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QUARTERLY PROGRESS REPORT

LABORATORY RECORDS 1954

STABLE ISOTOPE RESEARCH AND PRODUCTION DIVISION

April 1, 1952 to June 30, 1952

OAK RIDGE NATIONAL LABORATORY
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OAK RIDGE NATIONAL LABORATORY
OPERATED BY
CARBIDE AND CARBON CHEMICALS COMPANY
A DIVISION OF UNION CARBIDE AND CARBON CORPORATION
OAK RIDGE, TENNESSEE

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Index No ORNL-1356
This document contains 26 pages
This is copy 35 of 74 Series A

Subject Category Isotope Separation

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QUARTERLY PROGRESS REPORT
Stable Isotope Research and Production Division
April 1 1952 to June 30 1952

C P Keim Director

[redacted]

August 5 1952

OAK RIDGE NATIONAL LABORATORY
Operated By
CARBIDE AND CARBON CHEMICALS COMPANY
A Division of Union Carbide and Carbon Corporation
Oak Ridge Tennessee

Contract No W-7405-eng-26

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Index No ORNL-1356
Isotope Separation

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STABLE ISOTOPE SEPARATION RESEARCH AND CALUTRON OPERATIONS

C E Normand Department Head

INTRODUCTION

During the fourth quarter of fiscal year 1952 isotopes of tin barium and boron were collected by the Isotope Production and Process Development Department and the investigations of power regeneration reported in a previous progress report [1] were continued under an expanded program now designated as the "Controlled Collections Program"

COLLECTIONS

Three elements were processed yielding nineteen isotopically enriched samples These collections were made by operating personnel under the direction of W A Bell R L Caldwell G J Fisher C V Ketron L O Love and K A Spainhour

Tin Series FS-XAX

Collection of the isotopes of tin (Sn112 Sn114 Sn115 Sn116 Sn117 Sn118 Sn119 Sn120 Sn122 and Sn124) was started in February 1952 in the Alpha channels and was completed in April

Stannic chloride ($SnCl_4$) charge a liquid at room temperature was supplied from an external charge container through a control valve and

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1 "Quarterly Progress Report Stable Isotope Research and Production Division" ORNL-1298

3/8-inch charge feed line into the arc chamber of a low temperature double gas feed source equipped with a deep floating anode ionization chamber standard zero degree electrodes and a 1/8-inch ion exit slit The isotopes were collected in carbon receiver pockets placed behind 1/8-inch slits in a carbon defining faceplate

For the series a total monitored ion collection of 23 628 ma hrs during 885 hours of innage yielded the following estimated weights of tin isotopes

Sn112	0 72 gm
114	0 65 gm
115	0 70 gm
116	13 34 gm
117	7 66 gm
118	24 77 gm
119	9 42 gm
120	36 36 gm
122	5 39 gm
124	5 80 gm
Total	104 81 gm

Barium Series FT-XBX

Collection of barium isotopes (Ba130 Ba132 Ba134 Ba135 Ba136 Ba137 and Ba138) was started in March 1952 in the Beta channels and was completed in April No significant changes were made in equipment or operation as reported in the progress report for the preceding quarter [1]

A total monitored ion collection of 16 268 ma hrs during 554 hours of innage yielded the following estimated weights of collected isotopes

Ba130	0 16 gm
132	0 16 gm
134	2 66 gm
135	1 26 gm
136	9 09 gm
137	1 36 gm
138	68 80 gm
Total	83 49 gm*

*(11 3 grams rejected because of mismonitoring)

Barium Series FU-XAX

Barium was also processed in XAX beginning in April and ending in June 1952 High temperature source units were used in these as in the XBX collections of this element Large separation at the four-foot collection radius made possible the simultaneous collection of all seven of the barium isotopes

A total monitored ion collection of 19 457 ma hrs during 838 hours of innage yielded the following estimated weights of enriched isotopes

Ba130	0 21 gm
132	0 26 gm
134	2 40 gm
135	6 26 gm
136	8 81 gm
137	14 11 gm
138	67 68 gm
Total	99 73 gm

Boron Series FV-XBX

Collection of the isotopes of boron (B10 and B11) was started in April 1952 in the XBX channels and completed in June

The charge material was born trichloride (BCl_3) supplied from an external charge container to a double gas feed source Standard accelerating electrodes and 1/8- (or 3/16-) inch ion exit slits were used The standard arc chamber used in early runs (Figure 1a) was later modified by the insertion of a carbon baffle as shown in Figure 1b This modification provided more uniform distribution of vapor to the arc and at the same time confined the arc to a narrow region immediately behind the ion exit slit As a result the arc current was reduced from six amperes to three amperes and the average ion output was increased from about 15 ma to approximately 25 ma

A total monitored ion collection of 42 737 ma hrs during 1 740 hours of innage yielded the following estimated weights of enriched isotopes

B10	2 99 gm
11	14 23 gm
Total	17 22 gm

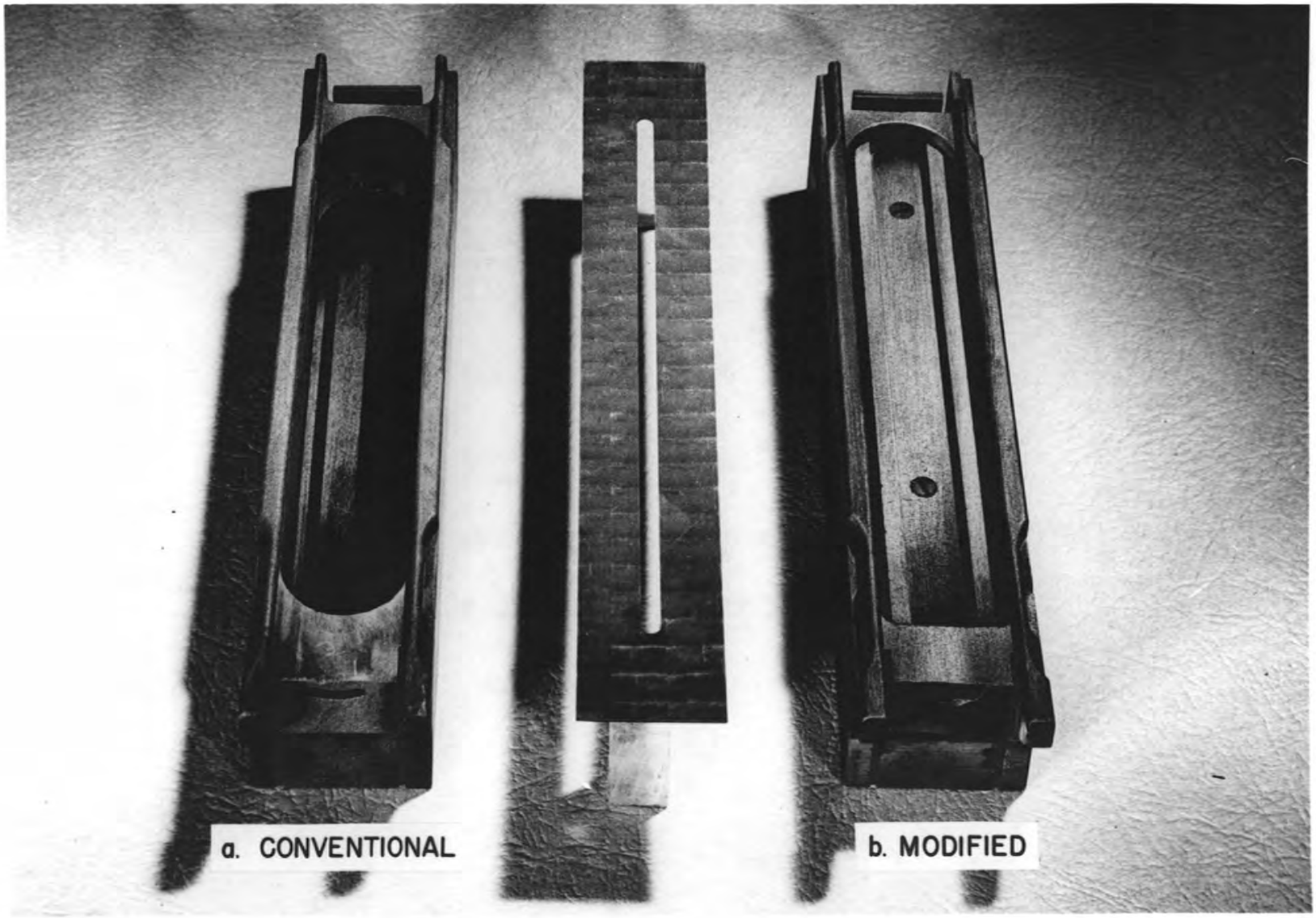

FIGURE I. ARC CHAMBERS

DEVELOPMENT

Controlled Collection (G M Banic W A Bell L O Love and W K Prater)

During this quarter a total of 1 142 hours of XAX tank time was devoted to continued investigations of power regeneration and to the associated problem of developing a high output ion source with primary attention being given to the latter problem Grid diode type sources were successfully operated with arcs up to thirteen inches in length yielding a total lithium ion output of as much as 2 300 ma at a collector plate placed near the 180 position Studies made of beam profiles in the collector region indicate acceptable resolution of the lithium beams at a radius compatible with operating values of the magnetic field and accelerating potential

A production type unit designed on the basis of these tests will be built and tested for output retention and purity of product

STABLE ISOTOPE CHEMISTRY RESEARCH AND PRODUCTION

Boyd Weaver Department Head

INTRODUCTION

The chief production functions of the Isotope Chemistry Research and Production Department are the supply of suitable charge materials for isotope separation by the calutrons chemical refinement of enriched isotopes and the preparation of materials for their mass analysis Chemical services and unusual compounds are also supplied to other departments of the Stable Isotope Research and Production Division

The department carries on research activities in various fields related to isotope production and use with current interest centered around the rare earth elements

All refined isotopes are stored in the chemistry depositories and the preparation of all samples for shipment is performed by personnel of this department Unused portions and many processed residues of isotopes are returned by research institutions for restoration in the laboratory's stock of isotopes Some of these require reprocessing to make them suitable for reuse others are too small quantities for further handling

QUARTERLY SAMPLE SUMMARY

Isotope samples on which refinement was completed	28
Samples in process of chemical refinement June 30	8
Samples awaiting chemical refinement June 30	11
Samples submitted to Mass Analysis Laboratory	24
Mass analyses reported	17
Samples awaiting mass analysis June 30	38
Stable Isotope shipments made	77

PRODUCTION OF CHARGE MATERIALS

Preparation and Purification (C W Sheridan C F Harrison and W C Davis)

Charge materials of barium cerium boron neodymium and lithium were prepared for calutron operations Equipment was assembled for the

preparation of ruthenium fluoride to be tested as a possible charge material

ISOTOPE REFINEMENT

Special Projects (W C Davis R L Bailey F B Thomas and F M Scheitlin)

Much of the effort of the department was devoted to special production and research projects which will be reported in a special report

Refinement (R L Bailey C F Harrison and F B Thomas)

Work was done on the chemical refinement of seven elements Iron calcium lead lithium cerium nickel and tin In three cases a whole series of isotopes was completed

Lead was refined by a procedure described previously [2] Production data for the series are tabulated below

Isotope	204	206	207	208
Weight of PbO	3 1986 g	8 7569 g	8 8321 g	9 1329 g
IMPURITIES FOUND (PER CENT)				
Al 0 06	Cu<0 04	Cu<0 04	Cu<0 04
Ba 0 02	Fe<0 04	Fe<0 04	Fe<0 04
Cu<0 04	Si <0 04	Mg<0 04	Mg<0 04
Fe 0 04	Mg<0 04	Si <0 04	Mn<0 04
Li 0 01	Sn 0 02		Si <0 04
Si <0 04			Sn <0 02

Tin was refined by a procedure which included the following steps

1 Ignition of carbon pockets
2 Reduction of tin in ash with hydrogen
3 Chlorination of metal and collection of chloride in HCl
4 Alkaline sulfide separation
5 Precipitation of SnS_2 from acid sulfide solution
6 Ignition of SnS_2 to SnO_2

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2 Y-636

It is to be noted that part of the product was lost by volatilization in the final step though both the sulfide and oxide are supposed to have very low vapor pressures at the temperature used Production data are tabulated below

Isotope	112	114	115	116
Weight of SnO_2	0 2702 g	0 1407 g	0 1139 g	5 6535 g

IMPURITIES FOUND (PER CENT)

Cu 0 04	Cu 0 04	Cu 0 04	Cu<0 04
Fe<0 04	Fe 0 04	Fe 0 04	Fe 0 04
K 0 01	Ni 0 08	Mg<0 04	Mg<0 04
Li 0 01	Mg<0 04	Na 0 01	Si 0 04
Mg<0 04	Si <0 04	Si 0 04	
Na 0 01			
Si 0 04			

Isotope	117	118	119
Weight of SnO_2	3 3495 g	15 0345 g	5 6257 g

IMPURITIES FOUND (PER CENT)

Cu <0 04	Al <0 04	Cu<0 04
Fe <0 04	Cu<0 04	Mg<0 04
Mg<0 04	Fe 0 08	
	Mg<0 04	

Isotope	120	122	124
Weight of SnO_2	21 1039 g	0 6684 g	0 7036 g

IMPURITIES FOUND (PER CENT)

Cu<0 04	Na<0 04	Cu 0 10
Mg<0 04	Si 0 04	Fe<0 04
		Na 0 04
		Si 0 08

Cerium was refined by a procedure which included the following steps

1 Removal of cerium from copper pockets with HNO_3
2 Separation from copper by repeated NH_4OH precipitations
3 Acid sulfide precipitation
4 Precipitation of hydroxides and alkaline sulfides in presence of tartrate
5 Acidification with HCl
6 Precipitation of cerium as oxalate
7 Ignition of oxalate to oxide

Since the charge material had been specially purified in this laboratory it was not considered necessary to remove other rare earths from the product Production data are tabulated below

Isotope	136	138	140	142
Weight of CeO_2	0 4622 g	1 3578 g	33 7744 g	7 8376 g

IMPURITIES FOUND (PER CENT)

		Al <0 04	
Cu <0 02	Cu <0 02	Cu <0 04	Cu <0 02
Fe <0 04	Fe <0 04	Fe <0 04	Mg <0 04
La <0 15	La 0 15	Mg <0 04	Nd <0 3
Mg <0 04	Mg <0 04	Na 0 01	
Na 0 01	Na 0 01	Si <0 04	
Si <0 04	Si <0 04		

ACTIVITIES ASSOCIATED WITH MASS ANALYSIS

Compound Preparatio (F M Scheitlin)

Isotopes of seven elements were prepared in suitable form for mass analysis Lead strontium lithium cerium copper calcium and vanadium

SEPARATION OF RARE EARTHS

Separation by Liquid-Liquid Extraction (F A Kappelmann F B Thomas F M Scheitlin R L Bailey and A C Topp)

Further progress was made in separating the constituents of a gadolinium concentrate and extraction data were obtained by batch extractions and by operation of a small Scheibel column

ISOTOPE SHIPMENTS AND REQUESTS

Preparation of Shipment

Seventy-seven individual samples were prepared for shipment to laboratories within and outside of the Atomic Energy Commission Several

samples returned by users were also processed for further usage Details of the shipments during the quarter and the present status of requests for isotopes are given in Table I

Stable Isotope Sales (Fees)

On the basis of the nominal charge of $50 00 made to most users of stable isotopes for individual shipments the following summary has been compiled

	January 1 to March 31, 1952	April 1 to June 30, 1952
Transfers within AEC	$2 450 00	$1 750 00
Cash Sales (Fees)	$1 650 00	$2 100 00
Total Sales (Fees) and Transfers to Date	$74 000 00	$77 950 00

TABLE I

SHIPMENTS OF STABLE ISOTOPES DURING APRIL MAY AND JUNE 1952

Request Serial No	Requestor	Isotope	Series No	Sample No	Quantity Shipped (mg)	Date Shipped
B 2564	General Electric Research Laboratory	Mo96	AW	166(a)	500 0	4 2 52
B 2585S	Argonne National Laboratory	Mo98	AW	168(a)	600 0	4 3 52
B 2584S	Argonne National Laboratory	Mo97	AW	167(a)	600 4	4 3 52
B 2583S	Argonne National Laboratory	Mo96	R T	56(a)	600 0	4 3 52
B 2582S	Argonne National Laboratory	Mo95	R T	55(a)	601 1	4 3 52
B 2580S	Argonne National Laboratory	Mo92	R T	53(ar)	479 6	4 3 52
B 2617S	Argonne National Laboratory	Zr94	EE	542(a)	56 3	4 4 52
B 2589	Washington University	Ge72	BD BE	206 211(a)	200 7	4 4 52
B 2590	Washington University	Ge74	BD BE	208 213(a)	201 0	4-4 52
B 2591	Washington University	Ni58	DF	422(a)	200 8	4 4 52
B 2592	Washington University	Ni60	J	28(ar)	200 5	4 4 52
B 2593	Washington University	Zn64	EK	556(a)	202 0	4 4 52
B 2594	Washington University	Zn66	EK	557(a)	200 6	4 4 52
B 2595	Washington University	Zn68	EK	559(a)	201 1	4 4 52
B 2596	Washington University	Fe56	CZ	404(f)	200 3	4-4 52
B 2597	Washington University	Cr52	CD	310(a)	201 0	4 4 52
B 2598	Washington University	Ti48	EN	578(a)	200 8	4 4 52
B 2549	Baird Associates Inc	Hg200	DR	488(a)	5 9	4 8-52
B 2550	Baird Associates Inc	Hg202	DR	490(a)	5 8	4 8 52
B 2567	University of California	Ti47	EN	577(a)	900 2	4 14 52
B 2568	University of California	Ti49	EN	579(a)	393 5	4 14 52
B 2609S	Argonne National Laboratory	Pb204	EO	581(a)	50 1	4 16 52
B 2610S	Argonne National Laboratory	Pb206	EO	582(a)	50 8	4 16 52
B 2611S	Argonne National Laboratory	Pb207	EO	583(a)	50 8	4 16 52
B 2612S	Argonne National Laboratory	Pb208	EO	584(a)	51 5	4 16 52
B 2646S	Oak Ridge National Laobratory	Sm144	FH	660(a)	5 1	4 18 52
B 2647S	Oak Ridge National Laboratory	Sm147	FH	661(a)	50 2	4 18 52
B 2567	University of Michigan	Cd106	AC	88(a)	60 6	4 28 52
B 2641	University of Illinois	Ni64	AS AT	146 152(a)	10 7	4 28 52
B 2642	Columbia University	Ni58	AS AT	141 147(a)	100 9	4 28 52
B 2643	Columbia University	Ge70	BD BE	205 210(a)	100 7	4 28 52
B 2677S	Oak Ridge National Laboratory	Sm144	FH	660(a)	5 2	4 28 52
B 2636	Ohio State University	Se80	FG	658(b)	300 5	4 29 52
B 2637	Ohio State University	Se78	FG	657(b)	301 6	4 29 52
B 2693	Washington University	Ca48	FI	653(a)	50 2	5 7 52
B 2662	Massachusetts Institute of Technology	Ge70	BD BE	205 210(a)	100 1	5 12 52
B 2632	University of Michigan	V50	FM	683(b)	10 3	5 12 52
B 2711S	Oak Ridge National Laboratory	Fe54	CI	328(a)	20 6	5-15 52
B 2712S	Oak Ridge National Laboratory	Fe54	DO	477(a)	20 4	5 15 52
B 2690	Princeton University	Mo92	AW	163(ar)	10 7	5 15 52
B 2691	Princeton University	Mo94	AW	164(a)	10 5	5 15 52
B 2703S	Argonne National Laboratory	Cd108	AX	170(a)	340 4	5 22-52
B 2704S	Argonne National Laboratory	Cd108	AX	170(ar)	61 1	5 22 52
B 2705S	Argonne National Laboratory	Cd110	CE	316(a)	600 7	5 22 52
B-2706S	Argonne National Laboratory	Cd111	CE	317(a)	600 5	5 22 52

Request Serial No	Requestor	Isotope	Series No	Sample No	Quantity Shipped (mg)	Date Shipped
B 2707S	Argonne National Laboratory	Cd112	CE	318(a)	599 9	5 22 52
B 2708S	Argonne National Laboratory	Cd113	CE	319(a)	600 2	5 22 52
B 2709S	Argonne National Laboratory	Cd114	CE	320(a)	601 4	5 22 52
B 2710S	Argonne National Laboratory	Cd116	AB	87(a)	400 1	5 22 52
B 2726S	Oak Ridge National Laboratory	K41	DT	496(a)	101 1	5 28 52
B 2698S	Ames Laboratory	Te130	FE	647(a)	501 7	5 28 52
B 2689	Columbia Laboratory	Sb121	BT	274(a)	25 4	5 28-52
B 2686	Carnegie Institution of Washington	Sr84	FL	679(a)	30 6	5 28 52
B 2723	The John Hopkins University	Li7	EZ	628(e)	502 6	6 10 52
B 2572S	University of California	Cr52	CD	310(a)	100 9	6 10 52
B 2749S	Oak Ridge National Laboratory	W183	EL	563(a)	500 8	6 10 52
B 2750S	Oak Ridge National Laboratory	W184	CL	343(ar)	401 1	6 10 52
B 2751S	Oak Ridge National Laboratory	W186	EL	565(a)	1 002 0	6 10 52
B 2644	Columbia University	S36	ES	599(a)	3 0	6 10 52
B 2732	Columbia University	S33	CX	392(a)	10 1	6 10 52
B 2447	University of Illinois	Zr96	EE	543(a)	50 7	6 10 52
B 2737	Massachusetts Institute of Technology	Li6	ED	537(b)	10 2	6 13 52
B 2764S	Oak Ridge National Laboratory	Mo98	RT	58(a)	151 3	6 13 52
B 2673	Air Force Cambridge Res Center	Zn68	BK	243(a)	19 3	6 13 52
B 2514	Florida State University	W184	BG	223(a)	50 5	6 16 52
B 2515	Florida State University	W186	EL	565(a)	50 2	6 16 52
B 2701	Northwestern University	Li6	ED	537(b)	75 3	6 27 52
B 1915S	Brookhaven National Laboratory	Ca46	FO	693(a)	15 1	6 27 52
B 2767	University of Illinois	Pb204	EO	581(a)	1 201 2	6 27 52
B 2768	University of Illinois	Tl203	BX	284(a)	2 001 1	6 27-52
B 2775	University of Michigan	Fe58	FN	688(i)	50 4	6 27 52
B 2776	University of Michigan	Mo98	AW	168(a)	50 7	6 27 52

UNFILLED REQUESTS ON HAND JUNE 30 1952

Requests for isotopes which have not been separated

Request Number	Date	Requestor	Isotope	Quantity Desired (mg)
S 148	10 21 47	Argonne National Laboratory	Ir191	20 30
S 148	10 21 47	Argonne National Laboratory	Ir193	300
S 297	10 2 49	Argonne National Laboratory	Pd102 104 105 106 108 110	200 ea
S 342	3 3 49	Oak Ridge National Laboratory	Gd158 160	10 ea
S 343	3 3 49	Oak Ridge National Laboratory	Dy158 160	10 ea

Requests requiring replenishment of Inventory (quantity insufficient)

S 324	3 2 49	Oak Ridge National Laboratory	C137	3 000
S 238	3 2 49	Oak Ridge National Laboratory	B11	10 000

Requests awaiting Purchase Orders

Request Number	Date	Requestor	Isotope	Quantity Desired (mg)
B 2499	2 19 52	University of Pennsylvania	Sb123	600
B 2500	2 6 52	University of Pennsylvania	Sn116	20
B 2688	4 29 52	Columbia University	Cr50	100
B 2758S	6 3 52	Ames Laboratory	Pb204	500
B 1709	6 19 52	Argonne National Laboratory	Ni64	300
B 2779	6 18 52	University of Pennsylvania	Pb207	300
B 2780	6 18 52	University of Pennsylvania	Pb208	250
B 2789	6 19 52	University of Michigan	Zn64	200
B 2788	6 19 52	University of Michigan	Ni64	100
B 2787	6 19 52	University of Michigan	Ni62	100
B 2786	6 19 52	University of Michigan	C137	150
B 2765	6 10 52	University of Michigan	V50	50

Requests with Purchase Order awaiting replenishment of inventory

B 269	3 17 52	Princeton University	B11	500

Requests held by AEC Isotopes Division

S 361		Argonne National Laboratory	Os192	10
S 483		Brookhaven National Laboratory	Ba132	100
S 581		Brookhaven National Laboratory	K40	10
B 397		University of Wisconsin	Ca46	10 1 000
B 736		University of Chicago	Ca46	10
B 875		University of Illinois	Ba132	10
B 1062		Kansas State College	K40	1 000
B 1161		Princeton University	K40	150
B 1696		Naval Medical Research Laboratory	Zn66	3 000
B 1709S		Argonne National Laboratory	Ni64	300
B-1819		University of Illinois	Ce136	10
B 2007S		Brookhaven National Laboratory	Ba132	10
B 2333		Princeton University	Gd152	50
B 2392		Ohio State University	Ba130	100
B 2396		Argonne National Laboratory	Ge76	50
B 2401		Argonne National Laboratory	Nd150	20
B 2577S		University of California	Gd152	10
B 2581S		Argonne National Laboratory	Mo94	600
B 2586S		Argonne National Laboratory	Mo100	600
B 2666		University of Michigan	C135	200

ISOTOPE ANALYSIS METHODS LABORATORY

J R McNally Jr Department Head

Nuclear Resonance (H E Walchli)

Numerous nuclear magnetic resonances are being measured in this laboratory with respect to either the sodium resonance frequency or the deuterium resonance frequency as reference Correlation of these to the same basis depends on the known sodium to deuterium resonance frequency New direct measurements of this ratio have been made using NaCl and NaBr in 20% D_2O without magnetic catalysts with the result

$$\frac{\nu_{Na}}{\nu_{D}} = 1\ 72328 \pm 0\ 00004$$

The limit of error of this measurement is given as 0 00004 and the actual measurement agrees approximately with the ratio calculated from the sodium-proton[3] and the proton-deuterium[4] ratios as 1 72318 The discrepancy is being investigated further

Further studies on the nuclear magnetic resonance in Se77 have revealed a chemical shielding effect of the order of 0 15% in liquid H_2Se as compared to the resonance in H_2SeO_3 [5] The direction of the shift is such that the H_2Se compound has less paramagnetic shielding and hence represents a truer "free nucleus" resonance

Attempts have been made to find hyperfine structure in the nuclear resonances by control over the internal quadrupole electric field gradient available in certain compounds No signal was detected in a frozen crystalline sample of VOCl nor in liquid tertiary butyl iodide $(CH_3)_3CI$ The weakness of the vanadium and iodine signals may be due to the long relaxation time or to a smearing-out of the signal due to quadrupole broadening effects Attempts to align the internal molecular fields by the electret principle may produce the expected structure Formamide $HCONH_2$ which was expected to give a quadrupole structure broadening in the nitrogen resonance actually gave a sharp line resonance at the N14 position

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3 Lindstron G Physica 27 412 (1951)
4 Levinthal E C Phys Rev 78 204 (1950)
5 Dharmetti S S and H E Weaver Jr Phys Rev 86 259 (1952)

Microwave Spectroscopy (H W Morgan and J H Goldstein*)

The J=2 to 3 rotational transition of the asymmetric top molecule vinyl acetylene has been observed in the 27 000 mc region Four of the predicted five rotational lines have been identified Preliminary frequency measurements and Stark effect measurements have been made and these confirm the identifications Equipment is being assembled for use in the region about 18 000 mc where the J=1 to 2 transition will be studied It is expected that experimental difficulties at 27 000 mc will prevent adequate measurement of excited vibrational states and of spectra arising from deuterium substitution

Calculations on the vinyl iodide molecule have been continued based on the previously reported hyperfine structures of the J=3 to 4 and J=4 to 5 transitions of the asymmetric rotor vinyl iodide [6] A report on this spectrum is being prepared for publication

A more detailed study of the hyperfine splittings and refinements in the application of second order theory to asymmetric top molecules have allowed small corrections to be made to certain of the parameters previously reported for vinyl iodide [7] The molecular parameters χ_{aa} and χ_{bb} which are components of ΔE along the inertial axes have been revised to

$$\chi_{aa} = -1656 \text{ mc}$$

$$\chi_{bb} = +770 \text{ mc}$$

A more exact computation of the asymmetry parameter κ yields

$$\kappa = -0\ 992155$$

Spectro-Isotopic Analysis (D D Smith G L Stukenbroeker G K Werner and R E Sladky**)

Numerous studies have been made on the precision of direct-reading photoelectric spectro-isotopic analysis of lithium Sampling time has

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* Consultant Emory University Atlanta Georgia

** On loan from Analytical Department Chemical Division Y-12

6 Morgan H W and J H Goldstein "Microwave Spectrum of Vinyl Iodide" presented at the Southeastern Section of the APS North Carolina State University Raleigh North Carolina April 10-12 1952

7 Morgan H W and J H Goldstein Quarterly Report ORNL-1298 January-March 1952

been reduced to about one man-hour per sample Reproducibility is of the order of 0 3% at the normal abundance level Gross errors introduced in day to day operation indicate the necessity of standardizing the equipment each day A total of 36 unknown standards were run with over-all precision of 0 9% L E Day to day standardization reduced this to 65% L E The advisability of making replicate analyses to reduce gross errors (24 of the aforementioned 36 determinations were within 0 25%) as well as to improve the precision was noted Precision in the range 50% to 90% Li6 was found to be of the same order of magnitude

Optical Spectroscopy (P M Griffin G L Stukenbroeker and R A Loring*)

A two-meter echelle spectrograph has been crossed with a large Littrow glass prism spectrograph and resolution exceeding 300 000 obtained [8] Pressed electrodes containing mixtures of U_3O_8 and copper powder have been found to give very satisfactory operation for Zeeman studies of uranium Improved Zeeman effects of uranium have been obtained in the long wave-length (>5000A) portion of the spectrum Vibration of the steel beam support of the echelle optics has been considerably reduced

Uranium as 1 part of UF_4 and 10 parts Al powder pressed into an aluminum hollow cathode and drilled out except for a thin sheath has been excited successfully to give high intensity Fabry-Perot spectrograms Samples of U235 and U233 are being prepared for hyperfine structure studies

IBM computations have been made for an extended energy interval (8000 cm^{-1} to 20 000 cm^{-1}) in a search for intercombinations between the two rare earth type energy level systems found in the spectrum of singly-ionized uranium (U II) [9] All previously known levels in this range were re-identified but no definite correlation link between Systems A and B has been established It is possible that System B (low electron configuration f^4s) may be lower than System A (low configuration f^3ds)

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* Research Participant University of Louisville Louisville Kentucky

8 Griffin P M R A Loring G K Werner and J R McNally Jr "Preliminary Design and Performance of a Fast High Resolution Echelle Spectrograph " presented at the Southeastern Section of the APS Raleigh North Carolina April 10-12 1952

9 See for example The Chemistry of Uranium Chapter II National Nuclear Energy Series McGraw Hill Book Company Inc New York 1951

Spectrochemistry (J A Norris M R Skidmore C E Pepper and Z Combs)

New rare earth standards have been prepared for analysis of gadolinium oxide raw materials and the calutron separated isotopes Standards for analysis of thoria have been prepared for the analytical range 80 ppm to 8000 ppm of rare earths preliminary results indicate a modified pyroelectric method is applicable Analysis of lithium as the sulfate using d c excitation has also proved promising

A total of 597 samples were analyzed for 9415 individual element determinations

X-Ray Spectroscopy (W F Peed H W Dunn and G L Rogosa*)

Power supply modifications of the XRD-3 x-ray unit have been completed A capacitor filter with current limiting resistors has been introduced to provide a stabilized d c high voltage for the x-ray tube and to minimize errors in correcting for counting losses The modified unit was tested by x-ray fluorescence of U_3O_8 for constituent Zr An absolute accuracy of ± 0 05% Zr to the 2 to 3% level was obtained

Approximately 185 samples were analyzed for compound identification in reactor loop studies and uranium-beryllium fluoride phase studies

The joint x-ray report with the Mound Laboratory on polonium has been completed and is in the process of reproduction

Reports and Papers in Preparation

1 H W Morgan and J H Goldstein "Microwave Spectrum of Vinyl Iodide "

2 P M Griffin and R A Loring "The Design and Performance of an Echelle Spectrograph "

3 W F Peed L E Burkhart R A Staniforth and L G Fauble Joint ORNL-Mound Laboratory report "The X-Ray Spectra of Polonium Atomic Number 84 "

4 G L Stukenbroeker and J R McNally Jr "Isotope Shifts in Thorium for Th230 and Th232 "

* Research Participant Florida State University Tallahassee Florida

Reports Submitted for Publication

1 J A Norris and C E Pepper "Quantitative Spectrochemical Analysis of Rare Earth Mixtures" to be published in Analytical Chemistry

2 E J Spitzer and D D Smith "Spectrochemical Analysis of Zirconium Oxide" to be published in Applied Spectroscopy August 1952

3 H E Walchli and H W Morgan "Magnetic Shielding Effects in Compounds of Vanadium" to be published in Physical Review August 1952

4 G K Werner "Semi-Automatic Recording of Wavelength Data" submitted to Review of Scientific Instruments

Reports Published and Papers Presented

1 G L Stukenbroeker D D Smith G K Werner and J R McNally Jr "Spectro-Isotopic Assay of Lithium" JOSA 42 383-6 (1952)

2 W F Peed and H W Dunn "Preliminary Investigation of Monochromatic X-Ray Absorption as a Method of Analysis for Uranium" ORNL-1265 (June 1952)

3 P M Griffin R A Loring G K Werner and J R McNally Jr "Preliminary Design and Performance of a Fast High Resolution Echelle Spectrograph" presented at the Southeastern Section of the APS, North Carolina State University Raleigh N C April 10-12 1952

4 H W Morgan and J H Goldstein "Microwave Spectrum of Vinyl Iodide" presented at the Southeastern Section of the APS North Carolina State University Raleigh N C April 10-12 1952

MASS SPECTROMETER LABORATORY

Russell Baldock Department Head

INTRODUCTION

During the fourth quarter of the fiscal year 1952 the remaining unidentified crystalline impurity isolated from fulinak cold legs was found to be KNa_2CrF_6 Much better data have been gathered in the study of uranium tetrafluoride The first blank has been examined in the new investigation of the stainless steel surface coating problem of interest in the homogeneous reactor experiment Tests have been conducted on structural parts for the new high temperature ion source for the mass spectrometer and its fabrication is progressing in addition extensive changes have been made in the electronic circuitry of the mass spectrometer which has resulted in marked improvements in its performance

REACTOR ASSOCIATED STUDIES

Fulinak (L O Gilpatrick)

The analysis of the pale green non-magnetic crystals has been completed The material was one of the contaminants found in the cold leg samples taken from stainless steel loops which failed by pluging The material proved to be KNa_2CrF Previously metallic iron and an alloy of chromium and iron had been identified

Uranium Tetrafluoride (L O Gilpatrick and J R Sites)

After considerable revision of the mass spectrometer a more refined study of the contamination of uranium tetrafluoride (UF_4) with higher valence compounds of uranium was started The present technique consists of continuously collecting data from a sample held at a constant temperature This approach has yielded much more consistent data than was previously available The fact has now been well established that the normal ratio of uranium tetrafluoride single plus $(UF_4)^+$ to uranium trifluoride single plus $(UF_3)^+$ lies close to 0 070 for pure UF_4 using 75 volt ionizing electrons and further that the disappearance of uranium pentafluoride as indicated by the above ratio

follows a regular course with time to yield smooth functions which indicate a definite end point for the production of uranium pentafluoride

Efforts are now under way to correlate the measured data with known sample impurities in order to work out a quantitative relationship

Stainless Steel (L O Gilpatrick and J R Sites)

Preliminary trials were made in the mass spectrometer of selected blanks of stainless steel as the first step in the eventual examination of the structure of the corrosion resisting coating formed by oxygen treatment of the metal surface This treatment has been of interest in the homogeneous reactor experiment

Hydrogen-Deuterium (J R Sites)

Using the amplidyne magnet supply and a conventional ion source two hydrogen-deuterium mixtures obtained from Dr Wethington of X-10 were run in the mass spectrometer The magnet coils were changed from the usual 12 coils in parallel to 4 inner coils in parallel and the two outer coils reversed so their magnetic fields would buck out that of the inner ones This loaded the amplidyne sufficiently in this low mass region and enabled the mass scanning to be done very slowly Different focus conditions were found for the masses 1 2 3 and 4 peaks A series of data was taken for the variables of the source and a best set of conditions was determined for all peaks Consistent results were not obtained possibly due to equilibration of hydrogen and deuterium on the source filament

A special gas tight source was fabricated to reduce this effect and a new inlet system was constructed using a Consolidated Engineering Corporation micromanometer to read the gas pressures in front of the leak valve The source proved inefficient probably because it did not incorporate an ion repeller plate No hydrogen or deuterium peaks were found even at high gas leak rates into the ion chamber

INSTRUMENTAL STUDIES

High Temperature Source (L V Wilson*)

Tests have been made on combinations of the metal surfaces molybdenum stainless steel and rhodium plate on stainless steel to determine to what

*Y-12 Engineering

extent they will weld under high temperature and vacuum conditions The combinations of rhodium plate and stainless steel proved to be satisfactory and fabrication of the high temperature source is proceeding The new unit is being readied for test in dry runs to determine its maximum temperature and general performance

MASS SPECTROMETER REVISION

Vibrating Reed Electrometer

The VX-41A electrometer circuit was replaced with an Applied Physics Laboratory vibrating reed electrometer which has scales of 1 2 5 10 20 50 100 200 500 and 1000 millivolts full scale It is very stable and has much smaller random noise than the previously used VX-41 circuit It has a usable sensitivity of about 10^{15} amperes of ion current to the collector plate in the receiver

Magnetic Field Supply

The amplidyne which supplied the magnet current was replaced by an electronic supply patterned after one designed by Shaffer at Brookhaven National Laboratory It is possible with this supply to remain on the side of a peak with very small random drift The built-in scanning device has some advantages over the old method but the spread in rates of scanning is small The complete mass spectrum can be scanned continuously any peak or group of peaks can be re-scanned and the settings are reproducible

Ionization Efficiency

A simple voltage divider circuit was designed for use in connection with an X-Y recorder to plot ionization efficiency curves automatically The potential difference between the filament and the ionization case was measured between two 250 ohm resistors connected at the lower end of two 2 5 megohm resistors The resulting curves were not of the correct shape possibly because the total current through one of the regulating resistors in the emission regulator was not linear with the electron acceleration voltage