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3683 WHITTAKER, CLARK - - -

## PROJECT COST RECORD

PROJECT TITLE: talc analysis by SEM or TEM

PROJECT NO.: MA 8683

SPONSOR: WHITTAKER, CLARK & DANIELS, INC.

DATE RECEIVED: 12 26 79

ADDRESS: ~~1000 Coolidge St~~

P.O. NO.: \_\_\_\_\_

~~South Plainfield, NJ 07080~~

COMMITMENTS: \$\_\_\_\_\_

Contact: Ray R. Krammes

Telephone No. : (201) 561 6100

SUMMARY OF PROBLEM:

Lab Notebook References:

No. \_\_\_\_\_ pp. \_\_\_\_\_ Initials \_\_\_\_\_

No.          pp.          Initials          \_\_\_\_\_

No.      pp.      Initials

Photomicrographic record no. \_\_\_\_\_

Sample disposal:

returned

filed (where) \_\_\_\_\_

discarded \_\_\_\_\_

## DIRECT COSTS

| (Exclusive of oper.<br>time on inst. below) |                       |        |           |               |          |             |      |
|---------------------------------------------|-----------------------|--------|-----------|---------------|----------|-------------|------|
| <u>Individual time</u>                      |                       |        |           | <u>Travel</u> |          |             |      |
| Date                                        | Name                  | Hours  | Charge    | Date          | Traveler | Destination | Cost |
| 12-26                                       | JCB<br><del>JCB</del> | 3<br>1 | 180<br>60 |               |          |             |      |
|                                             |                       |        |           |               |          |             |      |
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| <u>Instrument time</u>                      |                       |        |           |               |          |             |      |
|                                             | Date                  | Hours  | Charge    |               | Date     | Company     | Cost |
| SEM                                         | 12-26-79              | 1.5    | 113       | Photo         |          | In House    | 75   |
| TEM                                         | 1-31                  | 0.5    | 38        | Photo         |          |             | 15   |
| XRD                                         |                       |        |           | Materials     |          |             |      |
| EMP                                         |                       |        |           | Supplies      |          |             |      |
| IMP                                         |                       |        |           |               |          |             |      |
| ESCA                                        |                       |        |           |               |          |             |      |
| EMMA                                        |                       |        |           |               |          |             |      |
| MOLE                                        |                       |        |           |               |          |             |      |

Charge Summary

|              |    |
|--------------|----|
| Individuals  | \$ |
| Instrument   |    |
| Travel       |    |
| Outside      |    |
| Other        |    |
| Total Charge | \$ |

OK for billing  
Project Ldr \_\_\_\_\_  
Group Ldr \_\_\_\_\_

P.O. 20460 \$73

12074 293

$$\frac{1}{366}$$

TOTAL CHARGE \$ 366

BILLED TO CLIENT \$ \_\_\_\_\_

INVOICE NO. (S) 6019/31 Dec-19-f

1-28-80 new charge 113

PO.  
20692 / Rept 30 Jan 80

INV 61120-#113-f

PROJECT LEADER: J AB



Walter C. McCrone Associates, Inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

12 March 1982

Mr. Ray R. Krammes  
Whittaker Clark & Daniels, Inc.  
1000 Coolidge Street  
South Plainfield, New Jersey 07080

Dear Mr. Krammes:

We have examined the two talc samples you submitted for particle evaluation. They are identified as 14S04 and 14S45. These were examined on the scanning electron microscope (SEM) as the previous samples, and micrographs were taken at 220X and 1100X magnification (i.e., original magnifications on a 4 x 5 micrograph). Magnification bars are included on the 8 x 10 prints which properly relate to the particle sizes as enlarged.

Enclosed herewith for each sample are:

- 1) one 8 x 10 print at each magnification
- 2) one 2 x 2 slide at each magnification

Also enclosed are one 2 x 2 slide of each of the two 8 x 10 prints you submitted of Sample 229P73 and your original prints.

The two samples, 14S04 and 14S45, are very different. Sample 14S45 has a larger particle size range (i.e., larger and smaller particles) than Sample 14S04. Also Sample 14S04 is more flakelike than 14S45.

Thank you for consulting McCrone Associates and if you should have any questions concerning this work, please contact us.

Yours sincerely,

John A. Brown  
Research Associate

JAB:fe  
Encl.  
Ref: 8683

*Sent Certified*

2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616 • 312/842-7100 • CABLE CHEMICRONE

CONFIDENTIAL

McCrone2-00726

**SPEEDMESSAGE**

**WHITTAKER, CLARK & DANIEL S. NC.**

1000 COOLIDGE STREET  
SOUTH PLAINFIELD, N.J. 07080  
Phone: (201) 561-6100



M. 8683

DATE January 22, 1982

SUBJECT SEM Micrograph

to Mr. John Brown

Please discuss with Ian!

Please prepare micrograph of two talcs 14S45 and 14S04 at  
220X & 1100X.

We want 2 x2 slides and 8 x 10 prints.

Also 2 x 2 slides of two 8 x 10 prints identified as 229P73 Talc.

*Ray R. Krammes*

SIGNED Ray R. Krammes



Walter C. McCrone Associates, Inc.

CONSULTING ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

Date: 3-12-82

COMPANY: Whittaker Clark

PERSON (Contact): Ray Krammes

Dear Ray

Enclosed are the following data of your samples

SEM Prints original prints SEM Negatives ✓ Other \_\_\_\_\_

Examination date \_\_\_\_\_ of 14504  
14545

Your samples are:

Slides, prints, report under separate cover.

- 1) Enclosed \_\_\_\_\_
- 2) Discarded in two weeks unless otherwise notified \_\_\_\_\_
- 3) Taken back \_\_\_\_\_
- 4) Other \_\_\_\_\_

Thanks much for using our services.

Sincerely,

John A. Brown.

for W. C. McCrone Associates, Inc.

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

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walter c. mcrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

31 August 1981

Mr. Ray Krammes  
Whittaker, Clark & Daniels, Inc.  
1000 Coolidge Street  
South Plainfield, New Jersey 07080

Dear Mr. Krammes:

We have completed analysis of your talc sample using the CTFA J4-1 methodology. The sample was designated as 693R06 Talc.

Results of this analysis are given in the attached data sheet and the x-ray diffraction pattern.

Thank you for consulting McCrone Associates and if you should have any questions concerning these results, please contact us.

Yours sincerely,

Ralph J. Hinch, Jr.  
Research Chemist

RJH:fe  
encl.  
ref: 3591

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McCrone2-00729

Date 8-19-81

Client Whittaker, Clark & Daniels MA Project 3591

Sample Description G93 R06 TALC

X-RAY DIFFRACTION

Serpentine: Detected ✓ Not Detected         

Amphibole: Detected ✓ Not Detected         

Other Phases Present: Chlorite ✓ (?)  $\alpha$ -SiO<sub>2</sub> ✓ (trace)

Dolomite ✓ Calcite ✓ Magnesite          Mica ✓

Other:         

Comments:         

RJ Kinch, Jr

LIGHT MICROSCOPY

Chrysotile not detected %  $\alpha$ -SiO<sub>2</sub> trace %

Tremolite-Actinolite 0.1-0.5 total %          % fibrous

Nonfibrous Serpentine not detected

Others:         

Comments: All of the tremolite-actinolite is fibrous

Clark & Daniels

walter c. McCrone associates, inc.

X-RAY DIFF ACTION DATA

Proj No. 9

Sample No. 8

Att. 8

Power 5

Filter 50

R. di. uom. 47.2

Filter 8

Neutralize 0

S. Am. 4.4

Subst. 2.0

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Operator 15 81

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**SAMPLES**

FROM

**WHITTAKER, CLARK & DANIELS, INC.  
1000 COOLIDGE STREET  
SOUTH PLAINFIELD, NEW JERSEY 07080**

**To**

**Walter C. McCrone Association**

**2820 South Michigan Avenue**

**Chicago, Illinois 60616**

**Mrs. Lucy McCrone**

MA-  
3591

693R06 Talc

(CTFA Method J-4-1)

19 Aug 81

to Ralph

RRK/hm/RRK

Date 8-17-81

Forwarded Via: .....

Date Sent: .....

CONFIDENTIAL

McCrone2-00732



walter c. mcrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

30 January 1980

Mr. Ray R. Krammes  
Whittaker, Clark & Daniels, Inc.  
1000 Coolidge Street  
South Plainfield, New Jersey 07080

Dear Mr. Krammes:

We have examined your latest talc sample 339P73 by the scanning electron microscope and have taken comparable micrographs to those of the previous five samples (original magnifications were 220X and 1100X). *should be 229P73*

Enclosed are the following items:

2 x 2 in. slides - 1 each at 220X and 1100X  
8 x 10 in. prints - 2 each at 220X and 1100X  
4 x 5 in. prints and negatives - 1 each at 220X and 1100X

Thank you for consulting McCrone Associates and if you have any questions concerning this analysis, please call me.

Yours sincerely,

John A. Brown  
Research Associate

JAB:fe  
Encl.  
Ref: 8683

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McCrone2-00733



Walter C. McCrone Associates, Inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

2 January 1980

Mr. Ray R. Krammes  
Whittaker, Clark & Daniels, Inc.  
1000 Coolidge Street  
South Plainfield, New Jersey 07080

Dear Mr. Krammes:

We have examined the five talc samples you submitted to Mr. Ian Stewart via your letter of 19 December 1979. You desired a microscopic evaluation of these five samples to culminate in visual results such as 2x2 inch slides and 8x10 inch prints. The five samples were labeled 229P41, 229P50, 229P15, 229P28 and 229P09.

We chose to make the examination with the scanning electron microscope (SEM) because the range of magnifications is easier than on the TEM, and also the SEM provides information of the surface texture of particles which cannot be done on the TEM unless replicas of the particles are made.

SEM examination was made at various magnifications to evaluate the samples. You initially requested a single magnification to show surface texture and particle morphology. Because of the wide range of particle sizes in these samples, one magnification cannot show both features. Consequently after our phone conversation regarding this matter, two magnifications were chosen instead of one, at your request. The most appropriate magnifications were 220X to show particle distribution and 1100X to show particle surface detail. Slides were made at both magnifications for each sample and two each 8x10 inch prints of each sample were also made.

Each slide and print has a magnification mark on the image. At 200X the mark is  $10^{-4}$  (meter) which is 100  $\mu\text{m}$  (micron). At 1100X the mark is  $10^{-5}$  m which is 10  $\mu\text{m}$  long. The mark changes size when photographically enlarged so that it is an internal standard to which the particle sizes can be compared. The prints and slides have a sample identity at the bottom. Also the original magnification taken from the SEM directly on a 4x5 inch film is noted at the bottom right. This magnification is only good for the 4x5 inch prints which are also enclosed.

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McCrone2-00734

Mr. Ray R. Krammes  
Page two

Evaluation of the particle morphology and surface detail in terms of odor retention at this stage is unknown. There is a great difference between samples in particle size distribution which may or may not relate to odor retention. Certainly there is a big difference in total surface area which could relate.

Particle surface texture is diverse enough from particle to particle as well as from one part of a large particle to another part that a simple evaluation in terms of a single micrograph is not statistical enough to have a high enough confidence level to be usable. In order to use this feature for evaluation, a rather long and involved research effort would be required.

One feature of the particles which could relate to odor retention is the fact that the large particles are dense while the smaller particles are somewhat extended. If the large particles have very fine pores then odor retention would be good for them, while if they are solid then odor would be poor. The smaller particles because of higher surface area per weight may have better odor retention if the odor has good retention on the surface.

Evaluating these aspects could best be done by microtomy and intensive TEM evaluation, or by the B.E.T. nitrogen adsorption techniques which determine surface area and pore size distribution. Other adsorption techniques similar to the B.E.T. method might also be used with the odor material to evaluate odor retention if this were done on specially selected particle size powders.

Enclosed with this report are the following:

- 2x2 in. slides - 1 each at 2 magnifications on each sample
- 8x10 in. prints - 2 each of the area shown on the slides
- 4x5 in. prints - 1 each of the area shown on the slides
- 4x5 in. negatives - 1 each of the area shown on the slides

The 4x5 inch prints and negatives were the initial results taken directly from the SEM.

Mr. Ray R. Krammes  
Page three

Thank you for consulting McCrone Associates. If you should have any questions or comments regarding this work, please feel free to contact me.

Yours sincerely,

John A. Brown  
Research Associate

JAB:fe  
Encl.  
Ref: 8683

P.O. 20460 and 12074



# WHITTAKER, CLARK & DANIELS, INC.

1000 COOLIDGE STREET • SOUTH PLAINFIELD N J 07080

(201) 561-6100

CABLE "SLEINAD"

Telex: 138248

December 19, 1979

Mr. Ian M. Stewart  
Walter C. McCrone Associates  
2820 South Michigan Avenue  
Chicago, Illinois 60616

RECEIVED DEC 21 1979 *Samples too!*

Dear Mr. Stewart:

This is to confirm our phone conversation of Wednesday, December 19, 1979. The five talcs sent to you, namely 229P41, 229P50, 229P15, 229P28 and 229P09, are to be examined either in the TEM or SEM.

They should be examined under identical conditions and the same magnification. The preparation and magnification are left to your choice. We would like to show differences, if any, in particle shape and surface.

The resultant micrographs would then be used to prepare one 2 x 2 projection slide for each talc. We would also need two 8 x 10 prints of each of the field selected for the projection slide. On both the prints and the slide, we would like a measure of size, ie. 5, 10, 20 microns, whichever is appropriate.

We have a deadline whereby the slides and prints must be in our hands by January 8, 1980.

Very truly yours,

WHITTAKER, CLARK & DANIELS, INC.

*Ray R. Krammes*

Ray R. Krammes  
Manager-Technical Services

RRK/ceb

*8683 / J. Brown*

Branch Offices:

Chicago  
651 Executive Drive  
Willowbrook, Illinois 60521  
(312) 323-5775 & 5777

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1900 Big Beaver Road  
Troy, Michigan 48064  
(313) 640-6620 & 6621

Ohio  
2598 Merrittview Lane  
Cincinnati, Ohio 45231  
(513) 825-3424

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280 Bloor Street West  
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Ontario  
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McCrone2-00737

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For Whitaker  
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Folder  
not to be sent  
thanks  
Jab



