BETTER LIVING THROUGH MINERALS
X-RAY DIFFRACTION OF HOUSEHOLD PRODUCTS

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(*with apologies and thanks to Mike Holdaway*)

As you have learned in lecture, X-ray diffraction is a quick and valuable tool for identifying minerals. Minerals are an integral portion of our everyday life, in addition to composing our planet! They help bring electricity into our homes and remove our bathtub rings.

In this lab you will analyze the X-ray diffraction patterns of three household cleansers, *Ajax*, *White Magic*, and *Soft Scrub*, in order to identify the abrasive minerals in each. Haven't you wondered what puts the "soft" in *Soft Scrub*?

The diffraction patterns for the three samples are photocopied for you as the last three pages of this lab. These samples were run without using an internal standard so any peak shifts remain uncorrected relative to their ideal positions. This, combined with errors in hand measurement of peak positions, produce errors in your calculated d-values. Consequently, 2θ angles which have been corrected using a corundum standard are also provided.

ASSIGNMENT

1. Measure the 2θ angles for the peaks of the abrasive minerals from the diffraction patterns for each of the three cleansers. For each pattern, list the angles in order of decreasing intensity in the space provided.

2. Compare your measured 2θ angles to those values listed in the second column. These 2θ angles have been corrected using a corundum standard.

3. Calculate the corresponding d-values for each peak using the Bragg equation:

$$n\lambda = 2d \sin \theta$$
$$\lambda = 1.5405 \text{ Å} \ (\text{Cu } K_\alpha \text{ X-ray wavelength})$$

4. Match the sets of d-values to those in the table of d-values for common, light-colored minerals (found on the last page) to identify the abrasive mineral in each sample.

5. Answer the questions at the end of the lab.

Sample 1: **White Magic** Mineral: ____________________

2θ measured	2θ ideal	d-values measured	d-values ideal
....	31.20	....	....
....	51.50	....	....
....	50.95	....	....
....	41.56	....	....
....	45.21	....	....

Sample 2: **Ajax** Mineral: ____________________

2θ measured	2θ ideal	d-values measured	d-values ideal
....	26.71	....	....
....	21.03	....	....
....	36.50	....	....
....	50.18	....	....
....	40.35	....	....
....	60.04	....	....

Sample 3: **Soft Scrub** Mineral: ____________________

2θ measured	2θ ideal	d-values measured	d-values ideal
....	29.80	....	....
....	47.79	....	....
....	48.80	....	....
....	39.65	....	....
....	43.50	....	....
....	57.75	....	....
....	23.30	....	....

QUESTIONS

1. List the cleansers in order of **increasing abrasiveness.**

    1.

    2.

    3.

2. Which cleanser will get rid of your bathtub ring most easily?

3. Which cleanser(s) will be the least likely to scratch your bathroom fixtures or your marble sink?

4. What are some possible sources of error between the measured and ideal peak positions?

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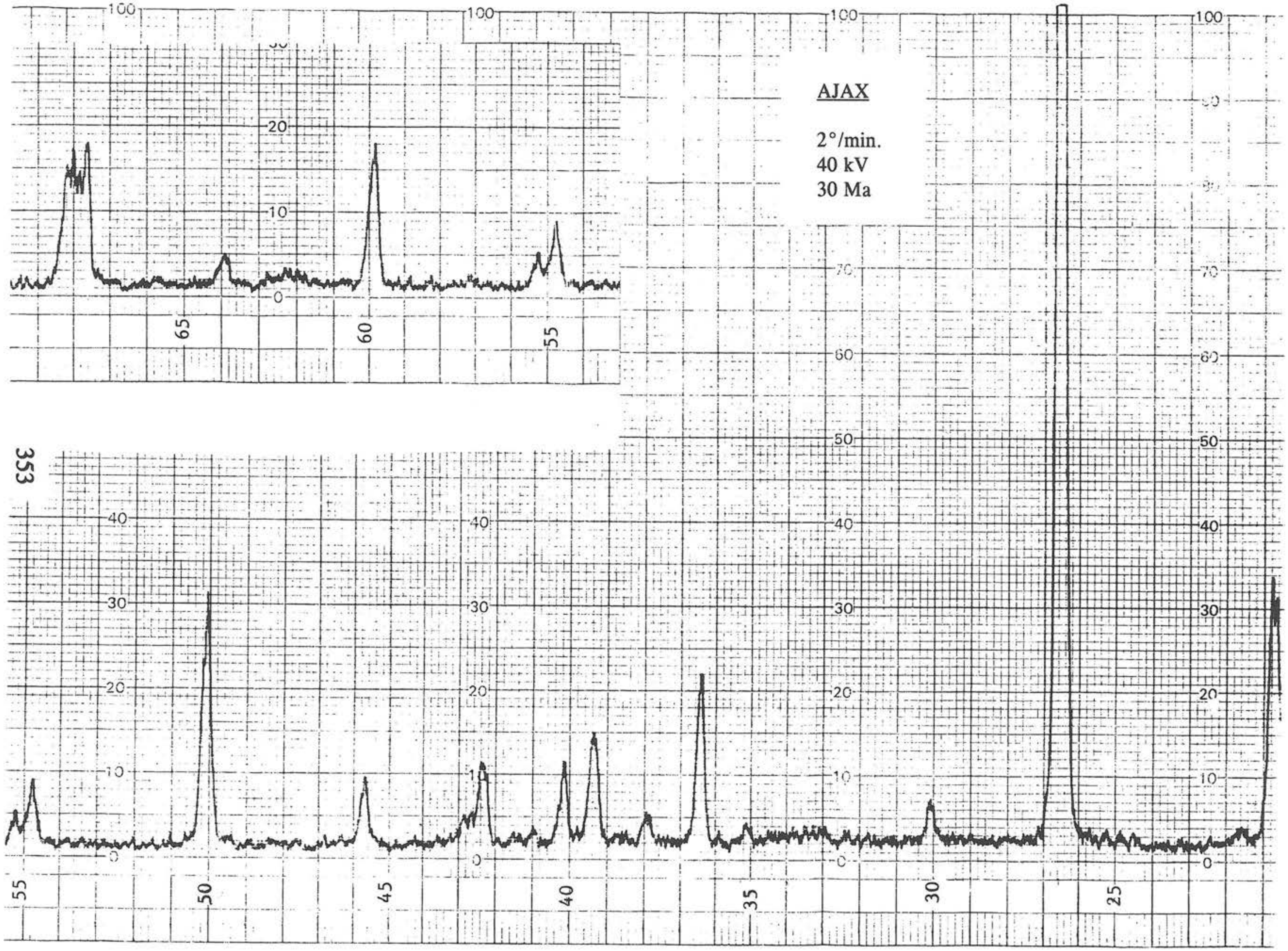
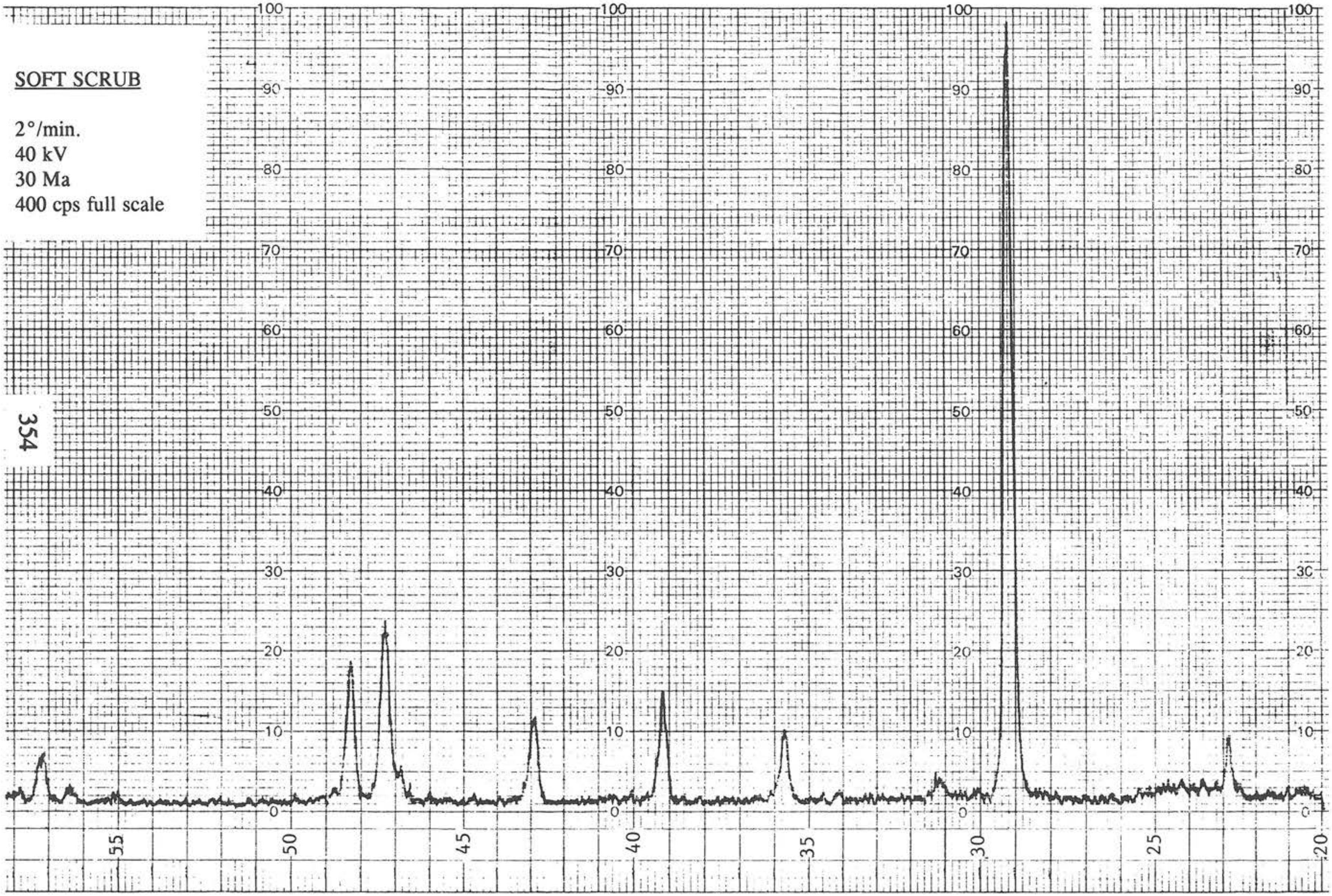
d-VALUES AND HARDNESS OF COMMON MINERALS

The last digit in () refers to the ideal intensity of the peak relative to the intensity of the most intense peak for that mineral. x = 100 (most intense), 9 = 90 %, 8 = 80 %, etc. H = hardness on Moh's hardness scale.

Mineral	H	d-values							
Anhydrite	3.5	3.50(x)	2.85(4)	2.33(2)	2.21(2)	1.87(2)	1.65(1)	1.75(1)	2.09(1)
Apatite	5.0	2.81(x)	2.78(6)	2.72(6)	3.44(4)	1.84(4)	1.94(3)	2.63(3)	2.26(2)
Aragonite	3.5	3.40(x)	1.98(7)	3.27(5)	2.70(5)	2.37(4)	2.48(3)	1.88(3)	2.34(3)
Barite	3.5	3.45(x)	3.10(x)	2.12(8)	2.11(8)	3.32(7)	3.90(5)	2.84(5)	2.73(5)
Calcite	3.0	3.04(x)	2.29(2)	2.10(2)	1.91(2)	1.88(2)	2.50(1)	3.86(1)	1.60(1)
Corundum	9.0	2.09(x)	2.55(x)	1.60(8)	3.48(7)	1.37(4)	1.74(4)	2.38(4)	1.40(3)
Dolomite	3.5	2.89(x)	1.79(3)	2.19(3)	1.78(3)	1.80(2)	2.02(2)	1.39(2)	2.67(1)
Fluorite	4.0	1.93(x)	3.15(9)	1.65(4)	1.12(2)	1.37(1)	1.25(1)	0.86(1)	1.05(1)
Gypsum	2.0	7.56(x)	3.06(6)	4.27(5)	2.68(3)	2.87(3)	3.79(2)	1.90(2)	2.08(1)
Halite	2.5	2.82(x)	1.99(6)	1.63(2)	3.26(1)	1.26(1)	1.15(1)	1.41(1)	0.89(1)
Muscovite	2.0	10.1(x)	3.36(x)	4.49(9)	2.57(9)	3.66(6)	3.07(5)	2.58(5)	5.04(4)
Quartz	7.0	3.34(x)	4.26(4)	1.82(2)	1.54(2)	2.46(1)	2.28(1)	1.38(1)	2.13(1)
Talc	1.0	9.35(x)	1.53(6)	4.59(5)	3.12(4)	2.48(3)	4.56(3)	2.60(2)	2.50(2)
Topaz	8.0	2.94(x)	3.20(7)	3.69(6)	2.36(5)	2.11(4)	3.04(4)	1.67(3)	2.38(3)
Wollastonite	5.0	2.97(x)	3.83(8)	3.52(8)	3.31(8)	2.47(6)	2.18(6)	1.83(6)	1.72(6)


WHITE MAGIC
2°/min.
40 kV
30 Ma
400 cps full scale
100
90
80
70
60
50
40
30
20
10
0
65
60
55
50
45
40
35
30


AJAX
2°/min.
40 kV
30 Ma
353



SOFT SCRUB
2°/min.
40 kV
30 Ma
400 cps full scale
100
90
80
70
60
50
40
30
20
10
0
55
50
45
40
35
30
25
20

354

INFORMATION FOR INSTRUCTORS

Purpose

The purpose of this lab is twofold: (1) to demonstrate the ease, utility, and applicability (including uncertainty) of X-ray diffraction (XRD) to mineral identification, and (2) to establish that minerals are a critical, if largely unrecognized, portion of our everyday life.

Previous knowledge required

Typically this lab is to be given as a supplement to a lecture on XRD. This lab assumes that the students have knowledge of the principles and theory behind XRD and other background information that you feel is necessary for an appropriate level of understanding (i.e. when coherent interference occurs and peaks are recorded, etc.).

Lab Materials

XRD patterns have been photocopied for the lab and are part of the lab handout. These patterns were run on an old Phillips diffractometer. Peaks from only one mineral are identifiable in each pattern. Additional materials needed for the lab include a ruler, a calculator and a pencil (I never allow students to use pen in lab).

Introduction to Lab

A short lecture precedes this lab which incorporates:

- sample preparation, random vs. preferred orientation, changes to diffraction patterns caused by various preparation methods, do's and don'ts of sample prep, etc.
- techniques for measuring peaks by hand and expected uncertainty (e.g. measure at 80 % peak height, record the peaks in order of decreasing intensity; 2θ angles can be off by ca. 0.5° leading to calculated d-values being off by ca. 0.02 Å). Also, remind students of significant figures.
- techniques for searching JCPDS data by hand (e.g. numbers at top of page are for most intense peak but listed in order of second most intense peak, or by computer if you have a CD based catalog).
- permitted difference between measured peaks and those listed (e.g. without internal standards 2θ may be off by 0.5°).
- reasons that the measured peaks may have slightly different d-values from those listed and may have different relative intensities (e.g. machine error, sample height, impurities, compositional variations, order-disorder, grain size, strain**).

**If measured peaks are not corrected using an internal standard or if minerals have compositional variations, etc. that cause the d-values to differ from those listed in the JCPDS files, the students have a difficult time matching d-values to minerals and the lab becomes frustrating. In addition, the relative intensity of the peaks is commonly different than the ideal (especially for lower intensity peaks). Hence, I provide them with the "corrected" d-values, arranged according to the ideal intensity, to aid in identifying the minerals and alleviate frustration.

Options for Customizing the Lab

This lab is extremely versatile and can be easily customized to your laboratory and facilities (or lack thereof) as well as for the time constraints. Below are several methods for presenting the lab, based on equipment and difficulty level.

- Samples:

  If XRD equipment and preparation facilities are available, students can prepare their own XRD samples. Then students can run the samples or a technician can run them. If an X-ray diffractometer is unavailable, the attached patterns can be duplicated.

- Peak Identification

  Hand or computer methods can be used for peak identification, again, dependent on equipment.

  1. Computerized peak search and mineral identification
     If the materials have been run on your XRD, peaks will be identified and d-values calculated. These d-values can then be entered into a computerized search routine to identify minerals. Computerized search routines are usually available with the machine software. Other computerized search routines can be used, if a machine is not available.

  2. Hand measurement
     Students can measure peaks by hand and calculate d-values. For mineral identification, they can match their calculated d-values by one of the following:

     (a) have a JCPDS book (or cards) available for matching d-values.
     (b) photocopy appropriate pages from JCPDS for students to search.
     (c) use selected information from JCPDS (on the back lab sheet) to identify peaks.
     (d) use computerized search routines.

  3. Provide students with corrected d-values
     Use the d-values provided (in case the peak positions have been displaced and are not corrected). This provides the quickest and least frustrating mineral identification.

- Answers:

  Sample 1. White Magic - Dolomite
  Sample 2. Ajax - Quartz
  Sample 3. Soft Scrub - Calcite

- Other Patterns provided:

  An additional XRD pattern is given (Kitty Max: montmorillonite, cristobalite?, illite, kaolinite) as supplemental material at the end of this lab. One copy is blank (for student use) and one has the d-values and mineral ID labelled (for instructor use). This pattern is more challenging which allows you to increase the difficulty of this lab. Minerals were identified with an automatic search routine, (a disclaimer?).

Additional materials and ideas

- These household cleaners are but a few of the household products available for this lab exercise. The supermarket offers any number of other interesting household products for your XRD enjoyment. Reading labels is fun and enlightening.

- As an additional assignment, *have the students bring their own favorite household products to X-ray*. They can peruse the grocery store and cosmetic counters to find any assortment of interesting mineral-containing items. For instance, Crest has TiO_2 but in sufficiently low quantities that we could not see the peaks.

- Additional suggestions (collected from workshop participants) include:

  - Dr. Scholl's products (e.g. kaolin, talc)
  - paint, sunscreens (e.g. rutile or brookite)
  - various cosmetics (e.g. talc, face powder, eye shadow)
  - other cleansers (e.g. Bon Ami with calcite and feldspar, Lava soap with glass)
  - toothpaste (e.g. Crest with sparkles of mica; Tom's of Maine with calcite and fluorite)
  - medicinal products (e.g. Kaopectate with attapulgite, Multivitamins with periclase, calcite)
  - office products (e.g. White Out)
  - food items (e.g. "Chocolate bar" with kaolinite; MacDonald's milkshake (after diluting, washing with detergent, and collecting sludge)-gypsum (or kaolinite?)--two people gave different answers!

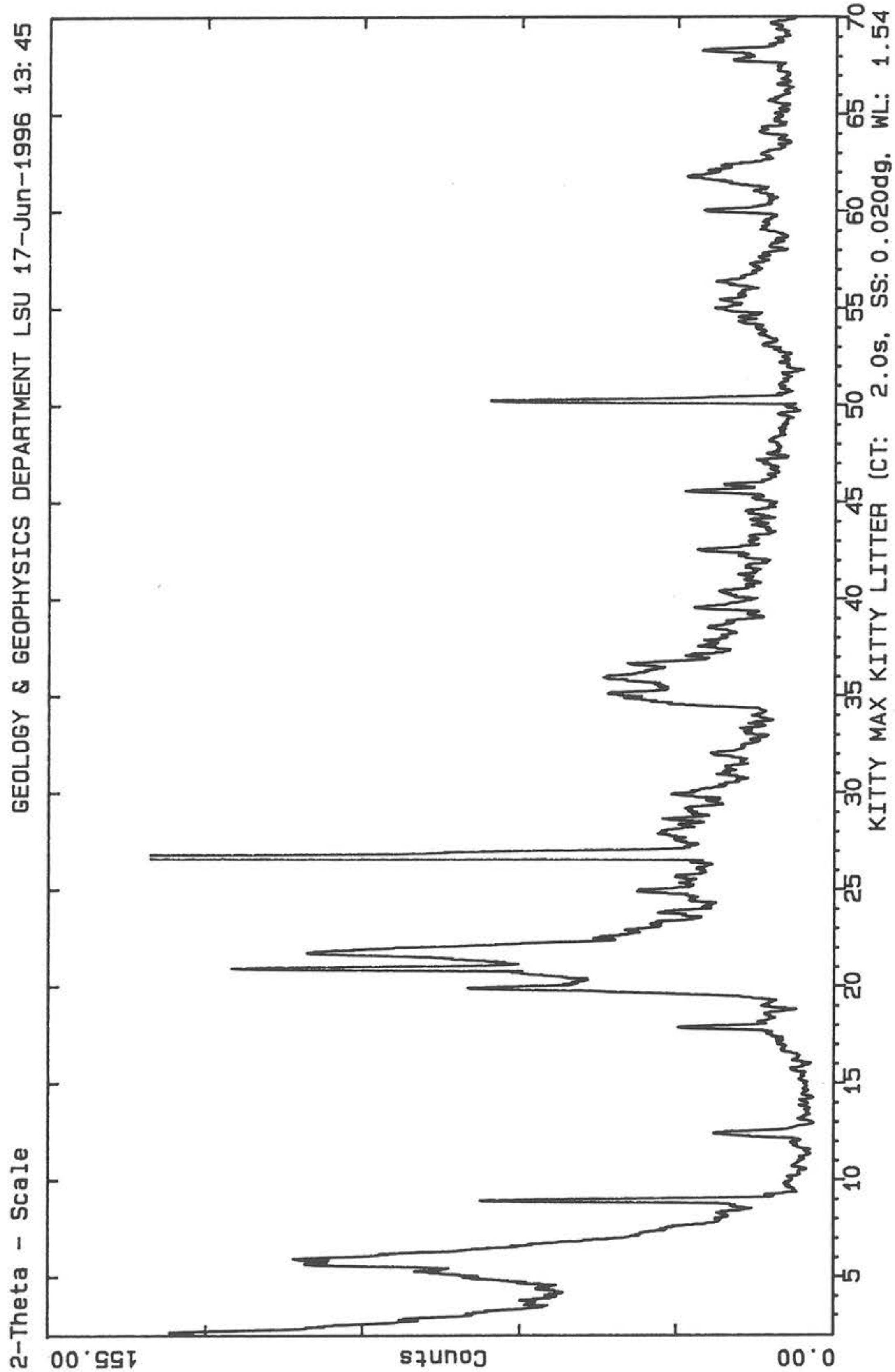
- Other "household products" that we ran also contain minerals but have messier/more complicated patterns. Minerals were identified with an automatic search routine. For the more challenging mineral identification:

  - Top Crest Cleanser: quartz, calcite, natrite, dolomite
  - Tidy Cat: illite, montmorillonite, kaolinite, cristobalite?
  - Tums: calcite, natron, talc, but the dominant phase is sucrose!
  - Baking Soda: Nahcolite
  - Sure deodorant: talc, albite (dried overnight in oven); talc (wet)
  - 1996 Ajax: Calcite, dolomite, quartz, natrite (An interesting note, Ajax apparently changed composition in the mid 90's from quartz dominant to calcite. Perhaps it was too abrasive?)

And for more straightforward mineral identification:

- Table salt and salt substitute

These are very simple, clean patterns. In addition to mineral identification of halite and sylvite, these can also be used to observe the size difference of K and Na (i.e. KCl and NaCl).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Counts
0.00
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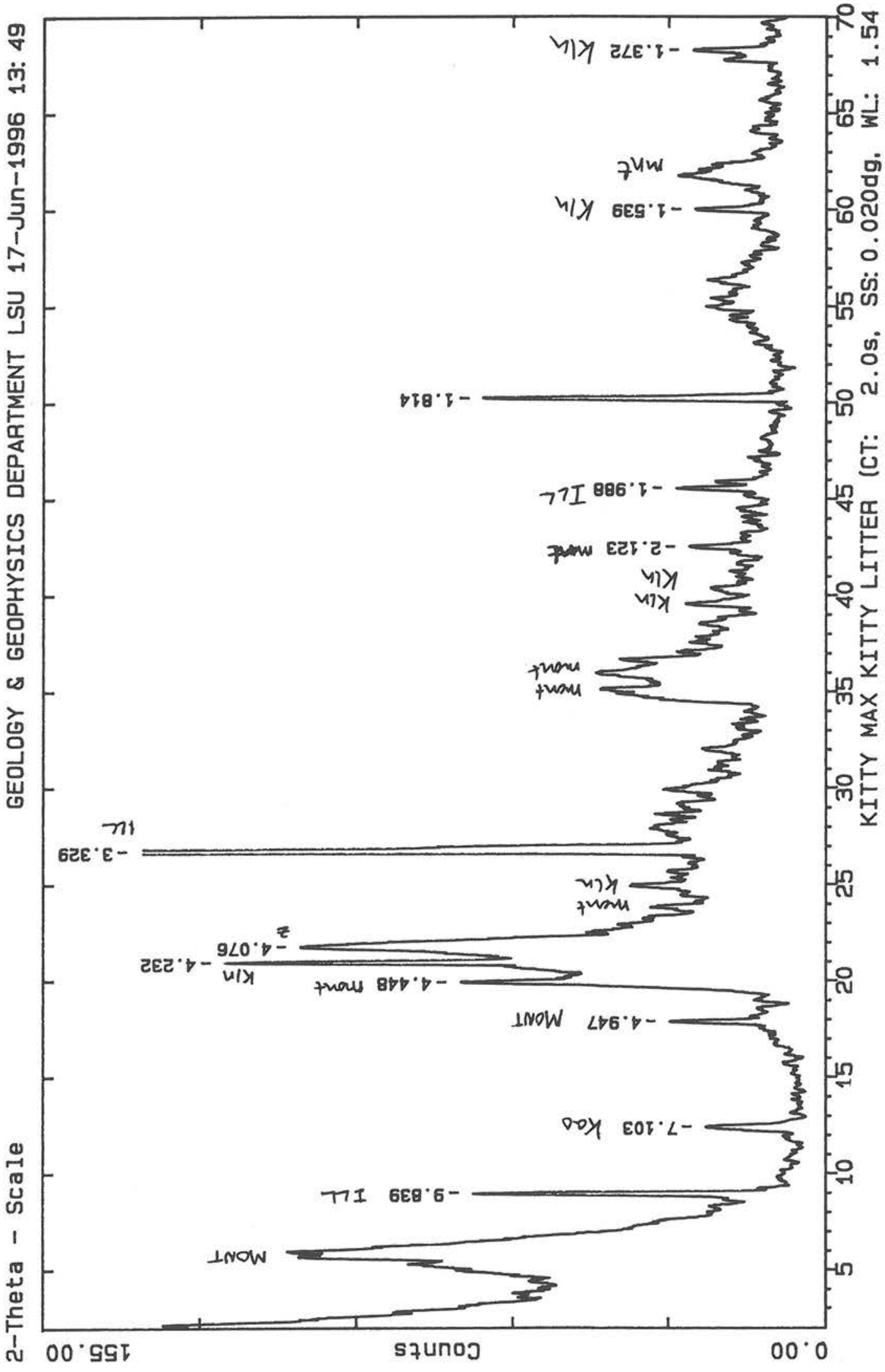
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MONT
-9.839 ILL
-7.103 Kao
-4.947 MONT
-4.448 mont
-4.232
Kln
-4.076
-3.329
ILL
mont
Kln
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mont
Kln
Kln
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mnt
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