UNCLASSIFIED UCRL-2065

UNIVERSITY OF CALIFORNIA — BERKELEY

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

RADIATION LABORATORY

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

UCRL-2065
Unclassified-Physics Distribution

UNIVERSITY OF CALIFORNIA

Radiation Laboratory

Contract No. W-7405-eng-48

SUMMARY OF RESEARCH PROGRESS MEETING OF NOVEMBER 20, 1952

Sergey Shewchuck

December 30, 1952

"Some of the results reported in this document may be of a preliminary or incomplete nature. It is the request of the Radiation Laboratory that the document not be circulated off the project nor the results quoted without permission."

Berkeley, California

SUMMARY OF RESEARCH PROGRESS MEETING OF NOVEMBER 20, 1952

Sergey Shewchuck

Radiation Laboratory, Department of Physics,
University of California, Berkeley, California

December 23, 1952

I. Preparation and Composition of TbF_4. D. C. Feay

The main purpose of this experiment was to obtain a compound of tetravalent terbium which would be more practical and suitable for calorimetric and other thermodynamic measurements than terbium dioxide, so that possibly a better comparison could be made of the properties of the lanthanide and actinide elements. Although terbium dioxide has been known before, it has never been actually prepared in a pure form due in part to its rather high crystal energy.

Of the rare earths there are very few elements with a plus four valence state. Cerium has a well known plus four state and even the plus five state has been obtained with extreme difficulty. The cerric ion is often used as a strong single electron oxidizing agent. Praseodymium reaches the plus four state only in its dioxide. Neither praseodymium nor terbium have a plus four ion stable in aqueous solution.

The first method attempted to obtain tetravalent states was to react solid trifluorides and oxides of the lanthanides with bromine trifluoride vapor at about 500°C. However, the only identifiable products were the trifluorides, identification being made by x-ray powder diffraction techniques. The method which was finally successful was the reaction of elemental fluorine with terbium trifluoride. The schematics of the apparatus used are shown in Figure 1.

The system was first swept out with argon to remove any gas that might react violently with fluorine. Then the argon flow was shut off and the fluorine flow and heating started. The fluorine first passes through a trap filled with potassium fluoride and chilled with dry ice. Any HF in the fluorine is then removed as KHF_2. Later, a second trap was inserted here to make the trapping more efficient. Then it passed through the reaction chamber. The trap just beyond the reaction chamber was filled with calcium bromide and cooled in a water bath only. The fluorine reacted with the calcium bromide forming

calcium fluoride and bromine which is less dangerous and easier to dispose of than fluorine. Not shown in Figure 1 is a beaker of sodium sulfite solution that oxidizes the bromine to bromide in order to remove bromine vapors from the air. After it was found that perfect efficiency could not be realized, the second trap was found to serve no useful purpose and was removed. The fluorine flow was continued for an hour after the temperature of the reaction zone reached about 350°C; the temperature of the furnace had been found to be about 550°C at this point. Then the heating was stopped but the fluorine flow was not stopped until the temperature of the furnace was down to about 200°C. The system was swept out with fluorine and the sample was removed and stored in vacuum in pyrex tubes.

The starting materials used were terbium trifluoride prepared from Tb_4O_7 and cerium trifluoride prepared from high purity cerric sulfate. Both samples were spectrographically analyzed after the tetrafluorides had been prepared and found to be better than 99.9 percent pure. Praseodymium, dyprosium, samarium and neodymium trifluorides were also tried as starting materials but no tetrafluorides resulted from any of them.

Computations on the purity and composition of the newly obtained tetrafluoride of cerium gave a value of 98.28 percent tetrafluoride, or $CeF_{3.98}$. The data on terbium tetrafluoride proved somewhat more difficult to interpret; however, two determinations gave $TbF_{3.97}$ and $TbF_{3.87}$, or an average composition of $TbF_{3.92}$. Terbium and cerium tetrafluoride are monoclinic with the zirconium tetrafluoride structure and, hence, are isomorphous with the actinide tetrafluorides. The lattice constants of these two compounds are:

	CeF_4	TbF_4
a	12.6Å	12.1Å
b	10.6	10.3
c	8.3	7.9
β	126°	126°

The terbium measurements are all plus or minus 2 percent. The decrease in the constants from cerium to terbium tetrafluoride is in agreement with the lanthanide contraction.

Terbium tetrafluoride is insoluable (or very, very slowly soluable in H_2O), dissolves slowly in HCl or HNO_3 with no visible reaction, dissolves in acidic KI solutions with the oxidation of iodide ion, and dissolves in $Al(NO_3)_3$ solutions with bubble formation. It, therefore, has about the same soluability properties as cerium tetrafluoride except that it dissolves with more visible reaction.

II. Study of Triton Production in d-d Collisions. C. S. Godfrey

This report covers the cross section angular distribution for the reaction $d + d \rightarrow H^3 + p$ using 190 Mev deuterons. It is hoped to compare this later with the cross section for the reaction $d + d \rightarrow He^3 + n$. A schematic of the equipment used is shown in Figure 2. The tritons measured ranged from about 170 Mev at 5° to 100 Mev at 30° in the lab system. The targets used were CD_2, CH_2 and C. Both CD_2 - CH_2 - C differences were obtained. Since the CH_2 count was slightly higher than the C count, the final cross section was based on the CD_2 - CH_2 difference. The distance of the proton telescope was chosen to give approximately equal solid angles in the center of mass system. The electronics took advantage of the time of flight difference between the proton and triton to discriminate against unwanted background. Under the conditions shown above, the C background rate was only 4 percent of the CD_2 rate. Accidental coincidences were checked by noting the counting rate at one-fifth beam intensity. The net counting rate was identical, showing that accidentals were negligible.

To make sure the counts truly represented the desired reaction, the following checks were made: (1) An additional delay of 5 millimicroseconds in the proton telescope wiped out the net counts. (2) Azimuthal or vertical movement of the triton telescope by two resolution widths wiped out the net counts. (3) Addition of enough absorber between the two crystals to stop the postulated particles wiped out the net counts.

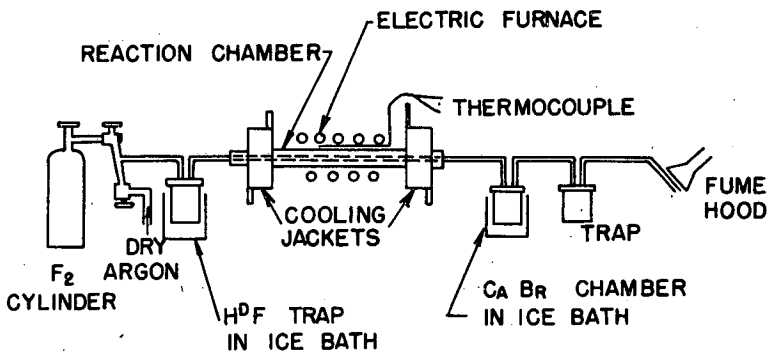
In order to be able to measure the cross section at various angles and various proton energies, it was necessary to move in the triton telescope to eight feet and eliminate one proton crystal.. Under these conditions, the background counting rate went up but not excessively. It ranged from 10 to 25 percent for the angles used. Data were obtained for the angles 45° to 60°, and

90° in the center of mass system. These data are shown in Figure 3. It is hoped to complete these data for the angles 15° and 30° in the near future. The steep rise in the forward direction suggests the possibility of a pick-up type of reaction. At any rate the complete cross section distribution will provide useful information on the internal momentum distribution in the deuteron.

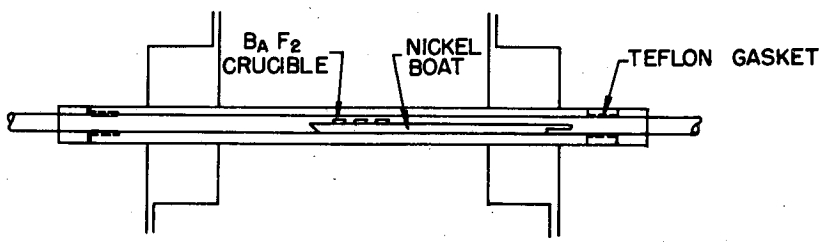
This preliminary experimental run was part of the work to be described and included in a thesis by the author to be completed sometime in the future.

Information Division
12/30/52 dm

GENERAL VIEW OF APPARATUS



REACTION CHAMBER



MU 3194

Fig. 1

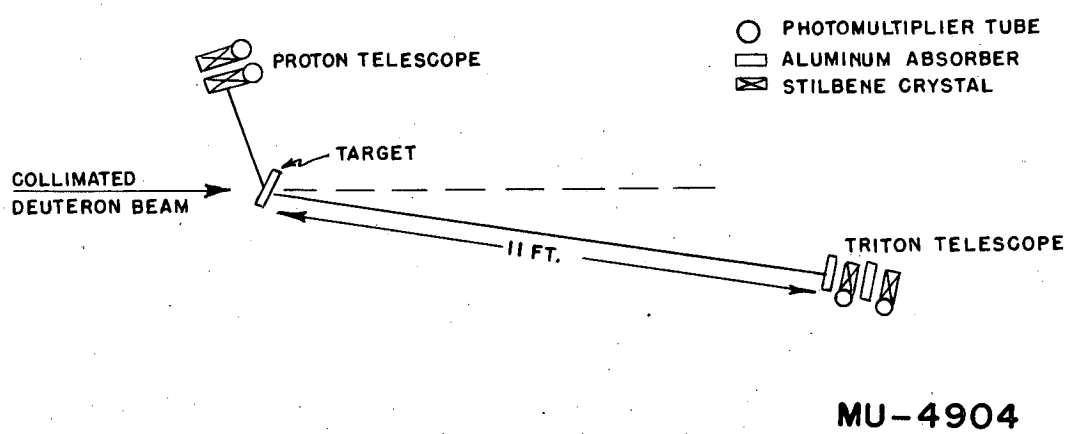

PHOTOMULTIPLIER TUBE
ALUMINUM ABSORBER
STILBENE CRYSTAL
PROTON TELESCOPE
TARGET
COLLIMATED
DEUTERON BEAM
11 FT.
TRITON TELESCOPE
MU-4904

Fig. 2

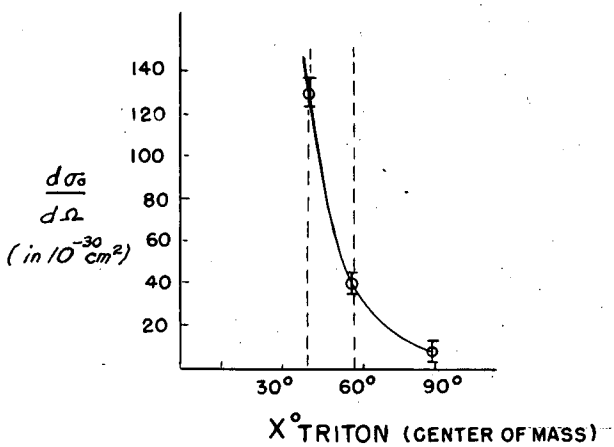

140
120
100
80
60
40
20
$\frac{d\sigma_0}{d\Omega}$
(in 10^{-30} cm^2)
30°
60°
90°
X° TRITON (CENTER OF MASS)

FIG. 3

MU-4903