

UNITED STATES DEPARTMENT OF THE INTERIOR  
GEOLOGICAL SURVEY

Sample preparation procedures for the analysis  
of clay minerals by X-ray diffraction

(A workshop syllabus prepared for the  
Denver X-ray conference at Denver University,  
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This report is preliminary and has not been reviewed for conformity with U.S. Geological Survey editorial standards and stratigraphic nomenclature. Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Geological Survey.

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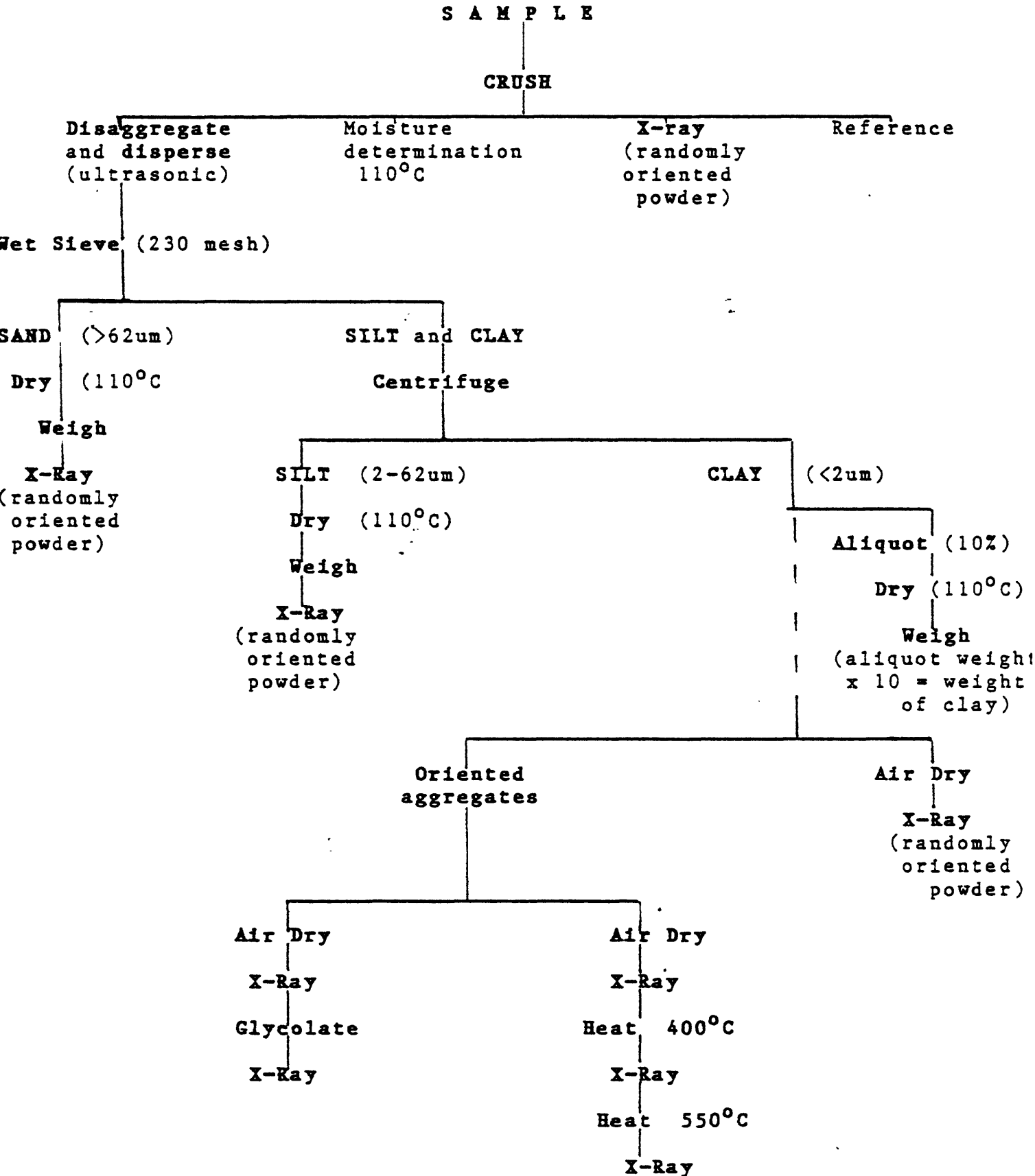
## A C K N O W L E D G M E N T S

Much of the procedural section of this manual comes from descriptions of techniques used in the Sedimentary Mineralogy Laboratory of the U.S. Geological Survey in Denver by Harry Starkey and Paul Blackmon. Their methodology has been formalized in U.S. Geological Survey Bulletin 1563, Starkey, H. C., Blackmon, P. D., and Hauff, P. L., in press, "The routine mineralogical analysis of clay-bearing samples," which may be available in late 1984. Other sections have been extracted from "ORIENTED SAMPLE MOUNTS FOR THE ANALYSIS OF CLAY MINERALS BY X-RAY DIFFRACTION," a syllabus prepared for the Clay Minerals Society Workshop conducted at the 1982 Annual Meeting in Hawaii.

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.....FLOW SHEET FOR SAMPLE TREATMENT PROCEDURES.....



## I. INTRODUCTION

This Syllabus is an informal presentation of the general procedures involved in routine sample preparation for clay mineralogy analyses. There is no intention to be all encompassing. The methods described and referenced are those in common use among Clay Mineralogists. They have been tested and found to give consistent results. Each worker must evaluate the procedure best suited to their applications.

### A. BIBLIOGRAPHIES

Work in any subject should be well grounded in reference materials. To that end, six bibliographies have been included with this outline. Procedures described throughout the outline are referenced where applicable.

### B. SOURCE REFERENCE CLAY MATERIALS

The Clay Minerals Society maintains a Source Reference Clay Collection. Samples of the more common and some rare clay species are available for a nominal charge. Contact:

Dr. W.D. Johns  
Department of Geology  
University of Missouri  
Columbia, Missouri 65211  
(314) 882-3785

## II. ACHIEVING THE CLAY FRACTION SUSPENSION

Rock samples are fractionated into **sand** (<62 $\mu$ m), **silt** (2-62 $\mu$ m) and **clay** (<2 $\mu$ m) sizes for ease of identification and estimate of relative amounts present.

### A. CRUSHING AND SPLITTING

1. Sample is crushed, not ground to 3-5mm particle diameter
2. A 2 gram split is used for whole rock XRD
  - a. helps determine soluble salts present
  - b. determines what pre-treatments and supplementary treatments are required
3. 25-30 gram (average) split for clay work
4. 1 gram split for moisture determination
  - a. weighed
  - b. heated for 1 hour at 110°C
  - c. weighed
  - d. sample weight varies with relative humidity

Table 1.--Chart showing representative centrifuging times for various particle size separations

Temp °C	T Min - sec	T - t <sub>d</sub> Min - sec	Temp °C	T Min - sec	T - t <sub>d</sub> Min - sec	Temp °C	T Min - sec	T - t <sub>d</sub> Min - sec
16	7 - 30	7 - 00	24	6 - 17	5 - 47	32	5 - 23	4 - 53
17	7 - 19	6 - 49	25	6 - 09	5 - 39	33	5 - 16	4 - 46
18	7 - 09	6 - 39	26	6 - 02	5 - 32	34	5 - 11	4 - 41
19	6 - 59	6 - 29	27	5 - 55	5 - 25	35	5 - 06	4 - 36
20	6 - 50	6 - 20	28	5 - 48	5 - 18	36	5 - 01	4 - 31
21	6 - 42	6 - 12	29	5 - 41	5 - 11	37	4 - 56	4 - 26
22	6 - 34	6 - 04	30	5 - 36	5 - 06	38	4 - 51	4 - 21
23	6 - 25	5 - 55	31	5 - 29	4 - 59	39	4 - 46	4 - 16

T = total time in the centrifuge

t<sub>a</sub> = time of acceleration = 30 seconds

t<sub>d</sub> = time of deceleration = 30 seconds

For less than 5 m u particle size, (240 RPM centrifuge speed)

For less than 2 m u particle size, (600 RPM centrifuge speed)

For less than 1 m u particle size, (1200 RPM centrifuge speed)

Assumed density of particles = 2.65

## B. DISAGGREGATION AND DISPERSION OF CLAY SPLIT

1. Soak overnight in ~200ml of distilled water
2. Ultrasonic treatment
  - a. 5-15 minutes to disaggregate and disperse
  - b. mechanical stirrer may also be used
3. If sample not disaggregated than crush (do not grind) gently - preferably with rubber pestle.  
Repeat ultrasonic or stirring action.

## C. SAND REMOVAL

1. Wet sieve through 230 mesh sieve to remove >62um sand size particles....save water and fines
2. Use a brush (typewriter eraser) - gently - to separate fine material from the sand
3. Sand size fraction
  - a. oven dry at 110°C
  - b. cool in desiccator
  - c. weigh to determine its percentage of whole sample
  - d. retained for later XRD

## D. SEPARATION OF SILT AND CLAY

1. Silt and clay are separated by **centrifugation** using Stoke's Law of settling
2. Assumptions which may bias results
  - a. density is taken at 2.65
  - b. particles assumed to be spherical (clays are usually platelets)
  - c. constant and reproducible acceleration and deceleration of the centrifuge
3. Flocculation
  - a. if clay is not in suspension, wash repeatedly with distilled water to deflocculate.
  - b. sodium hexametaphosphate (~0.25grams) may be added to encourage deflocculation
  - \*\*\*\* c. care must be taken when adding any chemicals to clay suspensions
4. Centrifugation time
  - a. clay/silt mixture put into 250ml centrifuge bottle
  - b. formula for calculating time to settle is given in Hathaway (1956)
  - c. TABLE I is a typical chart
  - \*\*\*\* d. note chart must be constructed for individual centrifuge
  - e. viscosity of water is temperature dependent

5. Centrifugation procedure
  - a. 250ml bottles are filled, balanced and placed into centrifuge
  - b. centrifuge is set so required speed (from Table I) can be reached in 30sec
  - c. required speed is maintained for determined time
  - d. deceleration must also be accomplished within 30 sec
  
6. Silt separation
  - a. when centrifuge is stopped, supernatant liquid is decanted into tall (Berzelius) 1000ml beakers
  - b. centrifugation and decantation is continued until supernatant liquid is clear enough to see through
  - c. silt fraction now remains in bottle
    - 1) transferred to evaporating dish
    - 2) oven dried at 110°C
    - 3) cooled in desiccator
    - 4) weighed
    - 5) retained for XRD

#### E. CLAY SIZE FRACTION

1. Clay is now suspended in large volume of water which must be reduced
  - a. drying clays out completely makes re-suspension difficult
  - b. therefore most, but not all, water must be removed
  
2. Water removal is done with a bacteriological filter candle connected to air and vacuum lines
  
3. Candle is placed in 1000ml Berzelius beaker
  - a. entire surface of candle is wetted
  - b. vacuum applied
  
4. Clay filter cake forms on filter candle.
 

Back pressure with air intermittently to remove cake from candle.
  
5. Process is continued until volume of suspension reduced to < 500ml
  
6. slurry
  - a. transferred to volumetric flask and brought to volume (500, 250, 100 ml)
  - b. flask shaken thoroughly
  - c. 10% by volume aliquot withdrawn by pipet
    - 1) transferred to evaporating dish
    - 2) oven dried at 110°C
    - 3) cooled in desiccator
    - 4) weighed
    - 5) weight = 10% of total clay fraction
  - d. 5-10ml of slurry is transferred to test tube for oriented mount



- e. remainder of slurry
  - 1) returned to filter candle
  - 2) nearly all water withdrawn
  - 3) air dried
  - 4) used for random powder XRD scan
    - a) for 060 determination
    - b) to determine any non-clays present

## F. RAPID SAMPLE PREPARATION PROCEDURE

### III. P R E - T R E A T M E N T S

#### A. INTRODUCTION

As few chemical treatments as possible should be used to avoid damaging or changing the clays in the sample (Grim, 1968, p.211; Brewster, 1980; Johns and Kurzweil, 1979). However, chemical pre-treatments become unavoidable in many instances. Other methods should be tried before chemicals are introduced into the sample.

#### B. ORGANIC MATERIAL REMOVAL

1. Organics mask peaks, lower intensities, and increase background
2. Methods
  - a. Langeveld and others, 1978
    - 1) comparison of 8 methods
    - 2) bromine oxidation or sodium hypobromite best
  - b. Troell, 1931
    - sodium hypobromite
  - c. Anderson, 1963
    - 1) sodium hypochlorite
    - 2) efficient and less destructive to clays and workers  
other methods i.e hydrogen peroxide
  - d. Hydrogen Peroxide Method
    - 1) 20-30% hydrogen peroxide
    - 2) gently heat on steam bath
    - 3) can lose pyrite with this method
    - 4) can create Ca-bearing minerals  
Jones and Beavers, 1963  
Martin, 1954
    - 5) can degrade the clays  
Douglas and Fiessinger, 1971

#### C. CARBONATE REMOVAL

1. Carbonates mask trace minerals  
carbonate cement interferes with size fractionation
2. Methods
  - a. HCl
    - 1) Grim, 1937 - weak solution
    - 2) Jurik, 1964 - 1N + heat
    - 3) Ellingboe and Wilson, 1964 - 10%
    - 4) Ostrom, 1961 - 0.11N

- b. Acetic Acid
  - 1) St. Clair, 1935
  - 2) Gault and Weiler, 1955
  - 3) Ostrom, 1961 - 0.3N
  - 4) Jackson, 1956 -  
method least destructive to clays
- c. The acid methods will attack the clays  
 Hectorite very susceptible (Ray and others, 1957)
- d. EDTA - removes magnesium from saponite and hectorite
  - 1) Hill and Runnels, 1960
  - 2) Glover, 1961
  - 3) Bodine and Fernalld, 1973

#### D. IRON OXIDE REMOVAL

- 1. Iron fluoresces producing a high background which can mask peaks
- 2. Method
  - a. Mehra and Jackson, 1960
  - b. dithionate-citrate buffered with sodium bicarbonate

#### E. AMORPHOUS MATERIAL REMOVAL

- 1. Amorphous material masks presence and amounts of clay minerals
- 2. Methods
  - a. Iron oxide (see D. above)
  - b. Silica
    - 1) Hashimoto and Jackson, 1960
    - 2) weighed sample placed in nickel beaker
    - 3) boiled in 0.5N NaOH
    - 4) quenched in ice water
    - 5) supernatant removed by centrifugation
    - 6) oven dried
    - 7) cooled
    - 8) re-weighed

### IV SUPPLEMENTARY PROCEDURES

#### A. SOLVATION

- 1. Solvation is the introduction of an organic molecule (ethylene glycol or glycerol are most common) into the interlayer positions of a clay mineral to promote expansion of swelling layers. The purpose being to differentiate between specific clay minerals.
- 2. The solvate can be applied by dripping manually onto the oriented mount; by placing the mount in a solvate vapor for an appropriate time; and by soaking the clay suspension with the solvate and then making the oriented mount.
- 3. Ethylene glycol is commonly used to differentiate smectites from other clay minerals (Brunton, 1955; Kunze, 1955). It is easy to apply (commonly by vapor) but may not remain in the lattice consistently over a useful time frame.

4. Glycerol is used to distinguish high and low charge varieties of smectites (Greene-Kelly, 1955) and between vermiculites and smectites (Brindley and Brown, 1980; Brindley, 1966). It is more difficult for some clays to accept (recommended procedure is to soak suspension overnight in glycerol solution) but remains in the lattice for long periods of time.

#### B. CATION SATURATION AND C.E.C.

1. Cation saturation is used to exchange a specific cation into the clay lattice to assure consistent responses to subsequent treatments.
  - a. Greene-Kelly Lithium test
  - b. charge differentiation in smectites and vermiculites
  - c. Potassium saturation to distinguish vermiculites
2. Cation Exchange Capacity  
Uses saturation with a cation to flush interlayer cations into supernatant which is then analyzed relative to species and capacity.
3. A bibliography relative to cation saturation is included

#### C. GREENE-KELLY LITHIUM TEST TO DISTINGUISH SMECTITES

1. to distinguish montmorillonite from trioctahedral smectites and beidellite
2. Method - Greene-Kelly, 1955; Schultz, 1978
  - a. saturate oriented aggregate with 3N LiCl
  - b. heat overnight at 200°C
  - c. treat with glycerol
  - d. montmorillonite does not expand
  - e. trioctahedral smectites and beidellite do expand

#### D. KAOLINITE VS. CHLORITE

1. 550°C
  - a. 14Å peak of chlorite increases
  - b. 7Å peak of kaolinite vanishes
  - c. 7Å chlorite can also vanish or be too small to detect
2. IF both well crystallized then the 004 chlorite peak at 3.55Å and the 002 kaolinite peak at 3.58Å can be distinguished.
3. Boiling in 2N HCl for 30 minutes removes the chlorite
4. Grind sample with potassium acetate to expand lattice (Andrew and others, 1960; Wada, 1961)

5. Grind sample with cesium chloride; treat with hydrazine and DMSO  
(Jackson and Abdel-Kader, 1978)

#### E. PHYSICAL CONCENTRATION

1. Further size fractionation of clays to  $<1\mu\text{m}$ ,  $<0.5\mu\text{m}$ ,  $<0.25\mu\text{m}$  can concentrate a specific mineral.
2. Heavy liquids (Bromoform) concentrate non-clays such as pyrite, magnetite, hematite and other heavy minerals
3. Magnetic separation using a Franz Isodynamic Separator

#### V. ORIENTED XRD MOUNTS

This subject is covered in great detail in the Syllabus "ORIENTED SAMPLE MOUNTS FOR THE ANALYSIS OF CLAY MINERALS BY X-RAY DIFFRACTION". Summary sections have been extracted from that Syllabus and included here.

##### A. WHY USE ORIENTED MOUNTS

1. As clays are phyllosilicates and usually crystallize in platelets, the basal reflections are naturally the strongest.
2. Basal reflections (001) are emphasized with an oriented mount; intensities are increased.
3. Clays typically have broad peaks which do not show up well in random mounts.
  - a. this can be a function of small crystal size
  - b. of mixed-layering
  - c. of a mosaic structure in larger crystals such that individual diffracting domains are only a few unit cells in size
4. Identification criteria for clays is based on the position of their 001 spacings and reactions of those spacings to treatments (heat, solvation, cation saturation).
5. Oriented samples are used to identify type, amount, and stacking arrangement of mixed-layer clays.
6. Enhancement of the 001 peaks is accompanied by a reduction in other, non-001 clay peaks and those of non-clay minerals which simplifies mineral identification by eliminating some overlapping of peaks.

## **B. SUBSTRATES**

.....I N T R O D U C T I O N.....

This section looks in detail at the various types of materials available as substrates for oriented mounts for X-ray diffraction analysis.

The three most commonly used materials are glass slides, ceramic tiles and cellulose membrane filters.

A compilation of detailed manufacturers specifications and comparison data from X-ray diffraction scans, energy dispersive spectra (EDS) and scanning electron microscope (SEM) photos are presented here for the readers' evaluation.

There is no intent by the editors to recommend any one product over another. Rather a wide choice of substrates is illustrated so that the user may decide which best suits his specific needs.

Sources of materials were also chosen indiscriminantly. Where possible an attempt has been made to give a minimum of two suppliers for each type of substrate. However, the important consideration is that only materials from those sources which the Editors' personally tested have been listed. Descriptive terminology is based on that used in the manufacturers' catalogs.

Some inappropriate materials have also been included. This is done to save the user time. Many catalog and manufacturer descriptions are incomplete.

The editors welcome additional information.

.....TYPES OF SUBSTRATES.....

A. GLASS

1. Petrographic Microscope Slides

- a. source
  - any scientific supply house
- b. format/physical characteristics
  - 1) 1"x3"
  - 2) can be cut to fit diffractometer sample holder
- c. comments
  - 1) can be stored
  - 2) inexpensive/disposeable
  - 3) easily cleaned with water

2. High Temperature Glass Slides -- Vycor

- a. source
  - 1) manufacturer
    - Corning Glass Works
    - Corning, New York
  - 2) fabricators
    - Corning sells only raw material to distributors who fabricate it into slides
    - a) Swift Glass Co.
      - 22nd street
      - Elmira Heights, new York 14902
      - (607) 733-7166
    - b) F.J. Gray Company
      - Jamaica, New York
      - (212) 297-4444
    - c) Industrial Glass Inc.
      - Sudden Service Glass Division
      - 3203 Fowler Street
      - Los Angelos, Calif. 90063
    - d) Optical Instrument Laboratory
      - P.O. Box 608
      - Bellaire, Texas 77401
      - (713) 772-7294

These are only a few of the possible distributors --  
A complete list is available from Corning

- b. format
  - cut and polished to customer's specifications
- c. comments
  - 1) somewhat expensive
  - 2) can be heated to +700°C
  - 3) reuseable

### 3. Fused Silica

#### a. source

Optical Instrument Laboratory  
P.O. Box 608  
Bellaire, Texas 77401  
(713) 772-7294

#### b. format/physical characteristics

- 1) 1" x 13/4" x 1/2 mm  
can be cut and polished to specifications
- 2) not 100% silica - has contaminants
- 3) will tolerate temperatures to 1225°F

#### c. comments

- 1) synthetically made

### 4. Porous Vycor Glass Plates

..an intermediate product obtained in manufacture of  
impervious SiO<sub>2</sub> glass

#### a. source

Corning Glass Works  
Scientific Glassware Department - Product Division  
Tubing OEM Sales  
Corning, New York 14830  
(607) 974-9000

#### b. format/physical characteristics

- 1) standard flat sheets 12"x12" (1/8, 1/4, 3/8" thick)  
other sizes available
- 2) can be cut to specified shapes
- 3) 96% SiO<sub>2</sub> + boric acid
- 4) pores 40-70Å (avg = 50Å)
- 5) void space = 28% of volume
- 6) surface area ~ 200m<sup>2</sup>/gr
- 7) absorbs water vapor up to 25% dry weight
- 8) can be taken to elevated temperatures (+700°C)  
must be cured first - see Corning Product  
Information Sheet

#### c. comments

- 1) clays deposited on it
- 2) no interference peaks
- 3) easily cleaned and re-cycled
- 4) somewhat fragile - handle carefully
- 5) will absorb organics from atmosphere which  
causes discoloration
- 6) store in deionized water to avoid contamination
- 7) activation  
before initial use must be activated -  
Corning supplies detailed instructions



d. cleaning

1. general

- a) soak in distilled water for several hours  
clay is softened--comes off with soft brush  
remove pencil label with cleanser
- b) E.G. removed by soaking with several  
changes of water
- c) use ultrasonic with care for short time  
only -- if temperature increases  $>20^{\circ}\text{C}$ ,  
fracturing can occur
- d) air dry on paper towels 1-2 hours
- e. put in drying cabinet at  $38^{\circ}\text{C}$  until dry
- f. dry when white opaqueness disappears

There is no quick way to remove water without  
damaging

2. organic

- a. use a strong oxidizer (30%  $\text{H}_2\text{O}_2$ , or  $\text{HNO}_3$ )  
plus a few crystals of potassium chlorate or  
sodium chlorate
- b. heat contaminated glass in this solution  
at  $100^{\circ}\text{C}$  until color disappears
- c. if  $\text{HNO}_3$  is used, wash several times in  
deionized water
- d. store in deionized water until activated

e. Reference

Corning Product Information Sheet  
Corning Glass Works  
Scientific Glassware Department  
Corning, New York 14839

5. Glass Filters

a. source

most filter manufacturers

Schleicher & Schuell  
Keene, New Hampshire 03431  
(603) 352-3810

b. format/physical characteristics

1. glass fibers
2. temperatures  $>500^{\circ}\text{C}$
3. highly resistant
4. depth type filter
5. 47mm discs

c. comments

1. give very little X-ray background
2. highly textured
3. can be used only once
4. thickness varies among types
5. more like filters than membranes  
use as pre-filter to membrane
6. fragile - seem to tear easily

B. QUARTZ

1. Fused Quartz Slides

a. source

Optical Instrument Laboratory  
P.O. Box 608  
Bellaire, Texas 77401  
(713) 772-7294

b. format/physical characteristics

- 1) 100% silica ...  
natural quartz is crushed and fused
- 2) will take temperatures to at least 1270°F
- 3) microscope type slides 1" x 1 1/2"  
can be fabricated to specifications

c. comments

- 1) amorphous - gives scatter hump at  $16 - 28^\circ 2\theta$   
(see Figure 3.
- 2) easily cleaned
- 3) very even, smooth surface
- 4) re-useable
- 5) moderately expensive
- 6) somewhat fragile

2. Quartz Filters

a. source

- 1) micro quartz fiber  
Gelman Sciences Inc.  
600 S Wagner  
Ann Arbor, Michigan, 48106  
(313) 665-0651
- 2) quartz micro fibre QM-A  
Whatman Inc.  
9 Bridewell Place  
Clifton, New Jersey 07014  
(201) 777-4825

- b. format/physical characteristics
  - 1) 25, 37, 47mm disks
  - 2) 8" x 10" sheets
  - 3) highly resistant
  - 4) silica fibers
  - 5) neutral pH
  - 6) tolerate temperatures to 500°C
- c. comments
  - 1) no interference peaks - scatter hump
  - 2) highly textured
  - 3) fiber - depth type filter

## C. CERAMIC TILES

### 1. Unglazed, Porous Tile

- a. source
  - Robertson Manufacturing Comapny
  - Tile Division
  - S. Pennsylvania avenue
  - Morrisville, Pa. 19067
  - (215) 295-1121
- b. format
  - 1) 6" x 6" squares
  - 2) can easily be cut to specifications
- c. comments
  - contains....quartz, feldspar, Ca/Mg-silicates

### 2. Bisque

- a. source
  - American Olean
  - 1000 Cannon drive
  - Lansdale, Pa. 19446-0271
  - (215) 855-1111
- b. format
  - 1) 6" x 6" squares
  - 2) can easily be cut to specifications
- c. comments
  - 1) contains...quartz, cristobalite, mullite, feldspar, corundum (tr)
  - 2) very crystalline - may prove to be difficult to mask with clay film

### 3. Porous Plates

#### a. source

University of Illinois  
Department of Ceramic Engineering  
105 S. Goodwin  
Urbana, Illinois 61801  
(217) 333-1771 George Conlee

#### b. format

4" x 4" x 1/8 - 3/16" thick

#### c. comments

- 1) reasonable
- 2) contains...quartz, feldspar, mullite
- 3) may be a little soft

### 4. Microporous Porcelain

#### a. source

Selas Corporation of America  
Flotronics Division  
1957 Pioneer Road  
Huntingdon Valley, Pa. 19006

#### b. format/physical characteristics

- 1) 1.5" diameter, 3mm thick
- 2) pores carefully controlled  
#02 grade 0.15u - 0.4u retention

#### c. comments

contains....mullite, quartz (tr), cristobalite (tr)

### 5. Porous Ceramic Membranes

#### a. source

SoilMoisture Equipment Corporation  
P.O. Box 30025  
Santa Barbara, California 93105  
(805) 964-3525

#### b. format/physical characteristics

- 1) 3" disk x 1/4" thick
- 2) can be fabricated to specifications
- 3) 5 bar = 0.576um pores
- 4) controlled pore size

#### c. comments

contains... quartz, cristobalite, mullite, feldspar,  
and ?

## 6. Ceramic Plates

### a. source

Coors Porcelain Company  
600 9 street  
Golden, Colorado 80401  
(303) 278-4000

### b. format

5cm x 2.5cm x 0.6cm thick  
probably fabricated to specifications

### c. comments

contains...corundum, cristobalite, mullite, quartz

## 7. Porous Plates

### a. source

Fisher Scientific Company  
711 Forbes Avenue  
Pittsburgh, Pa. 15219

consult catalog for ordering information

### b. format

6" x 6" - #CS 13-752

### c. comments

contains...corundum, cristobalite, quartz, mullite  
comes from Coors Porcelain Company

## 8. Ceramic Tile

### a. source

Monarch Tile Company  
P.O. Box 2041  
San Angelo, Texas  
(915) 655-9193

## **D. METALLIC SUBSTRATES**

### 1. Silver Membrane Filter

#### a. source

1) Selas Corporation of America  
Flotronics Division  
1957 Pioneer Road  
Huntingdon Valley, Pa. 19006  
(215) 672-0400

2) Millipore Laboratory Products  
80 Ashby Road  
Bedford, Ma. 01730  
(617) 275-9200

b. format/physical characteristics

- 1) Catalog # AG45 025 00
- 2) discs in several sizes  
47mm, 25mm, 37mm
- 2) pore sizes = 0.45um, 0.80um
- 3) thin - like a foil

c. comments

- 1) no interference peaks until  $\sim 38^\circ 2\theta$
- 2) expensive
- 3) brittle
- 4) re-useable
- 5) very versatile - adaptable to various sample holders

d. to clean

2. Sintered Metal

a. source

Mott Metallurgical Corporation  
Farmington Industrial Park  
Farmington, Conn. 06032  
(203) 677-7311

b. format/physical characteristics

- 1) porous stainless steel
- 2) available in sheets and discs
- 3)  $8\frac{1}{2}'' \times 10'' \times \frac{1}{16}''$  is suggested size
- 4) fabricate to desired size in a metal shop
- 5) 0.5um pore size
- 6) uniform permeability
- 7) maximum resistance to elevated temperatures and to corrosion

c. comments

- 1) can be machined to fit automatic sample changer
- 2) interference peaks (Fe) at 43.5, 50.75  $^\circ 2\theta$

d. to clean

- 1) after considerable use, may have to be re-surfaced by manufacturer
- 2) may warp and bend with use and have to be re-shaped

### **3. Stainless Steel Slides**

- a. source  
any common source for stainless steel
- b. format  
can be cut to any size specifications
- c. comments
  - 1) interference peaks do not come until  $\sim 43^\circ 2\theta$
  - 2) provide thermal stability
  - 3) may warp and bend, but can be re-shaped
  - 4) re-useable

## **E. CELLULOSE/ORGANIC FILTERS**

### **1. Mixed Esters of Cellulose - Membrane Filter**

- a. source  
Millipore Laboratory Products  
80 Ashby Road  
Bedford, Ma. 01730  
(617) 275-9200
- b. format/physical characteristics
  - 1) MF type Catalog # HAWP 047 00 for 0.45um/47mm
  - 2) biologically inert mixture of cellulose acetate and cellulose nitrate
  - 3) compatible with dilute acids and bases, aliphatic and aromatic hydrocarbons and non polar liquids  
(See Table I)
  - 4) tolerates temperatures  $< 75^\circ\text{C}$
  - 5) 70-80% void spaces
  - 6) pore sizes available from 8.0um - 0.25um
  - 7) diameters available from 13mm - 293mm
- c. comments
  - 1) gives only scatter hump with x-rays
  - 2) very resistant to glycerol

### **2. Nitrocellulose**

- a. source  
Schleicher and Schuell Inc.  
Keene, New Hampshire 03431  
(603) 352-3810

- b. format/physical characteristics
- 1) Catalog # BA 85 for .45um/47mm
  - 2) pore sizes = .45, .2, .15, .10, .05, .02um
  - 3) disks from 6-305 mm  
squares, sheets, rolls also available
  - 4) 60-80% pores by volume
  - 5) temperatures  
wet to 125°C  
dry to 80°C
  - 6) R.I. = 1.5  
becomes transparent in immersion oil
  - 7) ignites at ~170°C in open flame
  - 8) trace element content  
Na>Ca>K>Mg>Si>Al>Cu>Zn>Fe>Pb>Sr>Mn>Ni

- c. comments  
gives scatter hump with x-rays See Figure 3

### 3. Mixed Esters of Cellulose

- a. source  
Gelman Sciences Inc.  
600 S. Wagner  
Ann Arbor, Michigan 48106  
(313) 665-0651
- b. format/physical characteristics
- 1) Metrical Catalog # GN-6 for .45um/47mm
  - 2) mixed esters of cellulose
  - 3) pore sizes = 0.45um, 0.8um
  - 4) disks in several sizes - 13, 25, 37, 47mm
  - 5) temperature to 74°C
  - 6) ~80% void space

- c. comments  
scatter hump with x-rays

### 4. Porvic

- a. source  
Pritchett and Gold and E.P.S. Co. Ltd.  
137 Victoria St  
London, S.W.I. United Kingdom
- b. format/physical characteristics
- 1) Grade "M"
  - 2) 12" x 12" x 0.03" thick sheets
  - 3) polyvinyl chloride
  - 4) controlled pore size = 5um
  - 5) retains 2um size particles



c. comments

- 1) little scatter with x-rays
- 2) swells with glycerol
- 3) curls slightly upon drying
- 4) cannot be heated

**5. Mixed Esters of Cellulose**

a. source

Nuclepore Corporation  
7035 Commerce Circle  
Pleasanton, California 94566  
(415) 462-2230

b. format/physical characteristics

- 1) Membra-Fil
- 2) mixed esters of cellulose acetate and nitrocellulose
- 3) disk sizes = 37,47mm
- 4) pore sizes = .22, .45, .7, .8, 1.2um
- 5) temperatures  
    wet to 125°C  
    dry to 75°C

**6. Polycarbonate Surface Filter**

a. source

Nuclepore Corporation  
7035 Commerce Circle  
Pleasanton, California 94566  
(415) 462-2230

b. format/physical characteristics

- 1)
- 2) pore sizes = .4um
- 3) disk sizes = 47mm
- 4) very smooth surface

**C. METHODS USED TO ACHIEVE ORIENTED MOUNTS**

# ORIENTED X-RAY DIFFRACTION MOUNTS---METHODS

## I N T R O D U C T I O N

Oriented XRD mounts can be achieved by a great variety of techniques. It is the objective of Section III to abstract the majority of the methods found in the literature, outlining substrate material, apparatus, procedures and references. The articles have only been abstracted. The abstractors have added nothing to the original article information. Therefore any incompleteness of information is a function of the source. Since each researcher has a potentially unique application, this section offers a choice of all techniques so that the user may discriminate for himself.

### A. Sedimentation Onto Substrate

Methods using gravity to sediment or settle the clay material onto a substrate usually employ ordinary glass such as a microscope slide for the substrate. Porous glass plates or disks, high temperature glass slides, fused silica (fused quartz) or metal slides (stainless steel) can also be used.

No special apparatus is involved. A beaker, pipette and the substrate are the items most commonly used.

Two major procedures are followed. The <2um fraction of the suspension is pipetted onto a slide and air dried. A slide is placed at the bottom of a beaker and the clay is allowed to settle onto it.

### References

1. Burtner, 1974
2. Gipson, 1966
3. Hathaway, 1956
4. Jackson, 1956
5. Kinter and Diamond, 1956
6. Kittrick, 1961
7. Mitchell, 1953
8. Mossman and others, 1967
9. Oinuma and others, 1961
10. Rich, 1967
11. Schoen, 1964

### B. Centrifuge Onto Substrate

By definition, the substrate in this process would have to be non-porous. It is usually limited to a glass slide.

The apparatus used is a large centrifuge. The procedure requires that the slides be placed at the bottom of large centrifuge tubes filled with suspension. Centrifugal force, rather than gravity, is used to achieve the sedimentation of the clay onto the substrate.

#### References

1. Brown, 1953a, b
2. Dana, 1943
3. Devine and others, 1972
4. Spoljaric, 1971

#### C. Paste/Smear

Glass slides and other firm substrates such as ceramic tile and metal slides are used in this method.

As a rule no special apparatus is utilized. However, Tien, (1974) has developed a customized frame to hold the slides and control the thickness of the sample.

The procedure is very simple. The prepared clay paste is smeared on the glass slide or chosen substrate with a spatula.

#### References

1. Barshad, 1960
2. Theisen and Harward, 1962
3. Tien, 1974

#### D. Centrifuge Through Substrate

Porous ceramic tile, porous sintered glass or stainless steel are the substrates used for this method.

Besides a large centrifuge, the apparatus required is a special substrate holder which must be fabricated to fit into the trunnion cups (Kinter and Diamond, 1956).

The procedure involves fitting a substrate into the holder and pouring in the suspension. Centrifugal force drives the water from the suspension through the porous medium depositing the clay film onto the substrate.

#### References

1. Brown, 1953
2. Dana, 1943
3. Kinter and Diamond, 1956 (tile)
4. Kittrick, 1961 (tile, porous stainless steel)

## **E. Suction Onto Substrate**

This method utilizes a wide variety of substrates, all porous. Ceramic plates (tiles), various types of membrane filters (metallic, cellulose), glass and quartz fiber filters, sintered glass disks and porous stainless steel have been cited.

All techniques require a vacuum pump or a water aspirator and an apparatus to support the porous substrate. The apparatus vary from commercial membrane filter set-ups to custom built devices which hold porous ceramic, sintered glass or porous stainless steel plates. Those methods using flexible membrane filters also require a special holder to keep the membrane flat during XRD analysis.

The procedure involves pipetting a suspension into a reservoir over the porous substrate. Vacuum is applied and the suspension water is drawn through the substrate depositing the clay film on the surface of the substrate. Various treatments can also be performed while the substrate resides in the holder

### **References**

1. Brown, 1953 (glass)
2. Carlton, 1975 (tile)
3. Pevear, unpublished (tile)
4. Poppe and Hathaway, 1978 (silver membrane)
5. Quakernaat, 1970 (membranes)
6. Rich, 1969 (tile)
7. Shaw, 1972 (tile)
8. Starkey and others, 1983 (tile)
9. Tucholke, 1974 (silver membrane)

## **F. Suction and Transfer**

The only cited substrate is a membrane filter. These are usually cellulose.

The apparatus and procedure are very similar to those used in the preceding description. After the suspension water is suctioned through a membrane filter, the resulting clay film is transferred to a firm substrate, typically a glass slide.

### **References**

1. Brusewitz, 1982
2. Drever, 1973
3. U.S. Geological Survey, 1982

## **G. Pressure**

Substrates that can be used in this method are made of porous ceramic, glass or metal.

A custom made cell is the required apparatus, and holds the substrate. The suspension is placed in the cell and the water is forced through the substrate by pressure from above. The method is similar to that described under suction, but pressure is used instead as the moving force.

#### References

1. Bajwa and Jenkins, 1978

#### H. Pressed Disks

There is no substrate with this method.

The main apparatus is a hydraulic press. The powdered clay, usually mixed with a binder, is formed into a disk in a hydraulic press using a steel die. A special holder is required to position the disk in the diffractometer.

#### References

1. Cody and Thompson, 1976
2. Fenner and Hartung, 1969
3. Mitchell, 1953

#### I. Powder Camera

Technically there is no substrate involved in this method. However, the sample can be encased within a capillary tube of glass, plastic or cellulose.

Various procedures such as extrusion, rolling between the fingers and coating of fibers produce preferred orientation of clay specimens for the Debye Scherer camera.

#### References

1. Barshad, 1955 (capillaries)
2. Barshad, 1954 (capillaries)
3. Bradley and others, 1937 (capillaries)
4. Clark and others, 1937 (capillaries)
5. Cole, 1961 (instrument modification)
6. Neumann, 1956 (capillaries)
7. Owen, 1971 (collodion fibers)

**D. ADVANTAGES AND DISADVANTAGES OF THE METHODS**

**METHOD: SEDIMENTATION (Gravity or Centrifuge)**

**MEDIUM**

**ADVANTAGES**

1. easy and fairly quick to do
2. cheap and requires no special equipment
3. produces fairly good orientation
4. good for small samples

**Glass Slides**  
(non-porous)

**DISADVANTAGES**

1. size fractionation/mineral segregation or differential layering
2. can crack and warp with heat if ordinary glass
3. hard to get thick enough sample sometime
4. may get scatter from glass
5. dilute or thin suspension hard to use  
-as multiple applications may spall
6. does not hold glycol solvation as well as tiles
7. takes longer to dry which can cause segregation
8. smectites may crack, peel, or curl
9. chemical treatments not as easy to perform as with tiles
10. thickness variations over slide
11. peptizing agents or other residual salt from treatments will crystallize in dry clay - disturbs orientation and may produce interference peaks

- Glass Slides**
1. can be used in several methods
  2. retain glycol longer than non-porous substrate
  3. thermal stability

1. expensive
2. hard to maintain

TABLE III.--Advantages and Disadvantages of Sedimentation Methods

Advantages/Disadvantages



**METHOD: SMEAR/PASTE**

**MEDIUM**

**ADVANTAGES**

- Class Slides**
1. quick and easy method
  2. requires very little equipment
  3. easy method to teach and learn
  4. good orientation
  5. easy to get infinite thickness

**DISADVANTAGES**

1. segregation/layering if paste too thin
2. spalling, peeling, cracking, if too thick
3. thickness variations
4. thermal instabilities
5. uneven surfaces - causes inconsistent intensities and poor peak to background ratios
6. scatter from glass slide if clay film too thin
7. surface may deteriorate with storage
8. requires large sample

**Ceramic Plates**

1. heat resistance
2. retains solvating agent
3. absorbs excess water during smearing application
4. clay adheres to better than to glass
5. easy to achieve infinite thickness

1. interference peaks from tile if clay film too thin

hard to make paste with dilute suspension

TABLE IV.--Advantages and Disadvantages of Smear/Paste Method

## METHOD: SUCTION

### MEDIUM

### ADVANTAGES

- General**
1. no differential settling if filtration is rapid
  2. cation saturation, solvation, and washing are quick and easily done
  3. all suction methods give good orientation

### DISADVANTAGES

1. requires vacuum source
2. some methods take a long time
3. can lose sample if apparatus fails from leakage or breakage

### Tiles

1. Inexpensive medium
2. thermal stability and no warping
3. can withstand heat  $>550^{\circ}\text{C}$
4. holds glycol longer than non-porous glass slides
5. greater mechanical stability
6. re-useable
7. easy to clean
8. less spalling than glass
9. even sample surface for diffraction

1. If clay film is thin, may see minerals in tiles-interference
2. porosity and permeability can vary which may result in segregation if suction is too slow
3. can get differential thickness

### Sintered Metal

1. no interference peak until  $\sim 50^{\circ} 2\theta$
2. thermal stability
3. can be fabricated to fit automated XRD instrument

1. expensive
2. may rust or warp
3. may require special fabrication and reclamation techniques to preserve porosity

**METHOD: SUCTION**

**MEDIUM**

**ADVANTAGES**

- Membranes---**
1. relatively cheap - re-useable
- Cellulose**
2. excellent orientation
  3. quick method
  4. usually so fast there is not any segregation
  5. with masks or 25mm filters can process very small samples
  6. excellent, even surface
  7. properly transferred sample forms continuous layer preventing scatter from glass slide
  8. since very thin film can be applied, smectites rarely peel, crack or curl
  9. no interference peaks from cellulose filter  
if sample is not transferred to glass slide
  10. can easily be transferred to glass slide

**DISADVANTAGES**

1. cannot be heated (if clay is not transferred)
2. dissolved by certain solvents
3. if not transferred from membrane, mounting of membrane in sample holder can be difficult
4. equipment is somewhat specialized and requires vacuum
5. 25mm diameter samples may give distortion in 2  $\theta$  positions depending on machine parameters

**Membrane--  
Metallic  
(silver)**

1. re-useable
2. no interference until high 2  $\theta$  positions
3. thermal stability
4. can be fabricated to shape of sample holder

1. expensive

TABLE V.--Advantages and Disadvantages of Suction Methods

## VI. R A N D O M X R D M O U N T S

Randomly oriented sample mounts are used in clay mineralogy for whole rock scans and for XRD of the clay fraction to determine the position of the 060 reflection. Since random mounts have been discussed in detail in another part of this Workshop, they will not be dealt with here.

A detailed bibliography is included at the end of this Syllabus for the information of the reader.

## VII. X-R A Y D I F F R A C T I O N P A T T E R N S

### A. STANDARD IDENTIFICATION SCANS (done on oriented mounts)

1. Air dried - untreated
2. Ethylene glycol solvated  
vapor - 60 °C - 4 hours
3. 400°C  
heated for 1/2 hour
4. 550°C  
heated for 1/2 hour
5. When peak positions from the above four scans compared against those listed on the Flow Chart, most common clay minerals should be easily identified.

### B. CREATING THE X-RAY DIFFRACTION TRACE

1. A convenient method of setting up a set of clay scans is to stack them on the same trace - one above the other.
2. Use different colored inks for each treatment, but be consistent with ink color and treatment and sequence of runs.

## VIII. CLAY MINERAL IDENTIFICATION USING FLOW CHART

This Flow Chart summarizes the previously discussed treatments and the reactions of the clay minerals to these treatments. Used properly, it will aid in the identification of most clay minerals.

## **IX. BIBLIOGRAPHIES**

Detailed bibliographies dealing with the various topics presented in this Syllabus follow.

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## Solvation

O R I E N T E D   M O U N T S   R E F E R E N C E S

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