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PUERTO RICQ NUCLEAR CENTER

NEUTRON DIFFRACTION PROGRAM

Progress Summary Report No. 1

{10 BY UNIVERSITY OF PUERTO RICO UNDER CONTRACT

NO. AT (40-1-183 FOR U. 5. ATOMIC ENERGY COMMISSION

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NEUTRON DIFFRACTION

Progress Sucmary Report No.

March 1963)

Tomsel Alaodévar - Principal Investigator

with Guest Selentiete

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Puerto Rico Nuclear Center

operated by

university of Puerto ico

for the

U.S. Atonie Energy Comtesion

under Contract No. AT-(40-1)-1833,

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sa.

One of the major uses of a research reactor in basic investi-

Bation in the physical sciences is for neutron diffraction studies.

?The establishaont of © progran in thie ficld during the past year is expected to be of key inportonce in the future development of research 4m the physical sciences ot the Nuclear Center. A brief sumaty of Program accomplishsonte in 7¥-1964 is presonted in this report.

1. Spectroster Instat Lacion

Since thts wos the first year of the project, considerable

fraction of PY-1965 wae taken up with the construction, installation,

and ts

ting of equipeant. One of the Evo couble-erystal neutron spec trometers to be used in the program wont into research operation in January 1963. A view of this machine { shown in Pigure 1. A Liquid helium research cryostat is mounted on the couple table. Pigure 2 shove ¢ test pattern of powdered iron. This wae the first neutron

@iftraction data taken in Pusrto Ri

Installation of the other spectrometer is expected very soon,
This will be « high qwality, versatile instrument of the same basic design betng used by Brookhaven in ite epestroneter construction Progran for tho new sigh Flux Seam Reactor. The construction te being done at Brookhaven ta parsliol vith that program,

The

ectronater which 1s nov in use at the Huclear Center was also ?sequired under @ cooperative arrangeuent with Brookhaven. The in-pile colliaation, shutter, conochtonstor plug, and basic out-pile shielding

were constructed there on a cost reiabursencat bi

6. The at ffracto-

meter portion was already available end vas transferred to the Nuclear

Genter without cherge. This Latter portion of the spectroseter is en

Old, relatively sicple machine, vhich had becone obsolescent at

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Pigure 1, Neutron spectrometer now in research operation at the

Puerto Rico Nuclear Center. A liquid helium cryostat

Ls mounted in the sample position.

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NEUTRON COUNTS / 50 SECONDS

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Figure 2.

Me pomderes cytinavicai samp.

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3234 36 38 40 42 44 48 48 50 52

COUNTER ANGLE °2@

Neutron diffraction cest patcers of powered iron.

f{rst data taken on che specteoneter.

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wae

Brookhaven, and vas Literally rescued ftom the scrap heap. In

February an excellent sevtron dffractoneter was donated to the

University of Puerto Rico by the Westinghouse Research Laboratories,

4nd on azrangenene {8 being made vith the University to replace the
older sinple machine With the nev Yestinghouse unit. The old unte
WALL probably be eeanafecced at cost to ABC contract research at the
Georgia Institute of Technology

2 caroy

?The structure of Calo, (scheelite) vs

the {rst neutron dif-
fraction search problem undertaken at the tuclear Center. York vas
done in collaboration with Dr. ti. I. Kay of the Goorgia Institute of
Technology. (1)

?A number of crystals having the tetragonal-scheelite structure
hhave been exomined by X-rays. These studies have yielded the heavy
?atom positions, but in most cases the oxygens could be located only
from packing considerations, and in all cases the uncertainties in
?the oxygen parameters ere quite large, The discrepancies between
the scructures reported for various scheelite-type compounds are

ye than one would expect in an isomorphous series:

Nath neutron diffraction date an accurate direct refinement of

all poraneters is possible. Starting with oxygen coordinate para

eters of Sillen and aylander,(2) a set of single crystal (KOL)

(1) Poper submitted by il. 1. Kay, B.C. Frazer and T. Almodéver for

Presentation at the int'l Union of Crystallography, tome, Italy.

Sept. 9-18, 1963.

(2) L. G. eitlen and A. 1. tylander, Arkiv Kemi, Min. Geol. 17A,

to. 4 (1943).

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o3-

neutron data was refined by least squares to yield = 0.2617 + 0.0007,

1 = 0.1522 4 0.0009, and = = 0.0861 + 0.0002. These ney be compared

with the set proposed in the X-ray study of Sillen and tylLander:

= 0.25 + 0.02, y = 0.15 0.02, 2 = 0.075 + 0.015. Here onygen is

4a the 16 (6) general position of space group I 4j/a. An appreciable

change is noted in the 2 paraneter, although the neutron value fells

within the rather Large estinated error Limits

3. Balio, snd FopSi0,

Prelininary neutcon deta have been taken on these two compounds

using powder diffraction techniques. the magnetic spin structures of

these crystals are of principal interest, although the chesieal

structure of Bafi0, is also of considerable interest.

According to the ?ray study of Lander), che 112 ton in Basidy

is surrounded by oxygens in approxinately square planar coordination.

?The oxygen squares share edges to form 2ig-2ag cheins in the ¢ direction

of the orthorhombic Caem unit cell. ordi

sFLly, one vould not expect

Mi co have a magnetic moment vhen in sque

planar coordination, but

in this compound the moment appears to have nearly the ?normel? moment

of the divalent ton (approximately 2y,). Lander points out that thie te undoubtedly due to the unusually short Ml distance in the chains. ?This distance te only slightly lerger then in metallic nickel.

?The structure determination of Lander was not carried out with nigh precision, and the oxygen positions vere located mostly on the basis of packing considerations. Neutron date collected at room and Liquid aitrogen temperatures did not agree very vell with Lender's

proposed structure, although preliminary analysis indicates that his

() J. 3. Lander, Acta Cryst. 4, 148 (1951),

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general model is correct. The alternate sodel one can derive from

packing considerations is definitely incorrect. Adjustment of the

oxygen and bariun paramoters is now in progress, and preparations

are undervay to es

wine the crystal for mgnetic order at 1:quid

helium temperature,

Fep8i0, has Leen studied so far only at room texperature. The

observed and celeclated intensities for the chanical str:cture agree

fairly voll using the paraneters of ep510, (olivine) arehough

sone adjustment nay be necessary. An accurate chesteal structure to

especially inporteat in this case, for only half of the iron atons

are in symetry-fixed positions, Low temperature expericental studies

are planned for the sear future.

(4) YL v. Botov, 8M. Belova, HH. Andeianové

Dokl: Akad. ?vauk $852 81,399 (1951).

and P. R, Sufrnova;

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