximately 150 Å thick of gold-pallidum alloy to make them electrically conductive. The coated samples were then examined with a SEM at a normal accelerating voltage (21 kv). The SEM micrographs of the uncoated and coated samples were compared.

Results

12. The chemical and physical data for the original seven plasters were shown on Waterways Experiment Station (WES) Form No. 1114 as Test Report WES-305-81; these are shown as Tables 1 and 2 for chemical and physical data, respectively. Similar data for the additional samples of plaster AD-661 (2 and 3) are shown in Tables 3 and 4. The test results indicated that all of the plasters met the requirements of ASTM: C 59-76 for gypsum casting and molding plaster. Calculation of the plaster as gypsum indicated a range from 95.32 (AD-654) to 98.28 (AD-660, 661). As indicated in Tables 3 and 4, the two later samples of AD-661 were combined and tested as one sample; calculated gypsum content was 97.33 percent.

13. The results of XRD examinations are shown in Table 5. All plasters contained calcium sulfate hemihydrate (hemihydrate) as the expected major constituent. While routine XRD examination of the whole plasters did not suggest the presence of any other calcium sulfate phase, a special examination

* Used as AD-627 in work described in WES Miscellaneous Paper SL-81-2, "PSU/WES Interlaboratory Comparative Methodology Study of An Experimental Cementitious Repository Seal Material," Report 2, Final Results, Mar 1982, and WES Miscellaneous Paper SL-83-18 (SAND 83-7097), "Modification of Bell Canyon Test Grout," Sep 1983.

of additional partial XRD patterns did suggest that a little anhydrite was probably present in plasters AD-626, AD-654, AD-657, and AD-660; this was based on the presence of a weak peak at 2.84 to 2.86 Å. All of the plasters except AD-656 did show detectable anhydrite by XRD in the water-insoluble residues, but all or some of it may have formed during drying of the samples. Anhydrite was detectable in the as-received retarder (AD-664), as was dolomite. A total of 11 noncalcium sulfate phases were identified in the insoluble residues of the plasters. While not shown in Table 5, AD-626 was examined in its as-received condition. The plaster retarder (AD-664) contained hemihydrate and anhydrite in addition to a noncrystalline organic phase which would not be detectable by XRD. It was the only sample to contain any brucite (magnesium hydroxide); it was detectable in the as-received retarder.

14. As expected, comparison of XRD patterns of known alpha forms of plaster (AD-660, 661) with the known beta form (AD-656) plaster showed that they cannot be discriminated by XRD; this indicates they are the same in crystal structure.

15. The elements that were detected in plasters AD-626, AD-656, and AD-661 and in plaster retarder AD-664 by X-ray emission spectroscopy (XRE) are shown in Table 6. The presence of strontium in these samples was verification that the strontium sulfate mineral celestite was present in some samples (Table 5).

16. Examination of immersion mounts showed the following:

a. There was a distinct difference in particle morphology of known alpha (AD-660, 661) and beta (AD-656) plasters. Figures 1 through 4 illustrate this difference for plasters AD-656 and AD-661 in plane polarized light and in crossed polarizers.

b. Particles of the beta form of hemihydrate plaster AD-656 did not show crystal faces; they tended to be anhedral with a fibrous appearance in either type of lighting (Figures 1, 3). It was difficult to determine the extinction angle of the fibers; however, at high magnifications most of the fibers showed parallel extinction and were length-slow. Some did not show complete extinction.

c. Particles of the alpha form of hemihydrate plasters AD-660 and AD-661 did show crystal faces (Figures 2, 4). The particles were elongated with parallel extinction; individual particles ranged from euhedral to subhedral and were length-slow with fairly high birefringence.

d. As-received plaster retarder AD-664 was a mixture of several components. Both the elongated grains and the anhedral (fibrous) grains were present. The fibrous grains made up the larger percent of the two types. There was quite a bit of dolomite in this sample (Table 5). A large part of the sample consisted of yellowish, flaky particles that were isotropic. These isotropic particles were probably keratin.*

* This tentative identification by a chemist is based on odor after heating at less than 100° C.

e. As-received plasters D-53, AD-654, and AD-657 contained traces of small, brownish spheres or broken spheres. These are known to be a slight organic contamination in D-53;[1] it is likely they represent similar contamination of AD-654 and AD-657. AD-626 which is the same as D-53 also shows this contamination.

f. The water-insoluble material in AD-657 consisted of many whole or broken spheres that were opaque. Shards from these spheres were isotropic and had a brownish color. Some of these shards showed signs of devitrification. The above material was also found in the insoluble material from D-53 and AD-654. It was much more abundant in AD-657. Neither this material nor the organic type material was present in plasters AD-656, AD-660, or AD-661.

17. The examination of immersion mounts of each plaster showed they could be separated into two groups by appearance as follows:

Group 1	Group 2
AD-626 (D-53)	AD-656
AD-654	AD-657
AD-660	
AD-661	

Since this also separates the known alpha forms (AD-660, 661) from the known beta form (AD-656), the presumption is that this is a viable method of separating these forms and the method identifies AD-626 (D-53) and AD-654 as alpha forms and AD-657 as beta form.

18. DTA examination of the seven plasters and one retarder in an inert nitrogen atmosphere without any pretreatment of samples showed the following:

a. Samples D-53 and AD-626, AD-654, AD-660, and AD-661 all have a strong endotherm that ranges from 153° to 158° C; there is an immediate sharp exotherm following this peak that ranges from 182° to 190° C. These features for AD-661 are shown in Figure 5.

b. Samples D-53 and AD-626, and AD-654 had a weak endotherm at approximately 700° C. These three samples also showed a weak broad exotherm with the peak temperatures occurring at approximately 800° to 850° C.

c. Samples AD-656 and AD-657 both had a strong endothermic peak at 140° and 132° C, respectively. They also had a weaker unresolved endotherm that occurred at approximately 163° C. Neither sample had the exotherm at the approximate temperature range of 182° to 190° C that was present in the other five plasters. These features for AD-656 are also shown in Figure 5.

d. The retarder sample, AD-664, had two distinct endotherms at 136° and 152° C; in addition, there was a stronger endotherm at about 1013° C. The DTA curve of the mixture made by combining plaster AD-660 with a smaller amount of organic additive AD-627[1] also produced a strong endotherm at about 1013° C. Since this peak was probably due to organic keratin in

AD-664 and was due to a different organic (AD-627)[1] in the synthetic mixture, it is probably common to both materials but not diagnostic for either one.

19. Plaster samples AD-656, AD-660, and AD-661 representing the known beta and alpha forms were also examined by DTA in an inert nitrogen atmosphere after pretreatment in saturated magnesium nitrate solution as recommended by Mackenzie[2] to give a relative humidity of about 55 percent; the samples were left in this environment for about 14 days before examination by DTA. Pretreatment of plasters AD-656, AD-657, AD-660, and AD-661 over saturated magnesium nitrate solution in vacuum for 4 days before DTA examination as before was done. The effects of pretreatment of different forms of plaster was to minimize the differences found without any pretreatment. Pretreatment caused the presence of a weak endotherm at about 50° C, resulted in slight shifts of the major endotherm, and caused the development of a slight exotherm at about 185° C in the beta form plasters where none had been present before. In general, it appears that the presence of a distinct exotherm at about 185° C is characteristic of alpha form plaster and may be used to differentiate between the two forms. The difference in temperature of the major endotherm between the two forms of plaster appears less useful because it is sensitive to sample preparation techniques.

20. While the DTA method does provide a method for separating the alpha and beta forms and did separate them into the same grouping as light microscopy, the differences by DTA were more subtle than was realized at first. Examples of the features that have been discussed are shown in Figures 5 and 6.

21. Two plasters representing the alpha form (AD-661) and the beta form (AD-656) and the retarder (AD-664) were selected for SEM examination. Since micrographs of uncoated and coated samples showed essentially identical features, those of coated samples were selected since they were of better quality (Figures 7, 8, 9).

22. Figure 7 is a SEM micrograph of beta form plaster AD-656 at a magnification of 500 X. The particle size ranges from about 64 μm down to about 1 μm. The absence of crystal faces and the varied shape of the particles can be seen. Figure 8 is a SEM micrograph of alpha form plaster AD-661 at a magnification of 200 X. The particle size in the micrograph ranges from about 55 μm down to 1 μm. The presence of crystal faces on particles is apparent. Figure 9 is a micrograph of plaster retarder AD-664. It appears to be mostly beta form plaster; the organic retarder (keratin) is not readily evident. Particle size appears to range from about 44 μm down to 1 μm.

Discussion

23. It is generally known that beta hemihydrate plasters are finer and require more water and that separations by physical data and SEM are possible.[3-] Industrial plasters are usually alpha hemihydrate while building plasters

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* Personal communication with Dr. R. A. Kuntze of Ontario Research Foundation and R. Lang of U. S. Gypsum.

are beta and finishing plasters are alpha.* Plots (not shown) of the physical data from Table 2 show a separation of the plasters based on water demand for pastes or mortars that is identical with the one shown earlier for particle morphology based on microscope examination of immersion mounts.

24. Comparison of XRD patterns of all of the plasters did not provide a basis for separating them into alpha and beta forms of plaster (i.e., calcium sulfate hemihydrate). This was as expected.

25. Examination of all of the plasters as immersion mounts with a light microscope and by DTA showed that both methods provided a basis for separation into alpha and beta forms; the first by particle shape and the second by differences in temperature of the major endotherm and by the presence or absence or intensity of an exotherm near the major endotherm. The differences in DTA curves (Figure 5, 6) are similar to those shown in Figure 13.1 on page 397 of Mackenzie.(2) Later examination of two beta plasters (AD-656, 657) and two alpha plasters (AD-660, 661) by DTA after a sample pretreatment period showed that the difference may be as slight as the greater intensity of the alpha exotherm at about 185° C.

26. Limited examination of an alpha form plaster (AD-661) and a beta form plaster (AD-656) by SEM agreed with observations by light microscope that there was a recognizable difference in particle morphology.

27. The broad exotherm that appeared at 800° to 850° C on DTA curves of plaster samples D-53 and AD-626 and AD-654 was not identified. It was thought at first that it was due to the organic AD-627(1) that was identified in three amounts in these samples. However, the following observations rule out that explanation.

a. The peak did not show on the curve made from the synthetic mixture made by blending an organic additive and plaster AD-660.

b. The peak did not show on the curve made from plaster AD-657 which probably has a trace of the organic additive in it.

28. The brownish opaque spherical particles and the brownish shards found in the insoluble residues of D-53, AD-654, and AD-657 were probably glass with some of the shards in AD-657 having devitrified. If so, they are probably the smectite clay found in this sample (Table 5).

29. All of the sulfates were not usually completely removed by the storage in deionized water that was intended to dissolve calcium sulfate. There was always anhydrite in each insoluble material (Table 5) except for plaster AD-656.

Conclusions

30. Each of the six different plasters examined was composed mainly of plaster (calcium sulfate hemihydrate) with minor amounts of other crystalline phases.

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* Personal communication with Dr. R. A. Kuntze of Ontario Research Foundation and R. Lang of U. S. Gypsum.

31. In general, the present findings agree with those in the relevant literature[3,4,5,6] and personal communications* that say calcium sulphate hemihydrate exists in two forms (alpha, beta) which are morphologically different but structurally alike. The morphological differences are basically due to wet or dry methods of preparation.[3,4,5]

32. Specific findings were:

a. Determination of whether a plaster is of the alpha or the beta form can be made on the basis of physical properties, especially the higher water demand for beta form of plaster.

b. XRD does not differentiate alpha and beta forms of plaster because there are no structural differences.

c. Shape of plaster particles as seen in immersion mounts with a polarizing microscope provides a simple method for distinguishing the two forms of plaster. The alpha form is characterized by particles showing crystal faces (euhedral to subhedral shape) while the beta form lacks crystal faces (anhedral shape).

d. DTA can be used to differentiate between alpha and beta plaster. The temperature range of most interest on DTA curves for determining alpha and beta hemihydrate forms is in the range 100° to 200° C. The major endotherm, if there is more than one, will be at a slightly lower temperature (~20° C) for the beta plaster. A sharp distinct exotherm at about 180° to 190° C is also characteristic of the alpha form.

e. SEM can be used to differentiate between alpha and beta forms of plaster on the same particle shape basis as seen by optical microscopy.

33. Based on consideration of the five methods evaluated (chemical and physical, XRD, optical microscopy, DTA, and SEM) for study of plaster, optical microscopy is the simplest to use to distinguish between alpha and beta forms of plaster. Combined chemical-physical testing supplemented by XRD to determine phase composition is best for overall characterization.

34. This study verified that plasters AD-660 and 661 were of the alpha form, and that AD-656 was of the beta form; it also showed that D-53 and AD-626 and AD-654 were of the alpha form and that AD-657 was of the beta form.

35. The retarder (AD-664) contained substantial amounts of both alpha and beta forms of plaster, apparently as a carrier for the organic phase. None of the methods described in this report identified the organic phase, but it was thought to be keratin. Beta-form plaster was more common than the alpha form in this retarder. Since the organic material in this retarder, and presumably in other retarders, has a distinctive endotherm at about 1013° C, it should be possible to recognize its presence in a plaster by DTA and to quantify it if desired.

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* Personal communication with Dr. R. A. Kuntze of Ontario Research Foundation and R. Lang of U. S. Gypsum.

36. Since the alpha form plasters require less water, they should be the preferable ones for repository sealing applications.

37. Since characterization and evaluation of plasters is feasible, it should not be necessary to procure proprietary plaster products for use as admixtures in compositions of matter used for repository sealing work.

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TABLE 1: REVISED REPORT

SUBJECT			DATE	PAGE
Geo Chemical Program (Memo 81-19)			1 Oct 81	1 OF 2 PAGES
SOURCE OF DATA				
Test of Plasters Job No. 441-S866.11SC51				WES-305-81
COMPUTED BY	CHECKED BY		SECTION	
			Cement & Pozzolan Group	

Chemical Tests	METHOD	D53*	AD-626	AD-654	AD-656	AD-657	AD-660	AD-661
Free Water	C471	0.79	0.27	0.17	0.43	0.47	0.18	0.11
Combined Water	C471	20.15	19.99	19.95	20.46	20.26	20.57	20.57
Carbon Dioxide(CO_2)	LECO	0.45	0.31	0.54	0.12	0.38	0.09	0.10
SiO_2 & Insoluble Matter	C471	0.71	0.22	0.54	0.29	0.32	0.37	0.52
Al_2O_3(Difference)	C114	0.64	0.42	0.43	0.27	0.21	0.15	0.30
Fe_2O_3	C114	0.05	0.05	0.05	0.05	0.03	0.04	0.03
Al_2O_3 + Fe_2O_3	C471	0.69	0.47	0.48	0.32	0.24	0.19	0.33
CaO	C471	32.14	32.24	32.42	32.25	32.51	32.61	32.33
MgO	C471	0.23	0.25	0.34	0.06	0.15	0.08	0.09
SO_3	C471	44.94	45.70	45.21	46.23	46.04	46.28	45.94
Na_2O	C114	0.02	0.01	0.01	0.01	0.01	0.01	0.01
K_2O	C114	0.08	0.09	0.05	0.00	0.00	0.01	0.00
Cl^-	AgCl	0.00	0.00	0.00	0.02	0.08	0.02	0.00
NaCl		0.00	0.00	0.00	0.01	0.01	0.01	0.00
Total (C471, Para 14)		100.10	99.45	99.65	100.17	100.38	100.38	99.99
Calculated Compounds								
% Gypsum		96.28	95.51	95.32	97.76	96.80	98.28	98.28
%SO_3 combined as gypsum		44.77	44.42	44.33	45.46	45.02	45.71	45.71
% excess SO_3		0.17	1.28	0.88	0.77	1.02	0.57	0.23
% anhydrite ($CaSO_4$)		0.29	2.18	1.50	1.31	1.73	0.97	0.39
% CaO combined as gypsum		31.36	31.11	31.05	31.84	31.53	32.01	32.01
% CaO combined as anhydrite		0.12	0.90	0.62	0.54	0.71	0.40	0.16
% calcium carbonate $CaCO_3$		1.18	0.41	1.34	0.00	0.48	0.36	0.29
% Magnesium Carbonate $MgCO_3$		0.48	0.52	0.71	0.13	0.31	0.17	0.19

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Table 2

SUBJECT	DATE	PAGE 2 OF 2 PAGES
Geo Chemical Program (Memo 81-19)	1 Oct 81	

SOURCE OF DATA
Test of Plasters Job No. 441-S866.11SC51 WES-305-81

COMPUTED BY	CHECKED BY	SECTION
		Cement & Pozzolan Group

Physical Tests	D53*	AD-626	AD-654	AD-656	AD-657	AD-660	AD-661
ASTM C-472-73							
Free Water, %	0.75	0.32	0.22	0.37	0.56	0.15	0.09
Fineness:							
Passing No. 30 Sieve %:	99.9	100.0	100.00	100.00	100.00	100.00	100.0
Passing No.100 Sieve %:	99.7	99.8	99.7	97.7	99.8	99.9	99.8
A.P., C-204, m^2/kg:	382	393	419	701	467	390	370
Normal Consistency, Paste**	29.2	27.0	26.7	51.7	56.7	26.7	32.5
Time of Set, Paste, min:	63	86	100	18	18	14	23
Normal Consistency, Mortar:	11	11	11	20	21	13	14
Time of Set, Mortar, Min:	63	80	50	20	18	11	18
Compressive Strength, psi:	6270	5840	6160	2760	2330	5750	5870
Density, C-188, Mg/m^3:	2.72	2.74	2.72	2.66	2.70	2.73	2.73

*Proprietary product number

**Sodium Citrate added to the plaster in accordance with section 6.2 of ASTM C472.

R. E. REINHOLD
Acting Chief, Cement & Pozzolan Group

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Table 3

SUBJECT	DATE	PAGE
Geo Chemical Program (Memo 81-19)	25 Mar 82	1 OF 2 PAGES

SOURCE OF DATA
Test of Plasters Job NO. 441-S866.12SC51 AD 661(2&3)

COMPUTED BY	CHECKED BY	SECTION
		Cement & Pozzolan Group

Chemical Test	% As Received mean ±	95% C.I.(N)	% As Gypsum	Method ASTM
Free Water	0.25 ±	0.44(2)	0.21	C471
Combined Water	5.84 ±	0.08(5)	20.37 ± 0.07(3)	C471
CO_2	0.11 ±	0.02(3)	0.09	C471
SiO2 + Insol.Res.	0.24 ±	0.11(4)	0.20	See note 1
R2O3	0.16 ±	0.10(7)	0.14	C471
CaO	38.77 ±	0.20(4)	32.87	C114
MgO	0.10 ±	0.05(4)	0.08	C114
SO3	53.85 ±	0.28(4)	45.66	C471
NaCl	0.00 ±	0.00(2)	0.00	C471, See note 2
Total	99.32		99.62	
Fe2O3	0.04	(1)	0.03	C114
Al2O3	0.12		0.10	C114(Al2O3=R2O3-Fe2O3)
Calculated Compounds				
% Gypsum		97.33		
% SO3 Combined as gypsum		45.26		
% excess SO3		0.40		
% anhydrite(CaSO4)		0.68		
% CaO combined as gypsum		31.70		
% CaO as anhydrite		0.28		
%CaCO3		1.59		
%MgCO3		0.17		

NOTE 1: 15 minute digestion in 25 ml. of 20% HCl, filter, wash with water ignite 950°C.

NOTE 2: Chloride was extracted according to ASTM C471, then titrated with mercuric nitrate, according to "Standard Methods for the Examination of Water and Wastewater", 15th edition, 1980, APHA, Washington, DC, p270.

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Table 4

SUBJECT	DATE	PAGE
Geo Chemical Program (Memo 81-19)	25 Mar 82	2 OF 2 PAGES

SOURCE OF DATA
Test of Plasters Job No. 441-S866.12SC51 WES-100-82 AD661(2 & 3)

COMPUTED BY | CHECKED BY | SECTION Cement & Pozzolan Grp

Physical Tests	
ASTM C472-79	
Free Water, %	0.35
Fineness:	
Passing No. 30 Sieve %:	100.0
Passing No.100 Sieve %:	99.9
A.P., C-204, m^2/kg:	358
Normal Consistency, Paste**	33.3
Time of Set, Paste, min:	9
Normal Consistency, Mortor:	41
Time of Set, Mortar, Min:	7
Compressive Strength, psi:	5540
Density, C-188, Mg/M^3:	2.74
Density, C-472, #/cu.ft.:	124.72

**Sodium Citrate added to the plaster in accordance with section 6.2 of ASTM C472.

R. E. REINHOLD
Acting Chief, Cement & Pozzolan Group

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JANUARY 1951

Table 5

Crystalline Phases Identified in Six Plasters and One Plaster Retarder by X-Ray Diffraction

Crystalline Phases	Plasters						Retarder
	D-53	AD-654	AD-656	AD-657	AD-660	AD-661	AD-664
Calcium Sulfates							
Hemihydrate	X	X	X	X	X	X	X
Anhydrate	X	X	n.d.*	X	X	X	X
Other Phases							
Calcite	X	n.d.	X	X	X	X	n.d.
Dolomite	X	X	X	X	X	X	X
Celestite	X	X	X	n.d.	X	X	n.d.
Quartz	X	X	X	X	X	X	n.d.
Tridymite	Possible	n.d.	Possible	n.d.	n.d.	n.d.	n.d.
Plagioclase Feldspar	X	X	n.d.	X	n.d.	Possible	n.d.
Potassium Feldspar	X	n.d.	n.d.	Possible	n.d.	X	n.d.
Clay-Mica**	X	X	X	X	X	X	n.d.
Chlorite	X	n.d.	X	n.d.	X	X	n.d.
Smectite†	n.d.	n.d.	n.d.	X	n.d.	n.d.	n.d.
Kaolinite	X	n.d.	Possible	X	Possible	Possible	n.d.
Brucite	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	X

* Not detected.

** Clay-sized mica or illite or both.

† Swelling clay; the montmorillonite-saponite group.

Table 6

Chemical Composition of Three Plasters and One Plaster Retarder

Elements	Plasters				Retarder
	AD-626*	AD-656*	AD-661**		AD-664*
Calcium	X	X	X	X	X
Sulfur	X	X	X	X	X
Silicon	X	X	X	X	X
Aluminum	Trace	Trace	Trace	X	X
Potassium	Trace	Trace	Trace	X	X
Iron	Trace	Trace	Trace	X	X
Strontium	X	X	X	X	X
Chlorine	Trace	n.d.†	n.d.	n.d.	X

* As-received samples.

** Insoluble residue on a glass slide after heat treatment.

† Not detected.

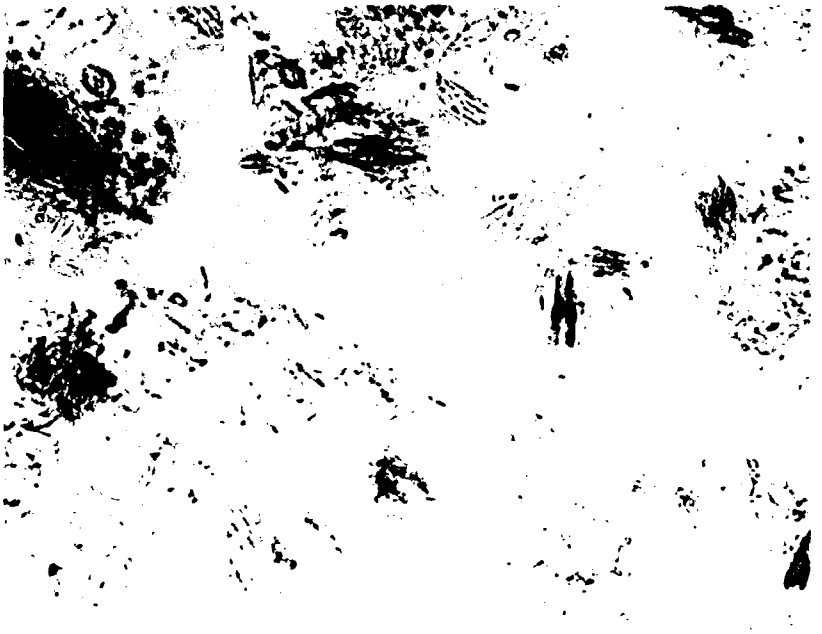

Figure 1. Beta form plaster AD-656 in plane polarized light, X 215

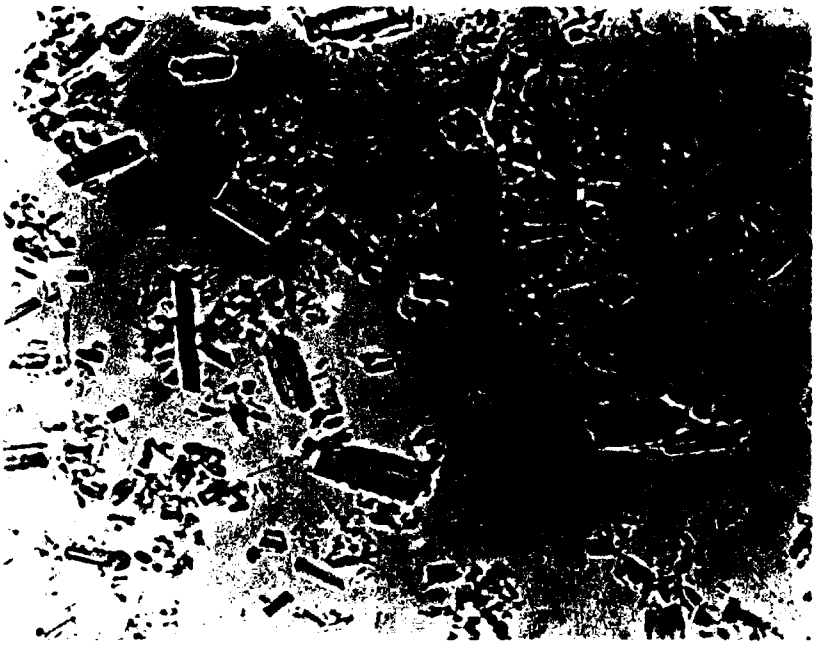

Figure 2. Alpha form plaster AD-661 in plane polarized light, X 215

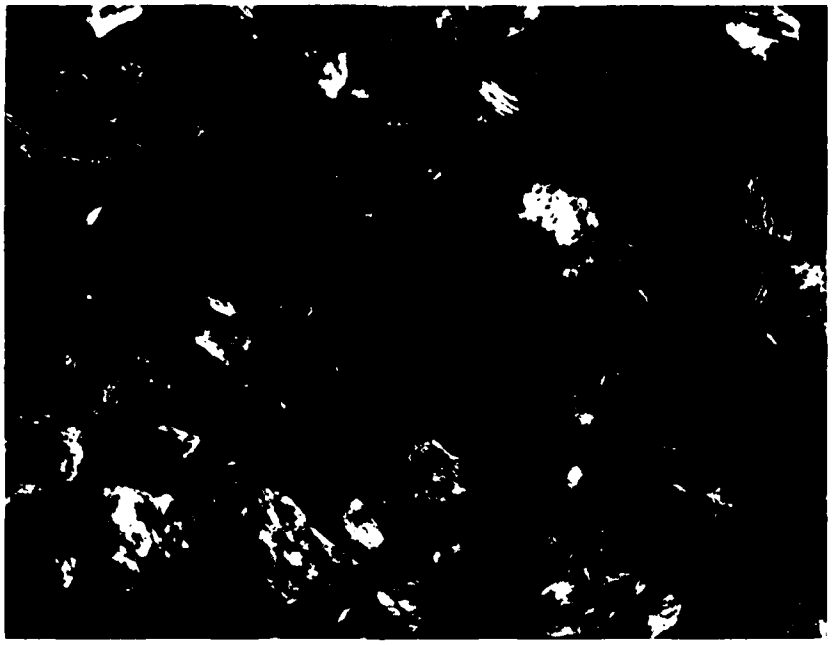

Figure 3. Beta form plaster AD-656 in crossed polarizers, X 215. Same field of view as Figure 1

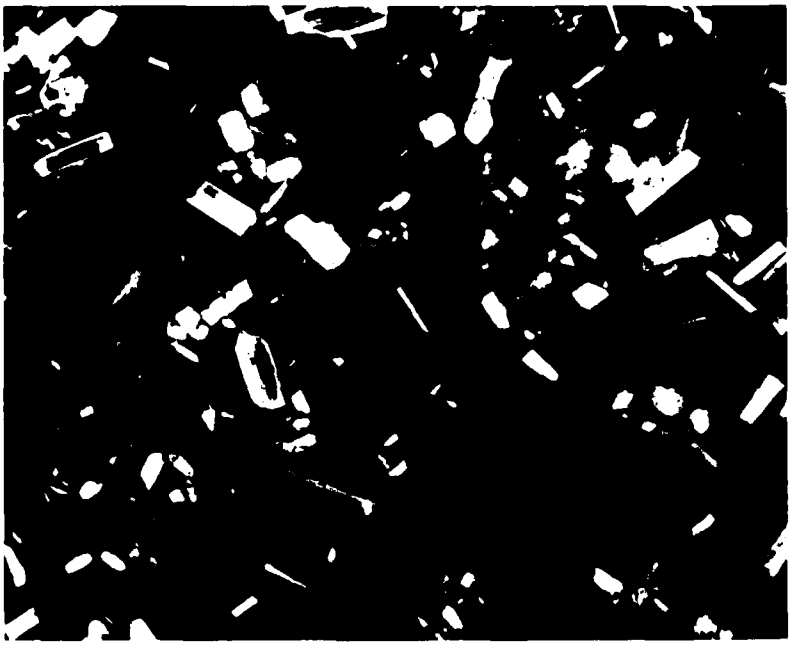

Figure 4. Alpha form plaster AD-661 in crossed polarizers, X 215. Same field of view as Figure 2

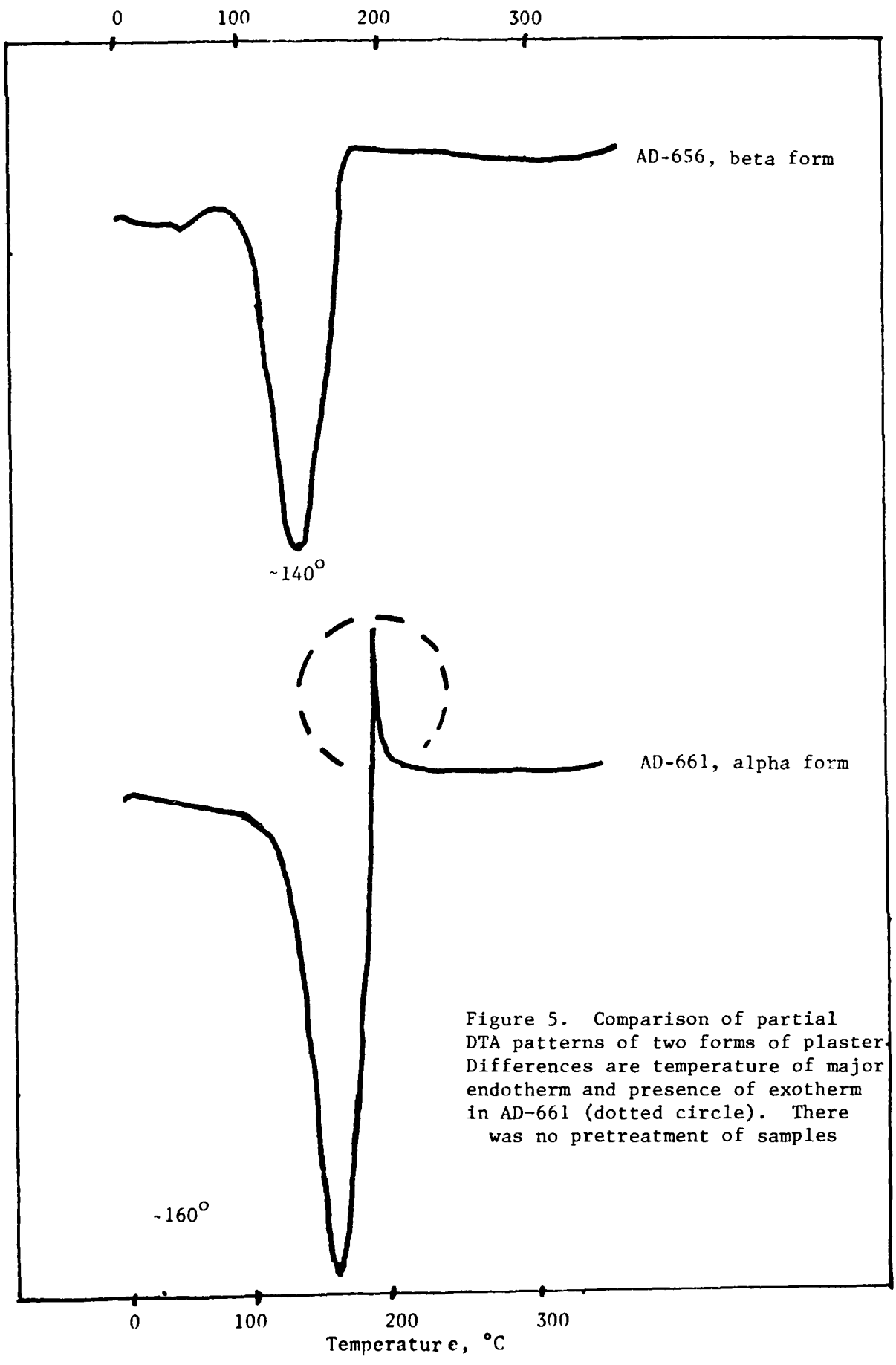

Figure 5. Comparison of partial DTA patterns of two forms of plaster. Differences are temperature of major endotherm and presence of exotherm in AD-661 (dotted circle). There was no pretreatment of samples

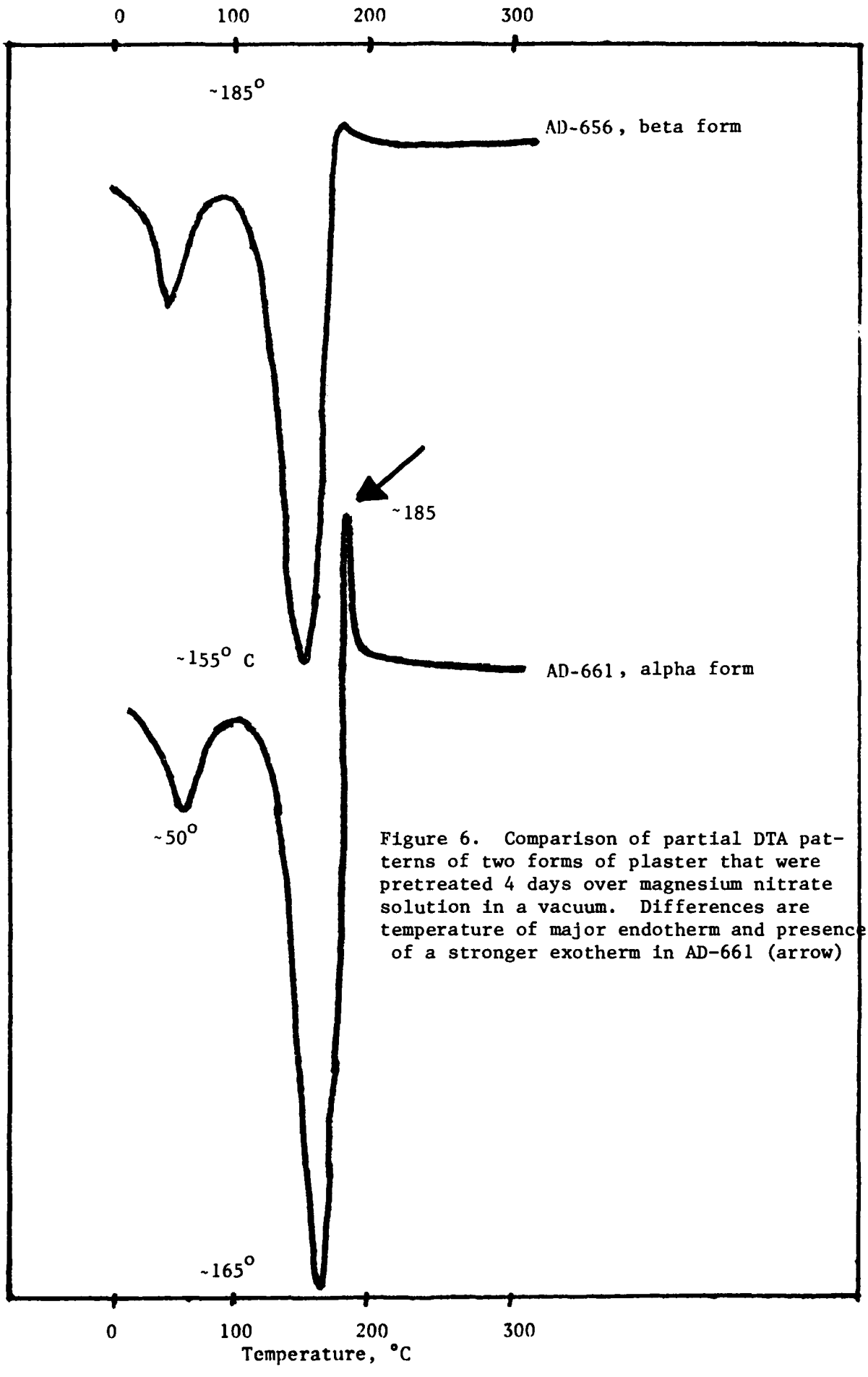

Figure 6. Comparison of partial DTA patterns of two forms of plaster that were pretreated 4 days over magnesium nitrate solution in a vacuum. Differences are temperature of major endotherm and presence of a stronger exotherm in AD-661 (arrow)

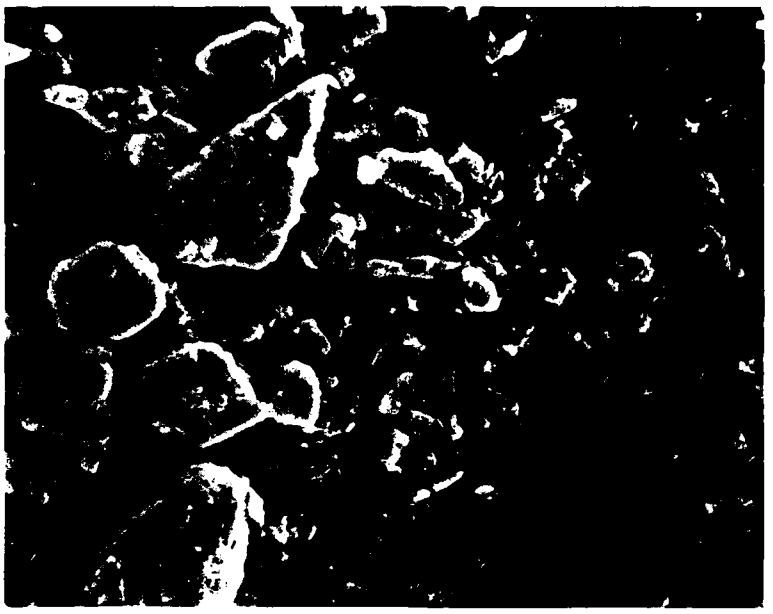

Figure 7. Beta form plaster AD-656, X 500 (Micrograph 06 1181-2). Shows absence of crystal faces on individual particles

Figure 8. Alpha form plaster AD-661, X 200 (Micrograph 06 1181-6). Shows presence of one or more crystal faces on particles

Figure 9. Plaster retarder AD-664, X 1000 (Micrograph 06 1181-7). Contains both forms of plaster seen in Figures 7 and 8 with more of the beta type (crystal faces absent). The large flat piece near the center that is covered with smaller particles may be keratin

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