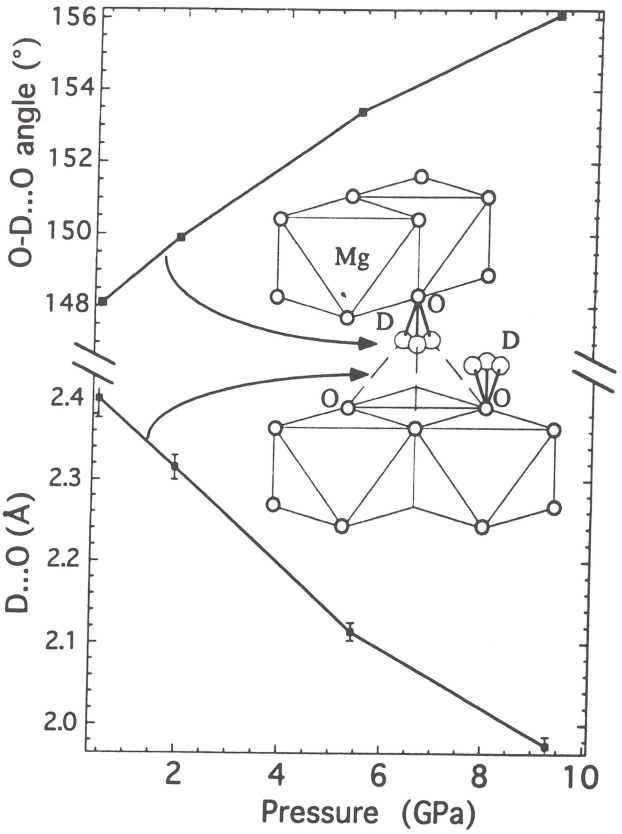

Fig. 1. Variation in the O-D···O angle and the interlayer O···D distance as a function of pressure found in this study. A polyhedral representation of the structure of deuterated brucite, $Mg(OD)_2$, is shown. The polyhedra are Mg-centered, and the D sites (shown as circles) are ⅓ occupied and at the 6*i* position.

D) in welded Au capsules held at 200 MPa and 600 °C for 1 week in cold-seal vessels. The product was confirmed as brucite by X-ray powder diffraction. In addition, the material was found to be well crystallized. In contrast, a second sample of deuterated brucite synthesized under autogenous conditions (200 °C) showed significant peak broadening, with peaks about three times as broad as those of the high-temperature sample. The high-temperature sample was used for neutron powder diffraction study.

A 152-mg sample of $MG(OD)_2$ was mixed with 27 mg of NaCl, which was used as an internal pressure calibrant. This was slurried with fluorinert FC-70 (3M company) and loaded into a Paris-Edinburgh high-pressure cell with tungsten carbide anvils (Nelmes et al., 1993) having a 100-mm^3 spherical sample volume. Metal gaskets (Ti-Zr) were used to contain the sample, and oil pressure in the ram was raised using a hand pump. The data were taken at 300 K on the high intensity neutron powder diffractometer (HIPD) at the Manuel Lujan, Jr., Neutron Scattering Center (LANSCE) at the Los Alamos National Laboratory. The HIPD has a flight path of 9.0 m and a resolution $\Delta d/d$ of 5–7 × 10^{-3}. Data were collected in two detector banks, centered at ±90° 2q, for about 12 h at average proton currents of 65 μA on the spallation source. Portions of the diffraction data collected in the 90° data banks are shown in Figure 2.[1]

The refinement was initiated using published data (Hyde and Andersson, 1989), with Mg at (0,0,0), O and D at (⅓,⅔,*z*). Several cycles of least squares were used to adjust the unit cell, background, and peak width parameters (Larson and Von Dreele, 1986) before adjusting the structure model. For pressures up to 1.9 GPa there was no broadening greater than instrumental and no evidence of the particle-size broadening observed in a previous study of deuterated brucite (Partin et al., 1994). However, at 5.4 and 9.3 GPa peaks were broadened by factors of 2 and 5, respectively. This was presumed to be due to deviatoric stress and sample heterogeneities (Weidner et al., 1994). The values of the pressure-induced isotropic microstrain, as estimated from the σ1 profile coefficient (Larson and Von Dreele, 1986), are 0.32, 0.62, and 1.12% for the data sets at 1.9, 5.4, and 9.2 GPa, respectively.

Three models for the structure were considered. Initially a model assuming isotropic thermal motion and all atoms on either the 3 or $\bar{3}$ axes was refined. A difference-Fourier map calculated from the data at 5.4 GPa clearly showed positive residual scattering around the site occupied by D. Further, models in which an anisotropic thermal parameter was refined for D resulted in the ratio U_{11}/U_{33}, increasing from 3 at 0.4 GPa to 10 at 5.4 GPa. The former ratio is in excellent agreement with that found in an earlier study by Zigan and Rothbauer (1967). Improvement to the overall fit of the isotropic model to observed data was obtained when D was moved from side 2*d* at (⅓,⅔,*z*) to 6*i* at (*x*,2*x*,*z*), with occupation factor ⅓. For example, the values for χ^2 for the 5.4 and 9.3 GPa data improved from 1.92 and 2.25 to 1.75 and 1.92, respectively. The values for R_{Bragg} for the 5.4 and 9.3 GPa data improved from 10 and 12% to 8 and 9%, respectively. The split-atom model was used for the analysis of the four data sets. In the final stages of the refinements, data from both the +90 and −90° banks were included. Refined parameters for the split-atom model are given in Table 1. The pressures were determined using the Decker (1971) equation of state for NaCl and the refined unit-cell parameters.

Following submission of this work, a redetermination of the room-pressure structure of portlandite, $Ca(OH)_2$, which is isostructural with brucite, was published (Desgranges et al., 1993). This determination, like that of brucite before it (Zigan and Rothbauer, 1967), noted the unusually large U_{11}/U_{33} ratio. A split-site model proposed by Desgranges et al. (1993) for the H position is similar to that proposed by ourselves for the D position in brucite at high pressure.

[1] A copy of Fig. 2 may be obtained by ordering Document AM-94-549 from the Business Office, Mineralogical Society of America, 1130 Seventeenth Street NW, Suite 330, Washington, DC 20036, U.S.A. Please remit $5.00 in advance for the microfiche.

From: john Parise <jparise@notes.cc.sunysb.edu>
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Date: Monday, July 21, 2003 12:37 PM

Figure 2 from Am Mineral 79, 193 - 196 (1994)

Individual banks + composite as JPEGS

AM-94-549 could not find!

john

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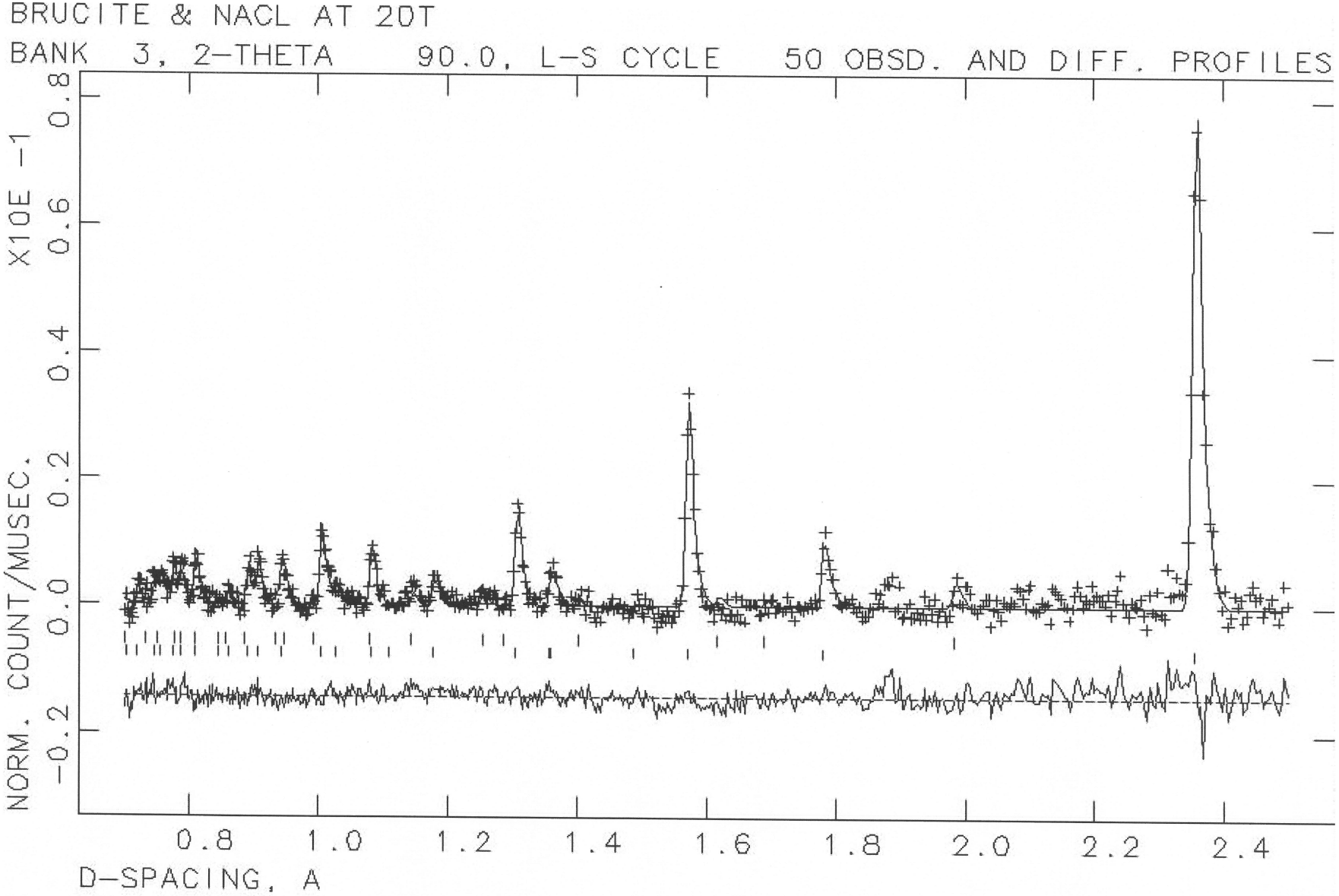
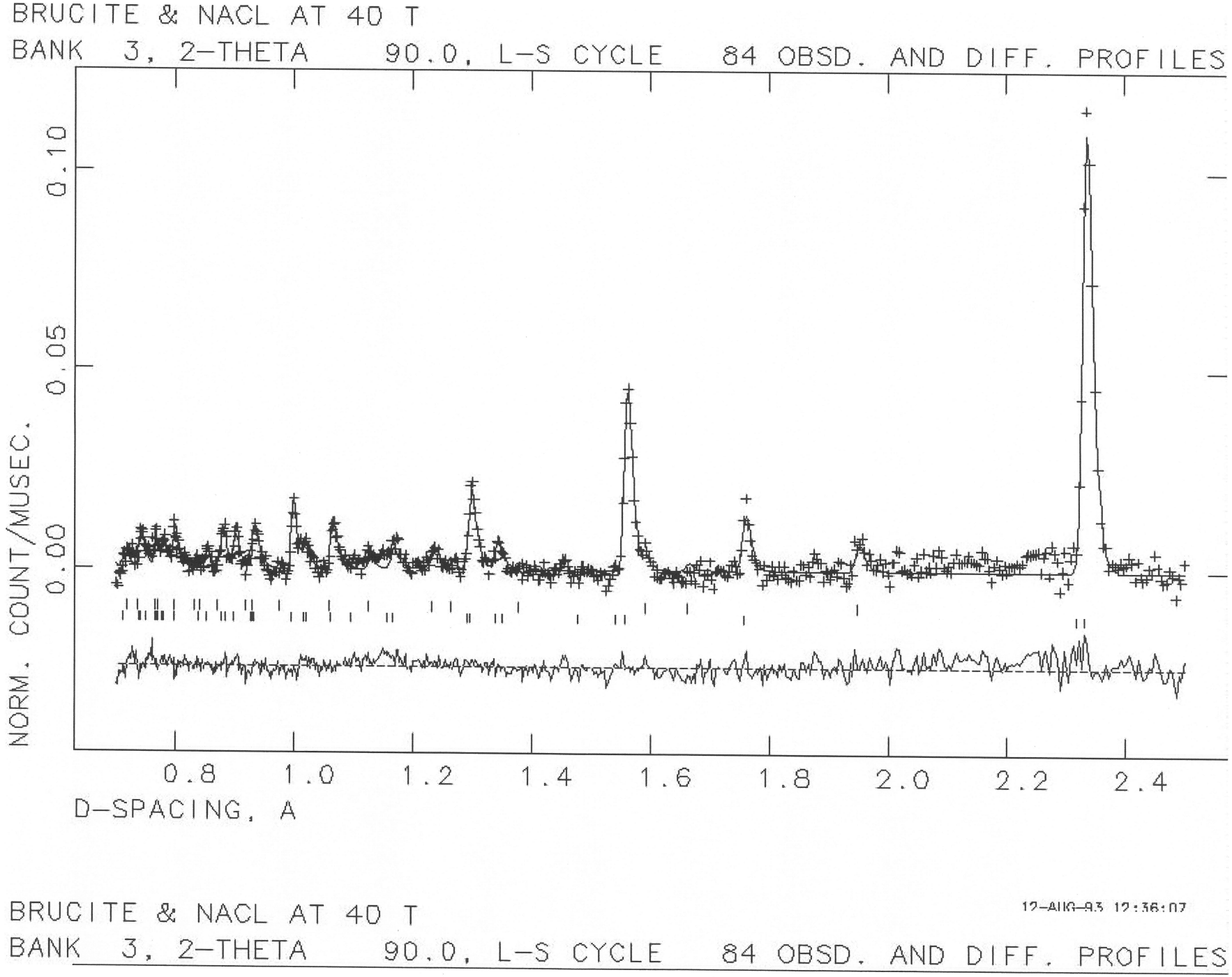
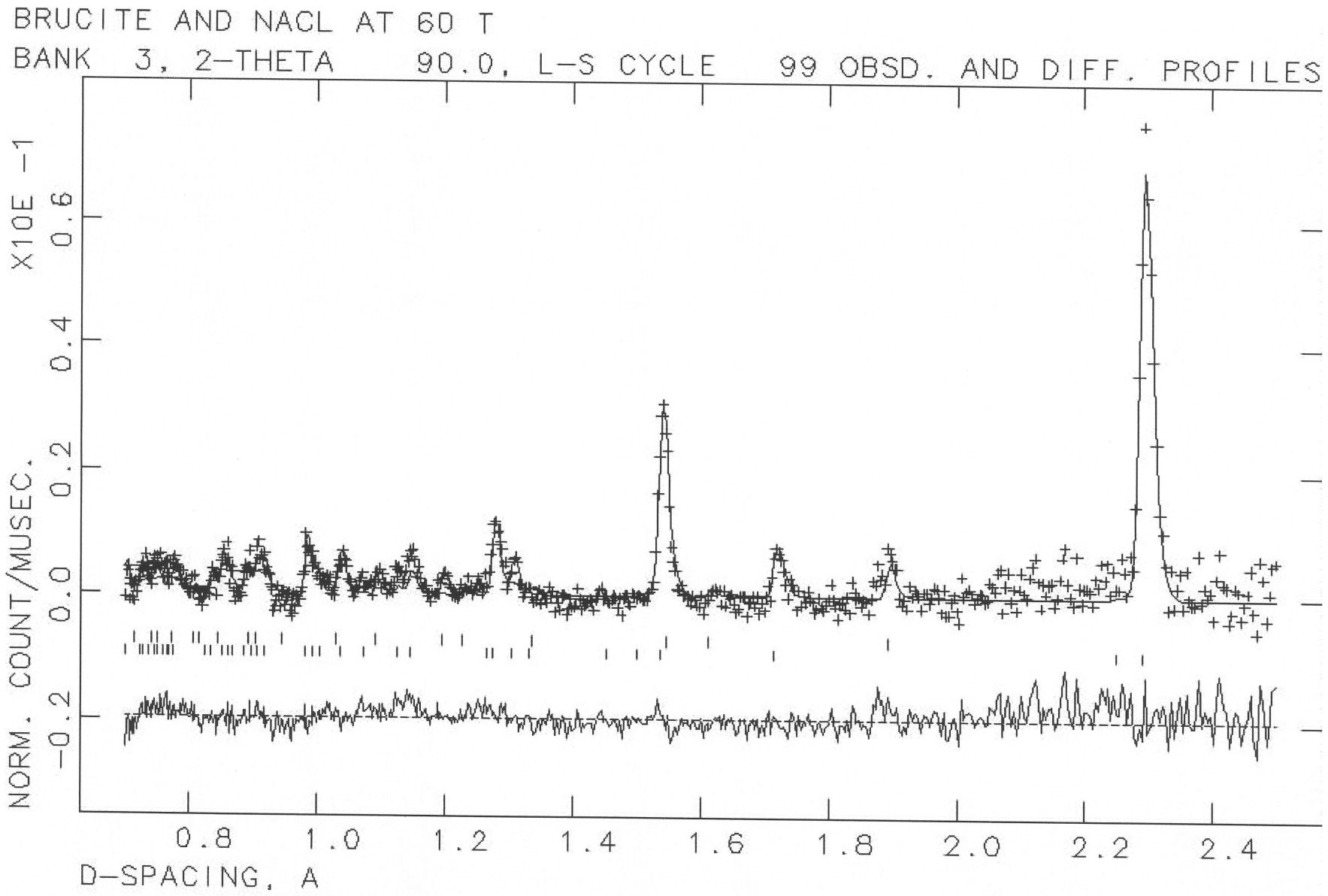


BRUCITE & NACL AT 20T
BANK 3, 2-THETA 90.0, L-S CYCLE 50 OBSD. AND DIFF. PROFILES

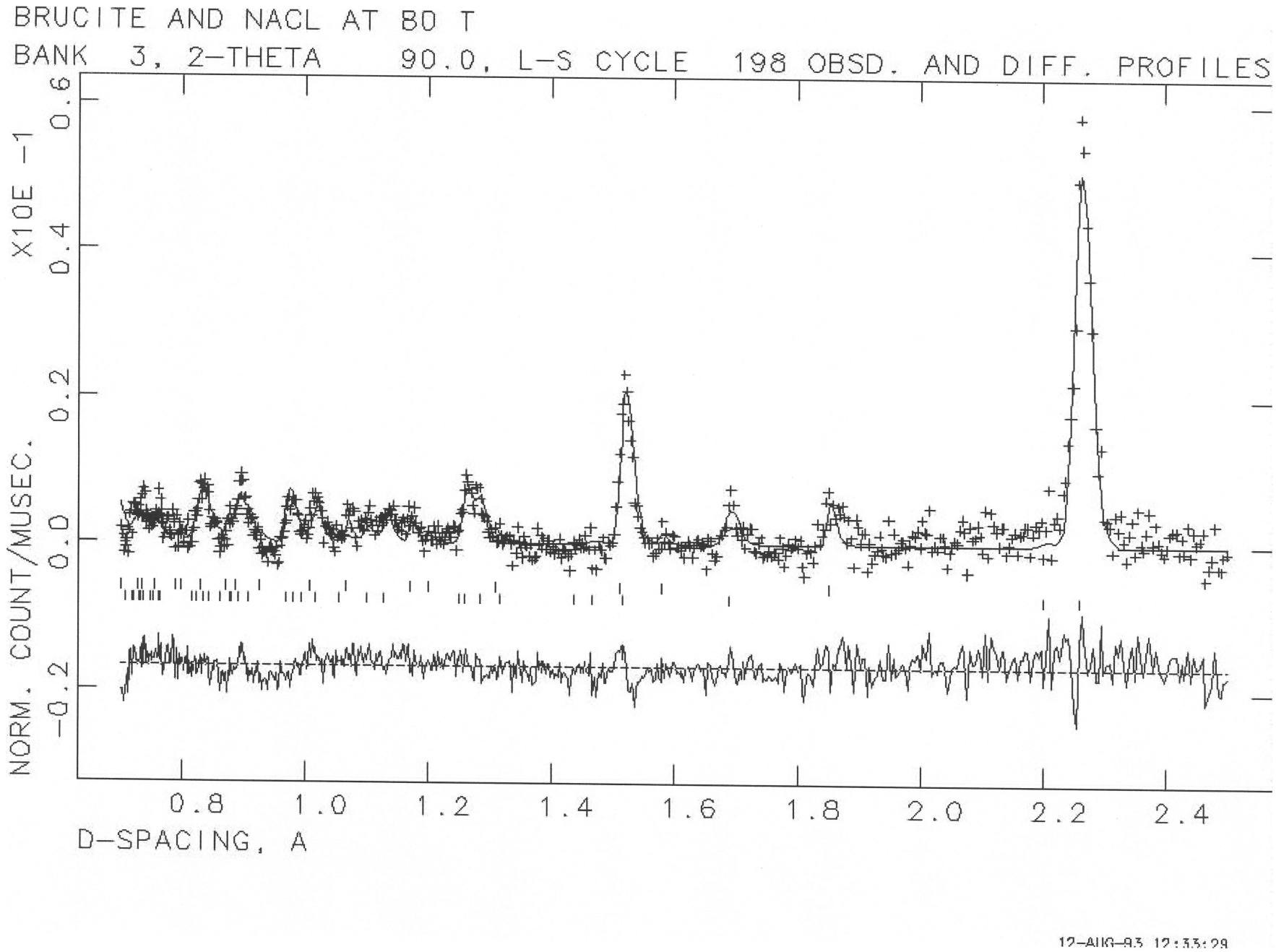
12-AUG-93 12:35:15


BRUCITE & NACL AT 40 T
BANK 3, 2-THETA 90.0, L-S CYCLE 84 OBSD. AND DIFF. PROFILES
NORM. COUNT/MUSEC.
0.00
0.05
0.10
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
D-SPACING, A
BRUCITE & NACL AT 40 T
12-AUG-93 12:36:07
BANK 3, 2-THETA 90.0, L-S CYCLE 84 OBSD. AND DIFF. PROFILES



BRUCITE AND NACL AT 60 T
BANK 3, 2-THETA 90.0, L-S CYCLE 99 OBSD. AND DIFF. PROFILES

12-AUG-93 12:37:15


BRUCITE AND NACL AT 80 T
BANK 3, 2-THETA 90.0, L-S CYCLE 198 OBSD. AND DIFF. PROFILES
NORM. COUNT/MUSEC. X10E -1
0.6
0.4
0.2
0.0
-0.2
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
D-SPACING, A
12-AUG-93 12:33:29