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QUARTERLY PROGRESS REPORT

LABORATORY RECORDS 1954

STABLE ISOTOPE RESEARCH AND PRODUCTION DIVISION

January 1, 1952 to March 31, 1952

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OAK RIDGE NATIONAL LABORATORY
Y-12 AREA
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-1298
This document contains 46 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

January 1, 1952 to March 31, 1952

C. P. Keim, Director

J. R. Walton, Editor

April 1, 1952

[redacted]

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-1298
Isotope Separation

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Y-497 July 1, 1949 to September 30, 1949

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Y-821 July 1, 1951 to September 30, 1951

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STABLE ISOTOPE SEPARATION RESEARCH AND CALUTRON OPERATIONS

C. E. Normand, Department Head

INTRODUCTION

During the third quarter of fiscal year 1952, the Isotope Production and Process Development Department placed primary emphasis on the collection of calcium, lithium, molybdenum, lead, cerium, tin, and barium isotopes and an investigation of the feasibility of power regeneration as applied to calutron operations.

COLLECTIONS

Seven elements were processed and forty isotopically enriched samples were collected. These collection activities were accomplished 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.

Calcium, Series FO-XAX

The collection of calcium isotopes (Ca40, Ca42, Ca43, Ca44, Ca46, and Ca48) was started in November, 1951 in the Beta channels and was completed in February, 1952.

There were no major changes in equipment as reported in "Quarterly Progress Report, Stable Isotope Research and Production Division," ORNL-1228.

As a result of buckling of the stainless steel ion exit slit charge vapor leaks occurred. Carbon was substituted; however, these cracked and warped worse than the stainless steel slits. The deep floating anode ionization chamber was replaced by a standard type. The stainless steel slits

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performed well with this standard chamber.

The peak output attained in 2,121 hours of innage was 184.5 ma. The total monitored ion collection for the series was 220,941 ma hrs. The estimated total collection is as follows:

Ca40	322.80 gm
42	1.45 gm
43	0.50 gm
44	5.54 gm
46	0.03 gm
48	0.43 gm
Total	330.75 gm

Lithium, Series FI-XAX

Lithium isotope collections (Li6 and Li7) were completed during the month of January. There were no changes in the equipment as reported in "Progress Report, Stable Isotope Research and Production Division," Y-821.

Molybdenum, Series FP-XAX

Molybdenum isotope collections (Mo92, Mo94, Mo95, Mo96, Mo97, Mo98, and Mo100) were started in January, 1952 and completed during the first week of February, 1952.

Molybdenum pentachloride ($MoCl_5$), which vaporized at 75°-$100^{o}C$, was used as charge material in a low temperature source equipped with standard electrode geometries and a 3/16-inch carbon ion exit slit. Both the deep floating anode and standard ionization chambers were tried without appreciable difference in operation being noted.

Carbon pockets were placed behind 1/8-inch slots in a carbon defining faceplate. To aid in obtaining high purity, pockets for masses 95 and 97 ion beams were placed in contact with the faceplate.

Temperature control was difficult early in the run when high electrical drains caused overheating. Additional cooling aided in this problem but longer bake-out periods would have been necessary to completely overcome it.

Growths on the collimating slot were minimized by using a short slot in a standard ionization chamber.

The total monitored ion collection was 666 ma hrs during 138 hours of innage.

The estimated weights that were collected are as follows:

Mo92	0.23 gm
94	0.20 gm
95	0.34 gm
96	0.42 gm
97	0.21 gm
98	0.71 gm
100	0.28 gm
Total	2.39 gm

Lead, Series FG-XAX

The collection of lead isotopes (Pb204, Pb206, Pb207, and Pb208) started in February, 1952 and was completed during March, 1952. Nine runs were initiated using Alpha 2 sources with a modified Alpha 2 receiver located at the 96-inch position. The source was equipped with standard Alpha 2 electrodes and 1/8-inch carbon ion exit slits. Lead chloride ($PbCl_2$), the charge material, vaporized in the 300°-400°C temperature range.

Special copper receiver pockets were fabricated for these collections. Pockets for masses 206, 207, and 208 were soldered together in a single unit, leaving triangular water cooling passages between the pockets. Pocket walls were 1/32-inch thick and the defining slots in the carbon faceplate were 1/8-inch wide.

Water leaks and electrical shorts in the receiver and lead deposits forming on the ion exit slit were the main problems during these collections.

There were 144 hours of innage during which the total monitored ion collection was 8,794 ma hrs.

Estimated total collection was as follows:

Pb204	6.22 gm
206	14.69 gm
207	14.14 gm
208	32.87 gm
Total	67.92 gm

Cerium, Series FR-XBX

Cerium isotope collections (Ce136, Ce138, Ce140, and Ce142) were started in February, 1952 and completed in March, 1952. The high temperature source was used embodying stainless steel electrodes and a 3/16-inch carbon ion exit slit. During the later runs, carbon electrodes were used in place of stainless steel.

Cerous chloride ($CeCl_3$), the charge material, did not require an arc support gas after the operating temperature (~750°C) was reached.

Water-cooled copper pockets were placed behind 1/8-inch slots in the receiver defining faceplate.

Persistent sparking, characteristic of cerium operations, was prevalent throughout these collections. The accelerating electrodes and the ion exit slit became coated with a cerium deposit.

The estimated weights as determined from 18,904 ma hrs of monitored ion collection are as follows:

Ce136	0.37 gm
138	1.18 gm
140	87.62 gm
142	9.68 gm
Total	98.85 gm

Tin, Series FS-XAX

Tin isotopes (Sn112, Sn114, Sn115, Sn116, Sn117, Sn118, Sn119, Sn120, Sn122, and Sn124) were collected in the Alpha channels. The operations started in February, 1952 and are being continued.

The low temperature, double gas feed, source was used with a deep floating anode ionization chamber, standard zero degree electrodes and a 1/8-inch ion exit slit.

Stannic chloride ($SnCl_4$), which is liquid at room temperature, was admitted from an external charge container into the arc chamber, through a leak valve and a 3/8-inch charge feed line.

Carbon receiver pockets were placed behind 1/8-inch slots in the carbon defining faceplate.

The estimated weights that have been collected are as follows:

Sn112	0.43 gm
114	0.41 gm
115	0.45 gm
116	7.93 gm
117	4.62 gm
118	15.00 gm
119	6.11 gm
120	22.04 gm
122	3.34 gm
124	3.29 gm
Total	63.62 gm

Barium, Series FT-XBX

Collection of barium isotopes (Ba130, Ba132, Ba134, Ba135, Ba136, and Ba138) were started in March, 1952 in the Beta channels and are being continued.

The low temperature source was used with standard, zero degree accelerating electrodes. The standard ionization chamber was equipped with a 3/16-inch stainless steel ion exit slit. Carbon slits were used in place of stainless steel to improve beam focus; however, the carbon buckled and its use was discontinued. Source castings were corroded by barium at elevated temperatures; therefore, a high temperature source was placed in operation embodying standard accelerating electrodes and 3/16-inch, 15 degree, carbon ion exit slits. Operation improved but poor focus persisted after barium growths formed on the inside surface of the ion exit slit.

Space limitations between adjacent pockets made it necessary to employ two types of receivers. Mass 137 and mass 135 beams were rejected in one receiver and mass 136 and mass 134 were rejected in the other. The carbon pockets were placed behind 1/8-inch slots in the carbon defining faceplate.

The following weights were estimated from 7,260 ma hrs of monitored ion beams:

Ba130	0.08 gm
132	0.10 gm
134	0.99 gm
135	1.26 gm
136	3.50 gm
137	1.36 gm
138	29.95 gm
Total	37.24 gm

DEVELOPMENT

Power Regeneration (G. M. Banic, W. A. Bell, L. O. Love, and W. K. Prater)

The feasibility of "power regeneration" in connection with the collection of large ion currents in the calutron is being investigated. If the method is successful, power generated by retardation of ions at the receiver could be used to supplement that of existing power supplies, thereby increasing the capacity of these supplies and reducing overall power requirements. Moreover, retardation of ions would reduce wear, erosion, and heat dissipation at the receiver. Thus two present limitations on high rate isotope production--power supply and receiver--could be removed by successful power regeneration.

An experimental unit consisting of a standard Beta source and specially designed receiver mounted on a single Alpha faceplate has been constructed. The accelerating electrode support structure is placed beneath the source thereby permitting the receiver to be mounted directly above the source for collection at a minimum beam radius of 5 1/4 inches. Source and receiver components are supported from four insulating bushings passing through the faceplate (Figures 1 and 2). The source can be assembled using grid diode or standard Beta geometry. The receiver can be operated at ground potential or at various potentials above ground. Lithium has been used as charge material because of the favorable separation of its two low mass isotopes.

Two primary aims have been the attaining of high outputs with good resolution, and the development of a receiver slit system permitting maximum reception when a retarding potential is applied at the receiver.

Ion currents of approximately one-half ampere per square inch have been attained; however, only about half of this was received in a grounded pocket behind a 7/16-inch defining slot. Although no particular effort to obtain high purity has been made, it is encouraging that 99.97 per cent pure Li7 and 80 per cent pure Li6 have been recovered. Quantitative recovery of one collection gave a receiver retention figure of almost 100 per cent.

Experiments are being continued both to increase ion output and to improve reception with regeneration.

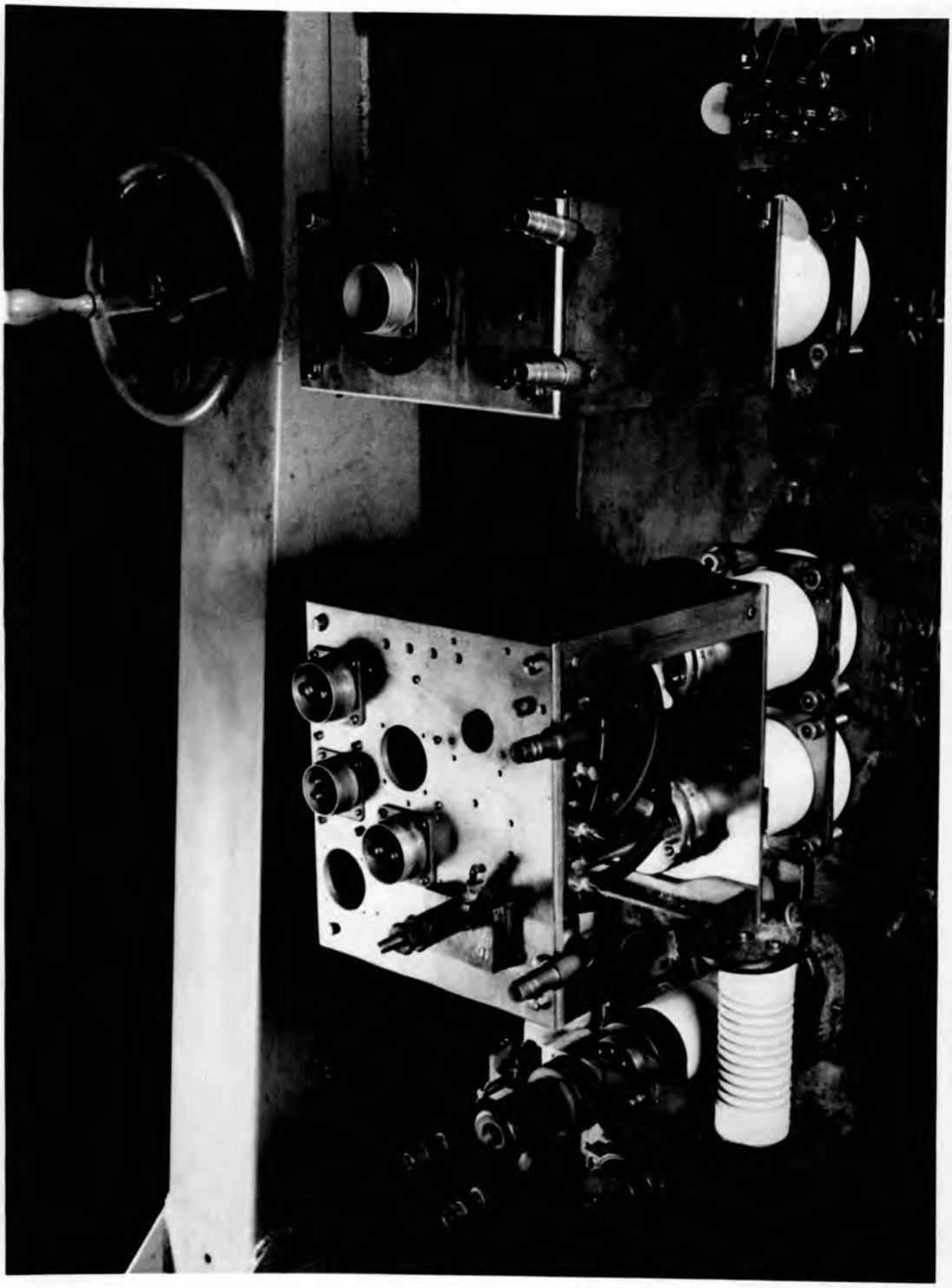


FIGURE I EXTERNAL VIEW OF POWER REGENERATION EQUIPMENT

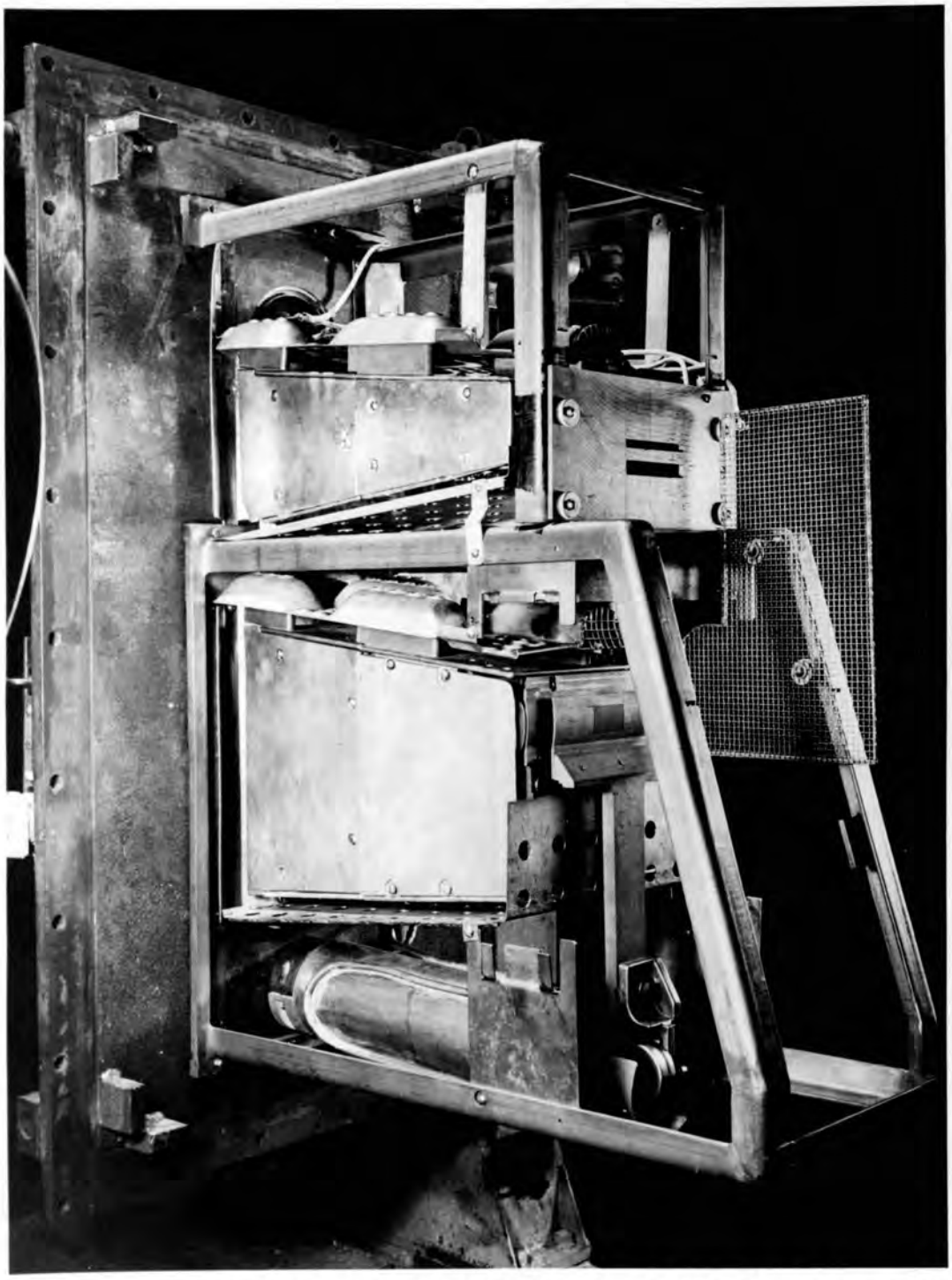


FIGURE 2 SOURCE AND RECEIVER OF POWER REGENERATION EQUIPMENT

Reports Issued

1. L. O. Love and W. A. Bell, "The Enrichment of Lithium 6 by The Electromagnetic Process," ORNL-1262.

Reports in Preparation

1. "Stable Isotope Separations in Y-12."

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 sample on which refinement was completed	21
Samples in process of chemical refinement, March 31	14
Samples awaiting chemical refinement, March 31	14
Samples submitted to Mass Analysis Laboratory	45
Mass analyses reported	28
Samples awaiting mass analysis, March 31	26
Stable Isotope shipments made	83

PRODUCTION OF CHARGE MATERIALS

Preparation and Purification (C. W. Sheridan)

Charge materials of lithium, calcium, lead, molybdenum, cerium, tin, and barium were prepared for calutron operations. Various chemical services were also supplied in those operations.

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 described in a special report.

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

Work was done on the chemical refinement of isotopes of eight elements: iron, calcium, lead, lithium, molybdenum, cerium, nickel, and strontium. No series of isotopes were completed in which new processes or techniques were involved.

ACTIVITIES ASSOCIATED WITH MASS ANALYSIS

Compound Preparation (F. M. Scheitlin)

Isotopes of seven elements were prepared in suitable form for mass analysis: iron, lead, calcium, strontium, lithium, molybdenum, and neodymium.

SEPARATION OF RARE EARTHS

Extraction from Nitric Acid by Tributyl Phosphate (F. A. Kappelmann)

Further progress was made toward obtaining gadolinium from a concentrate by operation of a pseudo-countercurrent batch process.

ISOTOPE SHIPMENTS AND REQUESTS

Preparation for Shipment

Eighty-three 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 a nominal charge of $50.00 made to most users of stable isotopes for individual shipments, to defray clerical and shipping costs only, the following summary has been compiled:

	October 1 to December 31, 1951	January 1 to March 31, 1952
Transfers within AEC	$ 2,250.00	$ 2,600.00
Cash Sales (fees)	1,600.00	1,550.00
Total Sales (fees) and Transfers to Date	$70,000.00	$74,150.00

TABLE I

Shipments of Stable Isotopes during January, February and March, 1952

Request Serial No.	Requestor	Isotope	Series No.	Sample No.	Quantity Shipped (mg)	Date Shipped
B-2290	Ohio State University	Ca48	FF	653(a)	50.0	1-4-52
B-2291	Ohio State University	Fe57	DL	463(j)	2,000.0	1-4-52
B-2292	Ohio State University	Fe58	DL	454(f)	150.0	1-4-52
B-2420S	Oak Ridge National Laboratory	Ca44	DI	444(a)	300.5	1-8-52
B-2323	University of Pittsburgh	Cu63	DU	497(a)	1,000.4	1-9-52
B-2324	University of Pittsburgh	Cu65	DU	498(a)	1,000.2	1-9-52
B-2388	Ohio State University	In113	ER	594(a)	500.8	1-14-52
B-2393	Ohio State University	Sr84	Z	78(ar)	200.2	1-14-52
B-2422S	Oak Ridge National Laboratory	Mo100	R-T	59(ar)	25.2	1-15-52
B-2421S	Oak Ridge National Laboratory	Mo98	R-T	58(a)	100.8	1-15-52
B-1603	Ohio State University	Sm144	FH	660(a)	50.1	1-16-52
B-1604	Ohio State University	Sm147	FH	661(a)	50.8	1-16-52
B-849	Ohio State University	Ca48	FF	653(a)	10.1	1-16-52
B-2397S	Argonne National Laboratory	Ga69	EI	554(a)	50.4	1-17-52
B-2398S	Argonne National Laboratory	Ga71	EI	555(a)	50.3	1-17-52
B-2399S	Argonne National Laboratory	Ge70	BD-BE	205-210(a)	50.6	1-17-52
B-2400S	Argonne National Laboratory	Ge74	BD-BE	208-213(a)	50.5	1-17-52
B-2402S	Argonne National Laboratory	Ge73	BD-BE	207-212(ar)	51.0	1-17-52
B-2403S	Argonne National Laboratory	Ge72	BD-BE	206-211(a)	50.2	1-17-52
B-2376	Washington Univ. School of Med.	Fe58	DO	480(a)	10.1	1-18-52
B-2458S	Oak Ridge National Laboratory	Sm147	FH	661(a)	400.5	1-22-52
B-2459S	Oak Ridge National Laboratory	Sm149	FH	663(a)	400.4	1-22-52
B-2460S	Oak Ridge National Laboratory	Sm154	FH	666(a)	400.6	1-22-52
B-2391	University of Illinois	Hg202	DR	490(a)	22.9	1-22-52
B-2383	University of Illinois	Hg200	DR	488(a)	21.6	1-22-52
B-2389	University of Illinois	Hg204	DR	491(b)	27.2	1-22-52
B-2455	Massachusetts Institute of Tech.	Zn70	EK	560(a)	150.2	1-23-52
B-2409	University of Illinois	Zr90	EE	539(a)	500.4	1-28-52
B-2439	Princeton University	Li6	FI	667(a)	50.0	1-28-52
B-2414	Ohio State University	Zn64	EK	556(a)	300.5	1-28-52
B-2413	Ohio State University	Ni58	AS-AT	141-147(a)	300.2	1-28-52
B-1601S	Brookhaven National Laboratory	V50	FM	653(b)	40.6	2-5-52
B-1191	University of Wisconsin	V50	FM	683(b)	100.5	2-5-52
B-2450S	Argonne National Laboratory	Fe54	FC	634(a)	1,001.2	2-6-52
B-2451S	Argonne National Laboratory	Fe56	CZ	404(f)	1,001.6	2-6-52
B-2445	University of Illinois	Zr92	CK	336(a)	100.4	2-7-52
B-2446	University of Illinois	Zr94	CK	337(ar)	100.1	2-7-52
B-2444	University of Illinois	Zr91	CK	335(ar)	100.7	2-7-52
B-2457S	Brookhaven National Laboratory	In115	ER	595(a)	10,001.4	2-7-52
B-2456S	Brookhaven National Laboratory	In113	ER	594(a)	700.8	2-7-52
B-2465S	Oak Ridge National Laboratory	Si30	AI-AJ	105-108(a)	20.2	2-8-52
B-2466S	Oak Ridge National Laboratory	S33	CX	392(b)	30.3	2-8-52
B-2471S	Oak Ridge National Laboratory	Nd146	EQ	591(a)	2.4	2-8-52
B-2472S	Oak Ridge National Laboratory	Nd142	EQ	597(a)	2.3	2-8-52
B-2473S	Oak Ridge National Laboratory	Nd143	EQ	588(a)	2.4	2-8-52
B-2474S	Oak Ridge National Laboratory	Nd144	EQ	589(a)	2.3	2-8-52
B-2475S	Oak Ridge National Laboratory	Nd145	EQ	590(a)	2.4	2-8-52

Request Serial No.	Requestor	Isotope	Series No.	Sample No.	Quantity Shipped (mg)	Date Shipped
B-2476S	Oak Ridge National Laboratory	Nd148	EQ	592(a)	2.5	2-8-52
B-2477S	Oak Ridge National Laboratory	Nd150	EQ	593(a)	2.4	2-8-52
B-2503S	Oak Ridge National Laboratory	Sn112	BI	227(a)	10.2	2-15-52
B-2489	Stanford University	Zn64	EK	556(a)	400.6	2-20-52
B-2478	Columbia University	K41	EX	623(e)	1,002.6	2-20-52
B-2405	Kansas State College	Sm144	FH	660(a)	32.8	2-21-52
B-2502S	Oak Ridge Institute of Nuclear Studies	Ti50	EN	580(a)	100.1	2-22-52
B-2507S	Brookhaven National Laboratory	Sm144	FH	660(a)	25.4	2-22-52
B-2508S	Brookhaven National Laboratory	Sm147	FH	661(a)	30.9	2-22-52
B-2509S	Brookhaven National Laboratory	Sm148	EM	570(a)	50.8	2-22-52
B-2510S	Brookhaven National Laboratory	Sm149	FH	663(a)	45.2	2-22-52
B-2511S	Brookhaven National Laboratory	Sm150	EM	572(a)	50.5	2-22-52
B-2512S	Brookhaven National Laboratory	SM152	EM	573(a)	50.1	2-22-52
B-2513S	Brookhaven National Laboratory	SM154	EM	574(a)	50.5	2-22-52
B-2492	Yale University	Si29	AI-AJ	104-107(a)	50.0	2-29-52
B-2432	University of California	Mo95	FP	697(b)	30.5	3-5-52
B-2433	University of California	Mo97	FP	699(b)	30.0	3-5-52
B-2525	Mass. Institute of Technology	Cr53	EU	611(a)	31.3	3-7-52
B-2559S	Oak Ridge National Laboratory	In113	BH	225(a)	584.1	3-11-52
B-2560S	Oak Ridge National Laboratory	In115	CQ	359(a)	12,379.9	3-11-52
B-2531S	Oak Ridge National Laboratory	Sm152	FH	665(a)	4,776.8	3-19-52
B-2532S	Oak Ridge National Laboratory	Sm154	FH	666(a)	2,209.0	3-19-52
B-2533S	Oak Ridge National Laboratory	Ce138	DW	502(a)	11.1	3-19-52
B-2534S	Oak Ridge National Laboratory	Hf174	ET	602(a)	10.3	3-19-52
B-2535S	Oak Ridge National Laboratory	Hf180	ET	607(a)	10.9	3-19-52
B-2565S	Iowa State College, Ames Lab.	Cr50	EU	609(a)	50.6	3-19-52
B-2352S	Univ. of Cal. Radiation Lab.	Hg204	DR	491(a)	62.9	3-25-52
B-2588	Carnegie Institution of Wash.	Ca44	CS	373(a)	30.6	3-27-52

Unfilled Requests on Hand March 31, 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):

Request Number	Date	Requestor	Isotope	Quantity Desired (mg)
S-324	3-2-49	Oak Ridge National Laboratory	Cl37	3,000
S-328	3-2-49	Oak Ridge National Laboratory	B11	10,000

Requests awaiting Purchase Orders:

B-2035	6-26-51	Mass. Institute of Technology	K40	15
B-2447	1-7-52	University of Illinois	Zr96	50
B-2499	2-19-52	University of Pennsylvania	Sb123	600
B-2500	2-6-52	University of Pennsylvania	Sn116	20
B-2514	2-12-52	Florida State University	W184	50
B-2515	2-12-52	Florida State University	W186	50
B-2551	2-27-52	University of Michigan	Cd108	100
B-2564	3-3-52	General Electric Res. Laboratory	Mo96	500
B-2567	2-28-52	University of California	Ti47	900
B-2568	2-28-52	University of California	Ti49	412
B-2572S	2-26-52	University of California	Cr52	100
B-2580S	3-11-52	Argonne National Laboratory	Mo92	600
B-2582S	3-11-52	Argonne National Laboratory	Mo95	600
B-2583S	3-11-52	Argonne National Laboratory	Mo96	600
B-2584S	3-11-52	Argonne National Laboratory	Mo97	600
B-2585S	3-11-52	Argonne National Laboratory	Mo98	600
B-2598	3-10-52	Washington University	Ti48	200
B-2597	3-10-52	Washington University	Cr52	200
B-2596	3-10-52	Washington University	Fe56	200
B-2593	3-10-52	Washington University	Zn64	200
B-2594	3-10-52	Washington University	Zn66	200
B-2595	3-10-52	Washington University	Zn68	200
B-2591	3-10-52	Washington University	Ni58	200
B-2592	3-10-52	Washington University	Ni60	200
B-2589	3-10-52	Washington University	Ge72	200
B-2590	3-10-52	Washington University	Ge74	200

Requests with Purchase Order awaiting replenishment of inventory:

B-269	3-17-48	Princeton University	B11	500

Requests held by AEC Isotopes Division:

S-361		Argonne National Laboratory	Os192	10
S-483		Brookhaven National Laboratory	Ba132	100

Request Number	Requestor	Isotope	Quantity Desired (mg)
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 Med. Research Laboratory	Zn66	3,000
B-1709S	Argonne National Laboratory	Ni64	300
B-1819	University of Illinois	Ce136	10
B-1915S	Brookhaven National Laboratory	Ca46	25
B-2007S	Brookhaven National Laboratory	Ba132	10
B-233	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-2447	University of Illinois	Zr96	50
B-2577S	University of California	Cd152	10
B-2581S	Argonne National Laboratory	Mo94	600
B-2586S	Argonne National Laboratory	Mo100	600

ISOTOPE ANALYSIS METHODS LABORATORY

J. R. McNally, Jr., Department Head

INTRODUCTION

The work reported by this department pertains to spectroscopic investigations of elements and their isotopes in their electromagnetic spectra in the range from nuclear resonances of wavelength 10^4 cm down to X-rays of wavelength 10^{-9} cm. Specific fields include nuclear resonances, microwaves and infrared, optical spectroscopy, and X-rays.

NUCLEAR RESONANCE

Chemical Shifts in Vanadium and Copper Resonances (H. E. Walchli)

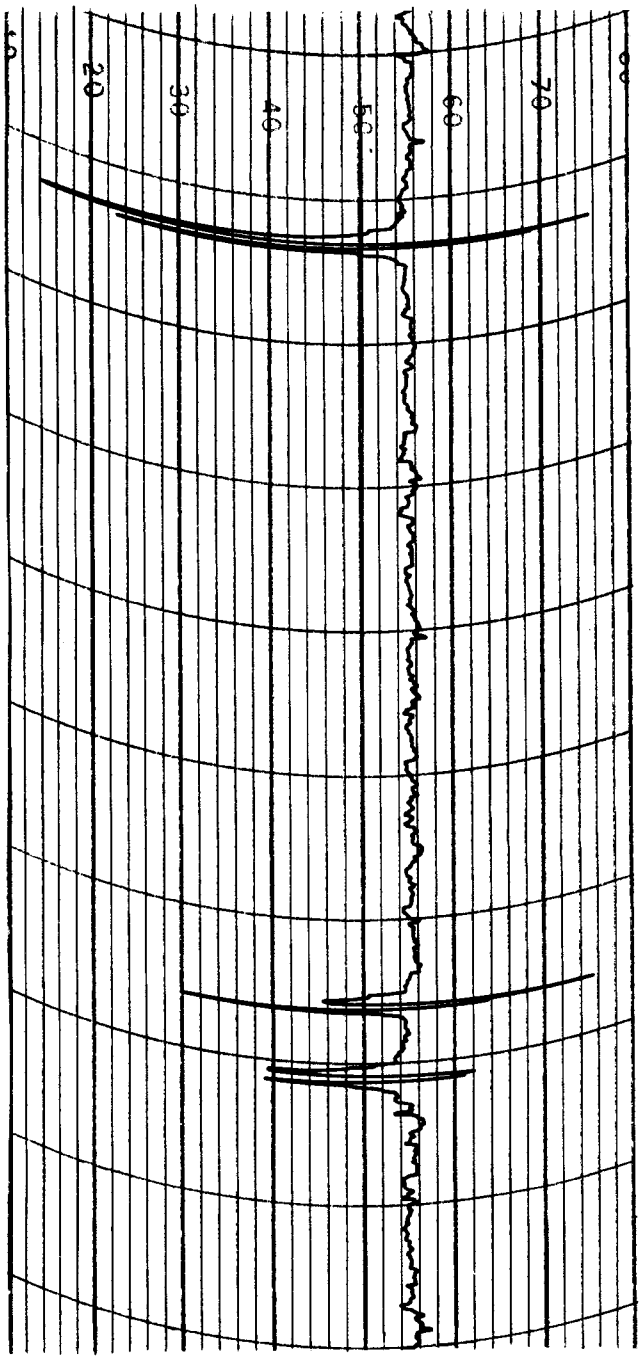
Earlier measurements of the characteristic nuclear magnetic resonance transitions in several vanadium compounds indicated the existence of pronounced chemical shift effects. These shifts have now been investigated more thoroughly and data are given in Tables II and III. The $_{23}V^{50}$ resonance frequency in vanadium oxytrichloride ($VOCl_3$) and its probable nuclear magnetic moment have been reported previously.[1] Line widths are narrow (less than 0.5 gauss) except for metallic V51 which gave 8 gauss half-width. This latter value is smaller than that given by Knight.[2] A frequency scan at a nominal magnetic field of 6741 gauss is given in Figure 3 for sodium metavanadate ($NaVO_3$) and $VOCl_3$.

TABLE II

Chemical Shifts in Nuclear Magnetic Resonances for $_{23}V^{50}$ and $_{23}V^{51}$

Isotope	Reference Isotope	Compounds	Frequency Ratio
V51	Na23	$NaVO_3$	.99386 ± .00002
		Na_3VO_4 NH_4VO_3	.99381 ± .00005
V51	Na23	$VOCl_3$	.99437 ± .00005
V51	Na23	V51 metal	.99996 ± .00001

1. See Quarterly Report ORNL-1228 and ORNL-1239; also, Physical Review, 85, 922-3L (1952).
2. Knight, W. D., Paper K-5, American Physical Society Meeting, Berkeley, California (December, 1951).



Na^{23} RESONANCE
7592.77 KC

V^{51} RESONANCE IN
$VOCl_3$ 7550.00 KC

V^{51} RESONANCE
IN Na VO_3

CHEMICAL SHIFT IN V^{51} NUCLEAR MAGNETIC
RESONANCES H = 6741 GAUSS
FIGURE 3

Chemical shifts have also been observed in the nuclear magnetic resonances of the copper isotopes. The percentage shift is the same for both copper isotopes indicating an extra-nuclear origin, which is probably of a chemical nature, for the shifts. Dickinson[3] discusses the contribution to such shifts of electron magnetic shielding effects, paramagnetic ion effects, bulk diamagnetism, electronic paramagnetic effects in metals, and others.

TABLE III

Chemical Shifts in Nuclear Magnetic Resonances for $_{29}Cu^{63}$ and $_{29}Cu^{65}$

Isotope	Reference Isotope	Compounds	Frequency Ratio
Cu63	Na23	Cu Metal/NaCl	1.00438
Cu63	Na23	CuCl, Cu_2Cl_2/NaBr	1.0020
Cu65	Na23	Cu Metal/NaCl	1.0759
Cu65	Na23	CuCl, Cu_2Cl_2/NaBr	1.0734

Miscellaneous Studies (H. E. Walchli)

Numerous additional nuclear magnetic resonances have been detected and measured. That for In115 was observed in $In(NO_3)_2$, but measurements have been postponed due to the poor signal to noise ratio. Measurements on In115 will be made later using the magnetic scanning method. Preliminary searches for resonances of Si29, Te123, Ga69, and Ga71 were made, but no signals were detected, presumably because of insufficient sample and the fast scanning rate used.

U235 as uranium hexafluoride (UF_6) was studied as the solid but with negative results. An oven has been constructed and tested to over 100°C. This oven is designed to hold the sample of UF_6 at just above its melting point (65°C). Further searches for the U235 nuclear resonance are being performed.

Final results on several isotopes are given in Table IV. Nuclear magnetic moments may be readily calculated from these frequency ratios[4] and tabulated values of nuclear spins and diamagnetic corrections; however, chemical shift corrections should be applied and the exact form of this correction is not as yet determined.

3. Dickinson, W. C., Phys. Rev., 81, 717 (1951).
4. See, for example, Walchli, H. E. and Ralph Livingston, "The Nuclear Magnetic Moment of Tc99," Phys. Rev., 85, 479L (1952).

TABLE IV

Nuclear Magnetic Resonances

Isotope	Reference Isotope	Compounds	Frequency Ratio
Li6	D2	$LiCl/D_2O$	0.9586
Li7	Na23	LiCl/NaCl	1.4692
F19	H	Aqueous HF	0.9408
Al27	Na23	$AlCl_3/NaBr+MnSO_4$	0.98509
P31	Li7	$H_3PO_4/LiCl$	1.0416
Ga69	Na23	$GaCl_3/NaCl$	0.9073
Ga71	Na23	$GaCl_3/NaCl$	1.1529
Br79	Na23	NaBr	0.94714
Br81	Na23	NaBr	1.02098
Rb87	Na23	RbCl/NaCl	1.2370
Cs133	D2	$CsNO_3/D_2O$	0.85448
Tl203	Tl205	$Tl(Ac)_2$	1.0098

MICROWAVE SPECTROSCOPY

Vinyl Iodide Microwave Spectrum (H. W. Morgan and J. H. Goldstein*)

The J=3 to 4 and the J=4 to 5 rotational transitions of the "slightly" asymmetric rotor, vinyl iodide, have been observed in the microwave regions near 25,000 mc and 32,000 mc, respectively. Four hyperfine patterns, corresponding to K=1, $\Delta K=0$ for a symmetric top, were chosen for accurate frequency measurements, and from these the parameters of the molecule were evaluated by application of second order asymmetric top theory.

Vinyl iodide is near prolate symmetric rotor with a $\kappa = -0.9921$ as determined in this work. The molecule is planar with the iodine nucleus lying on the axis of least moment, close to the center of gravity. Electron diffraction data allowed rough calculation of the moments of inertia and the absorption frequencies.

The absorptions corresponding to $\Delta K=0$ for the transitions observed are split apart, due to the asymmetry, and are spread over a 1000 mc region. Coupling between the large quadrupole moment of the iodine nucleus and the molecular rotation produces, for each $\Delta K=0$ case, a hyperfine pattern of some 11 observable lines covering some 200 mc. The splittings are such that the four patterns corresponding to K=1, $\Delta K=0$ lie well apart from the other multiplets and are easily studied, while all other patterns (K=1, $\Delta K=0$) lie superimposed. Attempts to resolve and measure these other patterns were unsuccessful.

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* Consultant, Emory University.

The four hyperfine patterns studied are identified by $J=3_{12}$ to 4_{13}, 3_{13} to 4_{14}, 4_{13} to 5_{14}, and 4_{14} to 5_{15}. In each pattern, the temperature dependence of all lines was checked to exclude lines due to high J levels and to vibrational states. The Stark effect was employed in assigning the proper F to F' for each line. This is illustrated in Figure 4 in which the strongest line in the 3_{13} to 4_{14} pattern is assigned F=11/2 to 13/2. For intensity comparison, the HDO line is also shown (natural abundance).

The quadrupole perturbation calculations[5] necessitated the computation of the necessary matrix elements for second order theory. The hyperfine pattern is dependent, in first order theory, upon the parameters χ_{aa} and χ_{bb}, components of the quadrupole coupling of the iodine, along the inertial axes a and b. In second order theory, it is dependent also upon χ_{ab} and the degeneracies which may exist. Approximate values of χ_{aa} and χ_{bb} were obtained from first order theory and used to obtain χ_{ab} and the second order corrections. From the corrected frequencies, χ_{aa} and χ_{bb} were re-evaluated. Thus, the zero order frequencies, moments of inertia, and asymmetry parameter were obtained.

Typical patterns, for the 3_{12} to 4_{13} and 4_{13} to 5_{14}, are shown in Figures 5 and 6. It should be noted that the 3_{12}-4_{13} drawing omits one line lying at 25.4 kmc, which was obscured by other overlapping patterns and could not be accurately measured. The relative intensities shown are those for the symmetric top case; the agreement with the observed intensities is found to be excellent. Figure 7 illustrates the contribution of second-order effects.

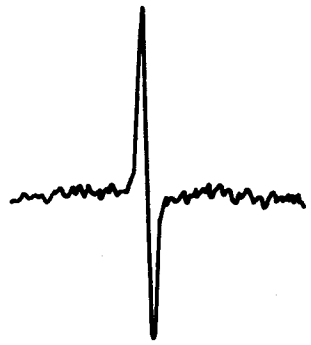
The parameters calculated for this molecule are given in Table V. χ_{zz}, obtained by a transformation to an axis system in which the z axis lies along the C-I bond, gives the coupling coefficient along the bond.

TABLE V

MOLECULAR PARAMETERS — VINYL IODIDE

A	3067.13 mc
B	52296.96 mc
κ	-0.9921
χ_{aa}	-1646 mc
χ_{bb}	+ 745 mc
χ_{ab}	- 757 mc
χ_{zz}	-1154 mc

5. Bragg, J. K., Phys. Rev., 74, 533 (1948).



HDO 22307 mc

(natural abundance)

RESOLUTION OF THE SIX

STARK COMPONENTS FROM THE

$J = 3_{13} - 4_{14}$, $F = 11/2 - 13/2$

TRANSITION AT 24942.47 MC

(approx. 850 volts/cm)

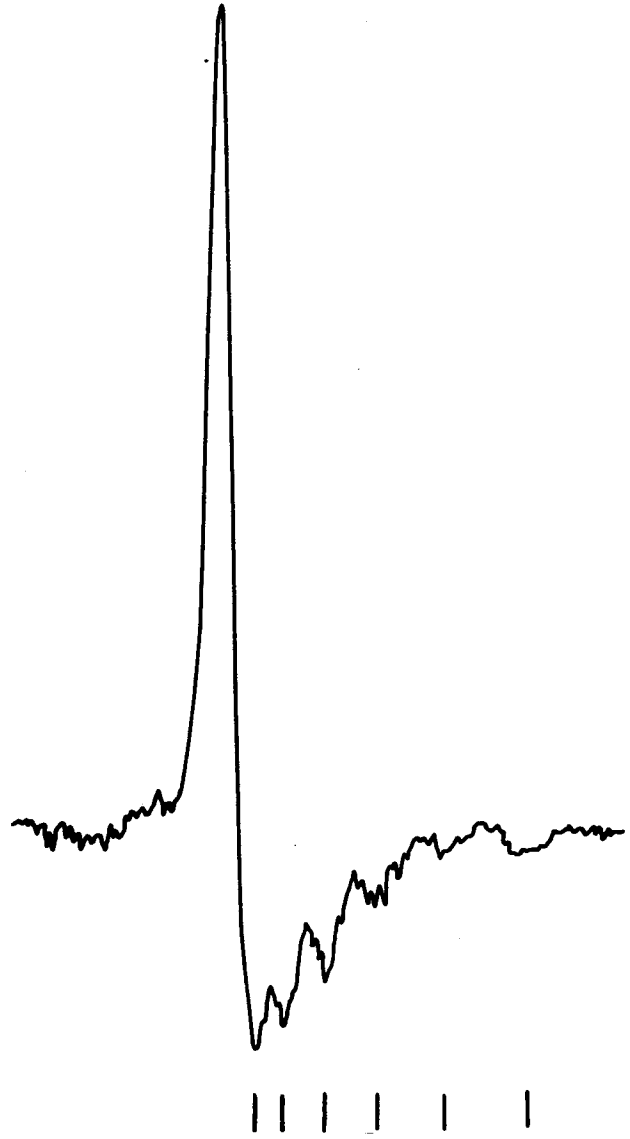
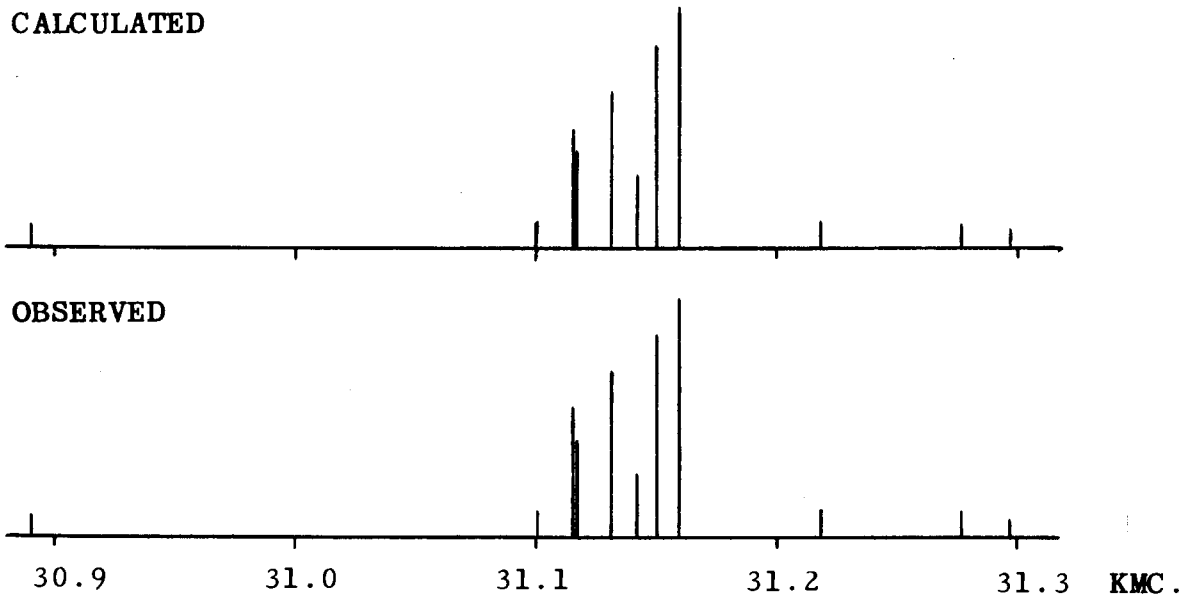
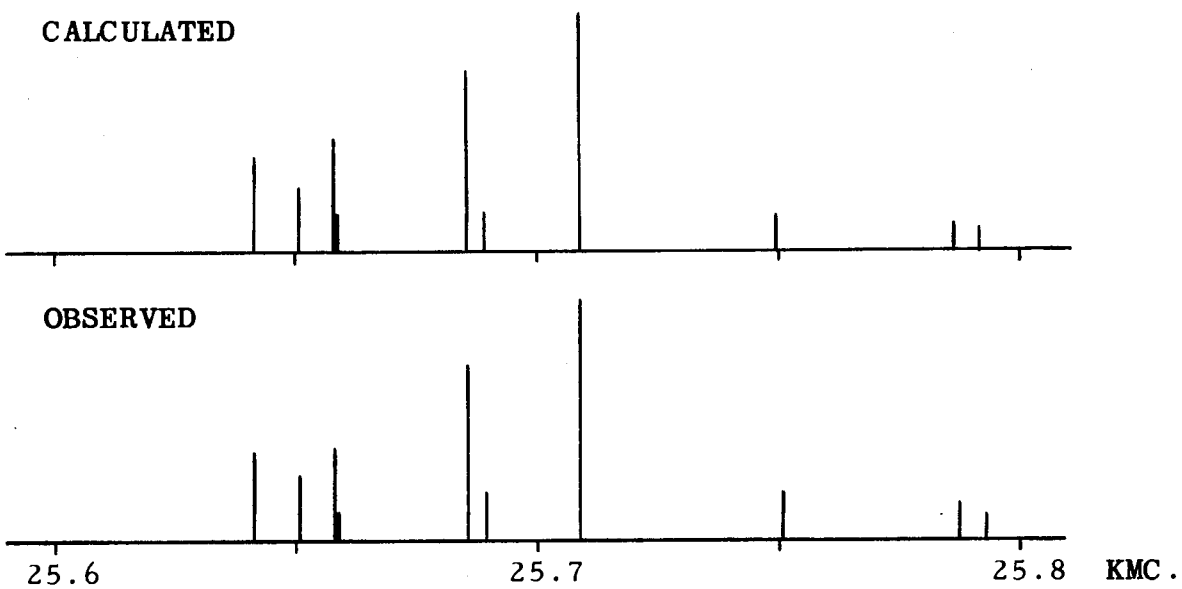


FIGURE 4 4 KC SQUARE WAVE RECORDING SYSTEM



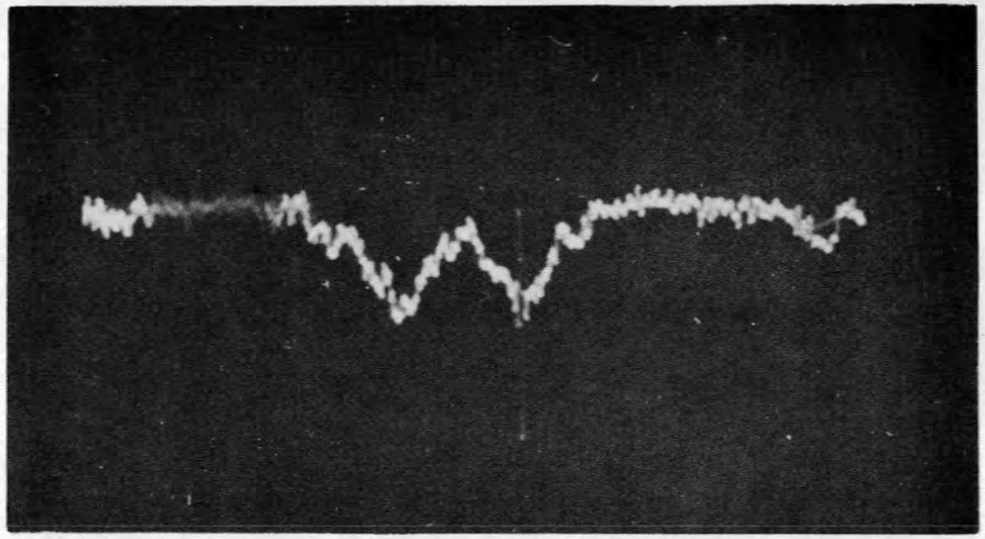
VINYL IODIDE $4_{14} - 5_{15}$

FIGURE 5



VINYL IODIDE $3_{12} - 4_{13}$

FIGURE 6



RESOLUTION OF THE $J = 3_{12} - 4_{13}$ TRANSITIONS

$F = 9/2 - 9/2$	25657.89 mc
$F = 7/2 - 9/2$	25657.34 mc

THE 3_{12} LEVELS ARE DEGENERATE IN FIRST ORDER THEORY,

BUT ARE SPLIT BY 0.52 MC IN SECOND ORDER THEORY.

FIGURE 7 100 KC SQUARE WAVE VISUAL SYSTEM

OPTICAL SPECTROSCOPY

Echelle Spectrograph (P. M. Griffin, R. A. Loring*, and G. K. Werner)

An external mounting for the 1200-line, 6-inch echelle[6] has been completed using a 183.9 cm focal length concave mirror (see Figure 8). Light from a source enters the horizontal slit and is collimated by the 8-inch diameter mirror onto the echelle; the diffracted beam of concentrated energy is then reflected from the mirror via a small reflecting mirror onto the vertical slit of a Littrow spectrograph. The horizontal dispersion is that of the Littrow prism-lens arrangement, namely, about 10 angstroms per millimeter at 5000 A; dispersion in the vertical direction is that of the echelle spectrograph, namely, $\frac{\lambda}{4f}$ or about 0.68 A/mm at 5000 A. The approximate order of interference with the echelle mounted at an angle of 63° is $2t/\lambda$ or about 450 at wavelength 5000 A. Theoretical resolution is then 450 times the number of lines (1200 or about 550,000). Actual resolution of the weak components of the mercury line 4358 A (Figure 9) is of the order of 300,000 giving spectrum purity of about 55 per cent for the echelle.

Zeeman Studies (P. M. Griffin and R. A. Loring*)

Zeeman spectra have been obtained for uranium, sodium, copper and zinc. The magnetic field has been measured from splitting of the sodium D lines at 25,400 ± 400 gauss. This includes a 100 gauss correction due to the Lande g factor error because of the anomalous magnetic moment of the electron.[7] Paschen-Back effects of the D lines are negligible in this calculation. Zeeman effects in uranium for lines at 4372 A are shown in Figure 10.

Classification of the energy level $^5L_6^0$ arising from the configuration f^3d^2 in the ionized uranium atom was made possible from some of this new Zeeman data on uranium. Previous IBM tabulations[8] had indicated that this level at 10740.96 cm^{-1} was possibly a real level, but no certain identification, especially of its quantum character, was available until now. The unusual existence of two, as yet unconnected, systems of energy levels in the spectra of U II is not yet explained (see Table VI). Van den Bosch has

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* Research Participant, University of Louisville.

6. Manufactured by Bausch and Lomb Optical Company, Rochester, New York.
7. McNally, J. R., Jr., Phys. Rev. 73, 1130 (1948).
8. McNally, J. R., Jr., unpublished data; see, also, J. R. McNally, Jr. and G. R. Harrison, Y-340, Feb., 1949.

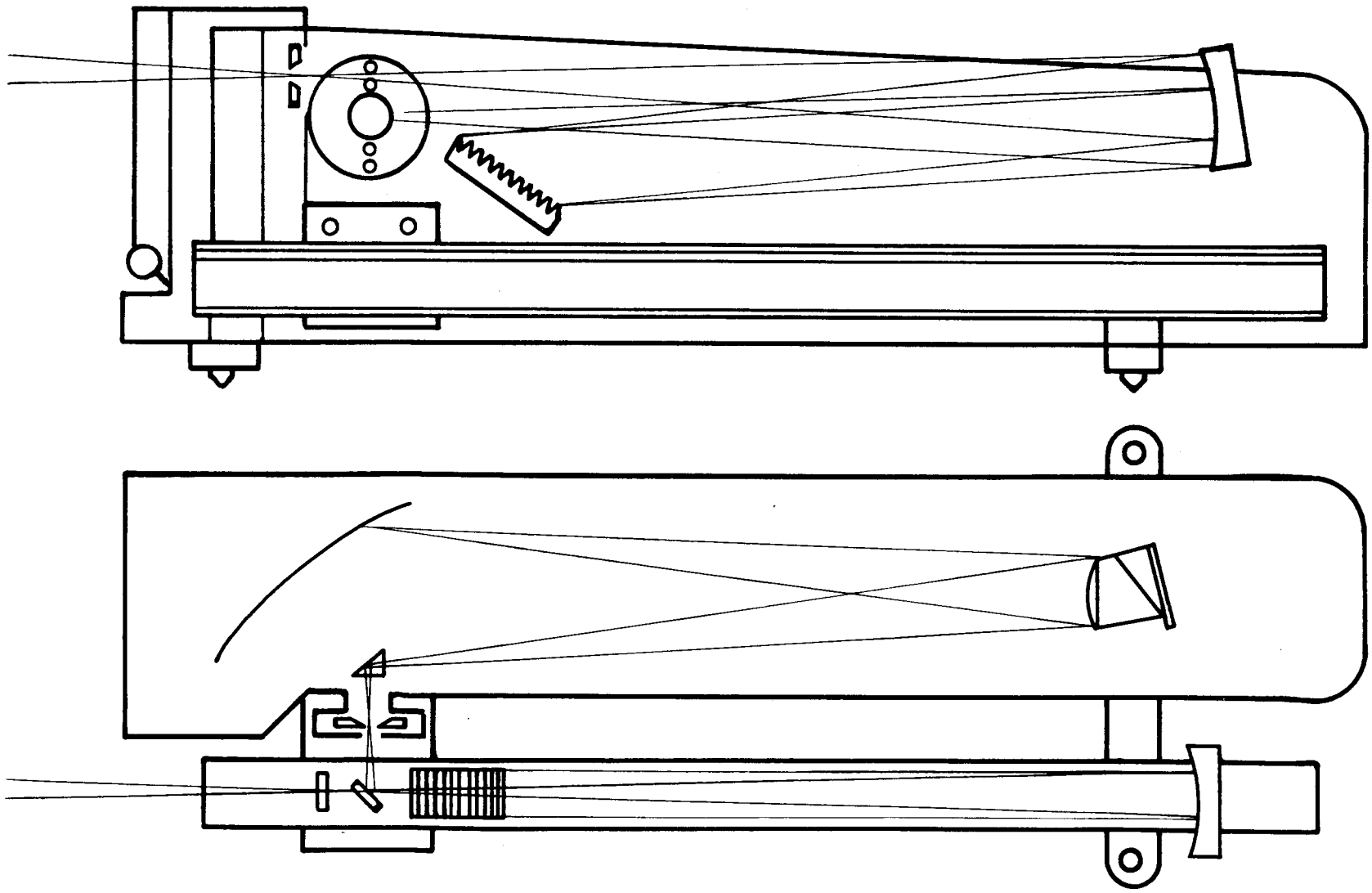

FIGURE 8 OPTICAL SYSTEM OF ECHELLE SPECTROGRAPH

FIGURE 9 HYPERFINE STRUCTURE OF THE 4358 A LINE OF Hg. "ECHELLOGRAM"

4372.572 4373.407

URANIUM PATTERNS

FIGURE 10

sought for energy differences of the order of x = -20,000 cm^{-1} between group A and B, whereas others conclude quite differently.[9] It is quite possible that Zeeman studies in the infrared region of the spectrum may resolve this problem. Preliminary work up to about 7000 A has given new data supplementing that obtained at the Massachusetts Institute of Technology with the high field magnet and large grating spectrographs.

TABLE VI

Comparison of Lowest Levels from Configurations of U II and Th II

	U II		Th II	
	Configuration	Lowest Level	Configuration	Lowest Level
Group A	$5f^3 7s^2$	0	$6d7s^2$	0
	$5f^3 6d7s^4$	289	$6d^2 7s$	1860
	$5f^3 6d^2$	4585	$6d^3$	7001
Group B	$5f^4 7s$	0 + x	$5f7s^2$	4490
	$5f^4 6d$	7850 + x	5f6d7s	6168
			$5f6d^2$	12,486

Photographic Assay (D. D. Smith and G. L. Stukenbroeker)

The assay procedure for isotopes of lithium has been modified to 30 ma hollow cathode operation with improvement in precision. Since photographic errors of only one per cent contribute a three per cent assay error, it has been necessary to standardize closely the processing of all photographic plates. Preliminary results indicate that these photographic processing errors are now of the order of one per cent.

A total of 106 unknown samples and 44 unknown standards has been analyzed during this quarter. Precision on the latter was $\sigma \cong 10$ per cent of the isotope abundance ratio.

Several sector steps are now in use in order to permit evaluation of lithium abundances over a wide range of isotope ratios. At present, assays can be performed only in the range 5 per cent to 95 per cent. Precision of abundance ratios is now estimated at $\sigma = 5$ per cent.

Photoelectric Spectro-Isotopy (G. K. Werner and O. B. Rudolph)

Phototube tests on various tube types indicated that the relative sensitivity was in the order 1P21, 1P28, 931A, and 1P22. No direct correlation

9. See Chapter II, The Chemistry of Uranium, National Nuclear Energy Series, Division VIII, Vol. 5 (1951).

between dark current and sensitivity existed. Dark current reduction and a sensitivity gain were obtained on three tubes baked at 100°C for three days. Tests on a "snooperscope" image converter showed no promise for assay purposes.

Direct reading photoelectric units were received from Baird Associates. Considerable testing and adjusting were involved prior to any attempts to measure light intensities. The direct reader was then set up to integrate photocurrents of 6707 A light from an incandescent tungsten filament source. Individual ratios of integrated photocurrents on two tubes separated by 0.15 A were found to be reproducible to $\sigma = 0.33$ per cent.

SPECTROCHEMISTRY

Alkali Studies (M. R. Skidmore, E. J. Spitzer, and J. A. Norris)

Individual alkali elements and sodium-potassium mixtures have been added as buffering agents to C standards (semi-quantitative analysis standards. Suppression of the ionized lines of the standard elements by about 50 per cent or the equivalent of one standard was noted, but irregular effects were noted for the neutral atom spectra.

The effects of chemical concentration by removal of alkali matrices show improvement in some cases over the direct spectrographic analysis. The low limits of sensitivity under investigation, however, indicate serious contamination may be introduced in the chemical concentration method. Table VII illustrates both phases of this technique.

TABLE VII

Chemical Concentration of Alkali Samples vs Direct Spectrographic Analysis

Sample	Iron		Copper		Magnesium	
	Chem. Con.	Direct	Chem. Con.	Direct	Chem. Con.	Direct
1	32	45	-	-	4	<1
2	7	<10	<.5	2	2	3.5
3	5	<50	<3	2	-	-
4	5	<50	<3	2	-	-
5	15	<50	3	19	-	-
6	8	<50	<3	6	-	-
7	21	<50	12	10	-	-

Rare Earths and Monazite Sands (J. A. Norris and C. E. Pepper)

New standards for the analysis of rare earth materials were prepared to cover the range 50 to 4000 μg of thorium/ml and 50 to 2000 μg of cerium/ml of solution. A spectrographic method for analysis of monazite sand for constituent thorium and other rare earths has been based on the rare earth analysis method developed in this laboratory.[10] Analysis of a monazite sand by chemical and spectrographic procedures is given in Table VIII.

TABLE VIII

Analysis of Monazite Sand

Material	Chemical*	Spectrographic
ThO_2	9.2%	9.8%
CeO_2	30.5	32.6
Pr_2O_3		3
La_2O_3	32.6	11.5
Nd_2O_3		16.5
Sm_2O_3		1.8
P_2O_5	30.7	not sought

* Data supplied by J. W. Robinson, Analytical Chemistry Division, ORNL

Miscellaneous (J. A. Norris, Z. Combs, C. E. Pepper, and M. R. Skidmore)

The procedure for analysis of mercury has been developed to permit analysis for calcium, copper, iron, magnesium, and tin to as low as 10 ppm. Statistical treatment of data on vanadium line intensity ratios gives a standard deviation of less than 1.7 per cent for lines originating from upper energy levels which are separated by less than 0.5 electron volts. Vanadium as the oxide was used in a hydrochloric acid solution at a concentration of 400 micrograms/milliliter.

Analyses

A total of 495 samples were analyzed for 7703 individual element determinations during this quarter.

10. Norris, J. A. and C. E. Pepper, "Quantitative Spectrochemical Analysis of Rare Earth Mixtures," Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March 7, 1952; also, submitted to Analytical Chemistry.

X-RAY SPECTROSCOPY

X-Ray Diffraction (H. W. Dunn)

Compound identifications in support of ANP, HRE, and other research projects were made in 107 instances. Samples investigated include fluorides, oxides, and chlorides of uranium, iron, lithium, potassium, sodium, beryllium, copper, zirconium, nickel, chromium, and phosphorus.

X-Ray Absorption (W. F. Peed and H. W. Dunn)

A control unit for introducing automatic recycling operations in obtaining X-ray absorption data on uranium solutions has been developed and is now being installed. Precision of counting and tabulating appears to be limited almost exclusively by the statistics of counting.

Preliminary investigations of the effect of a wide range of added lead impurity (0 ppm to 14,750 ppm) in a standard 3686 ppm uranium solution gave a standard deviation of 3.1 per cent or less, the largest error being for 14,750 ppm contained lead.

PUBLICATIONS AND PAPERS

Reports and Papers in Preparation

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

2. 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."

3. W. F. Peed and H. W. Dunn, "Monochromatic X-Ray Absorption Studies on Uranium in Solutions."

4. W. F. Peed, L. E. Burkhart,** R. A. Staniforth,*** and L. G. Fauble,*** "The X-Ray Spectra of Polonium, Atomic Number 84."

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* Research Participant, University of Louisville.
** Now with Y-12 Plant.
***Mound Laboratory, Miamisburg, Ohio.

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

Reports Submitted for Publication

1. J. A. Norris and C. E. Pepper, "Quantitative Spectrochemical Analysis of Rare Earth Mixtures," submitted to Analytical Chemistry.

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

3. G. L. Stukenbroeker, D. D. Smith, G. K. Werner and J. R. McNally, Jr., "Spectro-Isotopic Assay of Lithium," submitted to Journal of Optical Society of America.

Reports Published and Papers Presented

1. H. E. Walchli, R. Livingston, and William J. Martin, "The Nuclear Magnetic Moment of Tc99," Phys. Rev. 85, 479L (1952).

2. J. R. McNally, Jr., "New Techniques in Mass Analysis," Bulletin of Applied Spectroscopy, Vol. 6, No. 2 (Feb., 1952).

3. H. E. Walchli, W. E. Leyshon, and F. M. Scheitlin, "The Nuclear Gyromagnetic Ratio of V50, and Measurements of Rb85 and C135," Phys. Rev. 85, 922-3L (1952); also, ORNL-1239.

4. J. R. McNally, Jr., "Atomic Spectroscopy and Separated Isotopes," American Journal of Physics 20, 152-60 (1952).

5. J. R. McNally, Jr., "Spectro-Isotopy - A New Field of Spectroscopic Endeavor." Paper presented at Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March 5-7, 1952.

6. J. A. Norris and C. E. Pepper, "Quantitative Spectrochemical Analysis of Rare Earth Mixtures." Paper presented at Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March 5-7, 1952.

7. E. J. Spitzer and D. D. Smith, "Spectrochemical Analysis of Zirconium Oxide." Paper presented at Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, March 5-7, 1952.

MASS SPECTROMETER LABORATORY

Russell Baldock, Department Head)

INTRODUCTION

During the third quarter of fiscal year 1952 uranium fluoride studies were made for the ANP project, mass cracking patterns of silicon tetrafluoride were observed, two vacuum gasket materials were investigated, and an attempt was made to use a dry chemical reaction inside the mass spectrometer.

AIRCRAFT FUEL STUDIES

Fulinak (L. O. Gilpatrick and J. R. Sites)

Two runs were made on samples of crystal formations taken from the cold leg of a plugged stainless steel convection loop. The first sample showed boron trifluoride (BF_3), silicon tetrafluoride (SiF_4), a small amount of potassium, but no uranium. No other elements were detected. The second sample was made up of non-magnetic crystals separated from the fulinak. Boron trifluoride and mercury appeared, but no silicon or uranium were indicated.

Silicon Tetrafluoride (L. O. Gilpatrick, R. E. Rummel,* and J. R. Sites)

Silicon tetrafluoride was prepared and operated in the mass spectrometer. The mass cracking pattern and metastable peaks were essentially the same as that of the silicon tetrafluoride found in the uranium fluoride as reported in the "Quarterly Progress Report, Stable Isotope Research and Production Division," ORNL-1228.

Uranium Tetrafluoride (L. O. Gilpatrick and J. R. Sites)

A sample of uranium tetrafluoride (UF_4) that had been exposed to the air for three months was used as a charge material. This run was made to determine whether the charge material had hydralized and thereby would

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* Research Participant, Chemistry Department, Vanderbilt University.

produce, when heated, uranium pentafluoride (UF_5). The pattern obtained was the usual UF_4 pattern with no appreciable change. A sample of UF_5 was obtained from the K-25 plant of Carbide and Carbon Chemicals Company. This sample produced extremely intense peaks of uranium hexafluoride ions (UF_6^+). The sample was heated at approximately 100°C for a few hours after which the intensity of the UF_6^+ peak was reduced to the same order of magnitude as the UF_5^+ peak.

Uranyl fluoride (UO_2F_2) was tested. All the predicted peaks of UO_2F_2, uranium oxytetrafluoride (UOF_4) and UF_5 were found. Apparently any UO_2F_2 in UF_4 could give rise to the UF_5 patterns usually found in material that had not been heat treated.

FUNDAMENTAL STUDIES

Vacuum Materials (J. R. Sites)

A 3/8-inch long by 1/16-inch diameter sample of a silicone rubber (Figure 11) was heated in the source oven of the mass spectrometer and the resulting mass cracking pattern (Figure 12) was observed. A series of hydrocarbon ion peaks appeared at low temperatures during the early part of the run. The expected groupings of dimethyl siloxane peaks did not appear until higher temperatures were reached. After being heated to a maximum 250°C the silicone rubber lost all resilience and crumbled easily when pressed.

A similar sample of teflon (Figure 11) was investigated, but it had to be heated above 250°C before any characteristic peaks appeared. The mass cracking pattern (Figure 13) was simple, and appeared to be groupings of tetrafluoroethylene, the base material. The data found confirms the work of Lewis and Naylor[11] on the pyrolysis of teflon. The ion patterns agree closely with the relative abundances of tetrafluoroethylene as reported by the National Bureau of Standards.[12]

One drop of Octoil-S diffusion pump oil was warmed in the spectrometer source oven. A mass interference pattern (Figure 14), similar to the background, was observed after the cold trap between the diffusion pump and the spectrometer analyzer went dry. These background peaks probably arise from decomposed or oxidized diffusion pump oil. The series of ion peaks observed in this run was less complex with prominent peaks only below mass 100.

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11. Lewis and Naylor, J. Am. Chem. Soc., 69: 1968 (1947)
12. Catalog of Mass Spectral Data, American Petroleum Institute, Vol. I, Research Project 44.

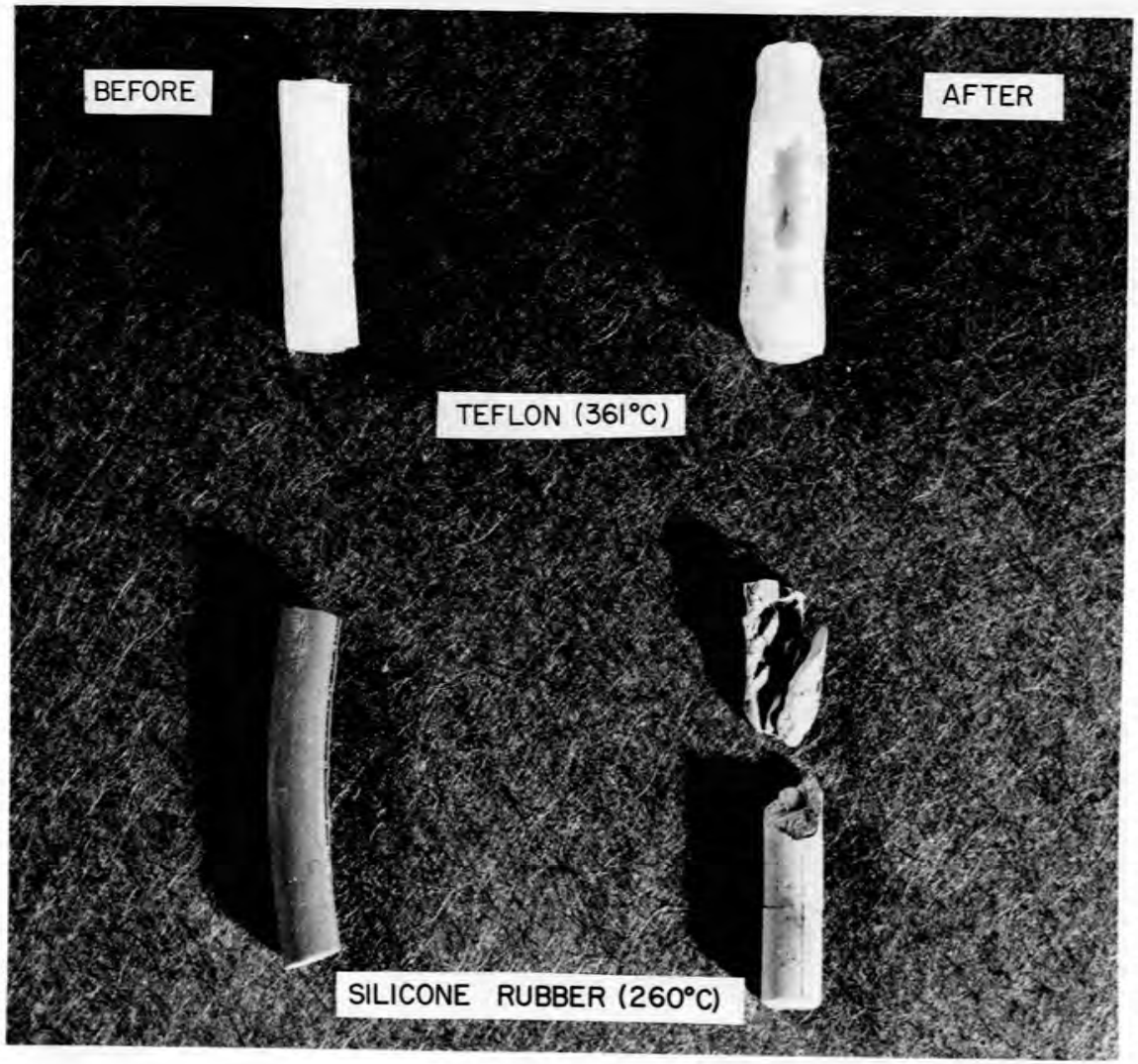

FIGURE 11 MASS SPECTROMETER SAMPLES

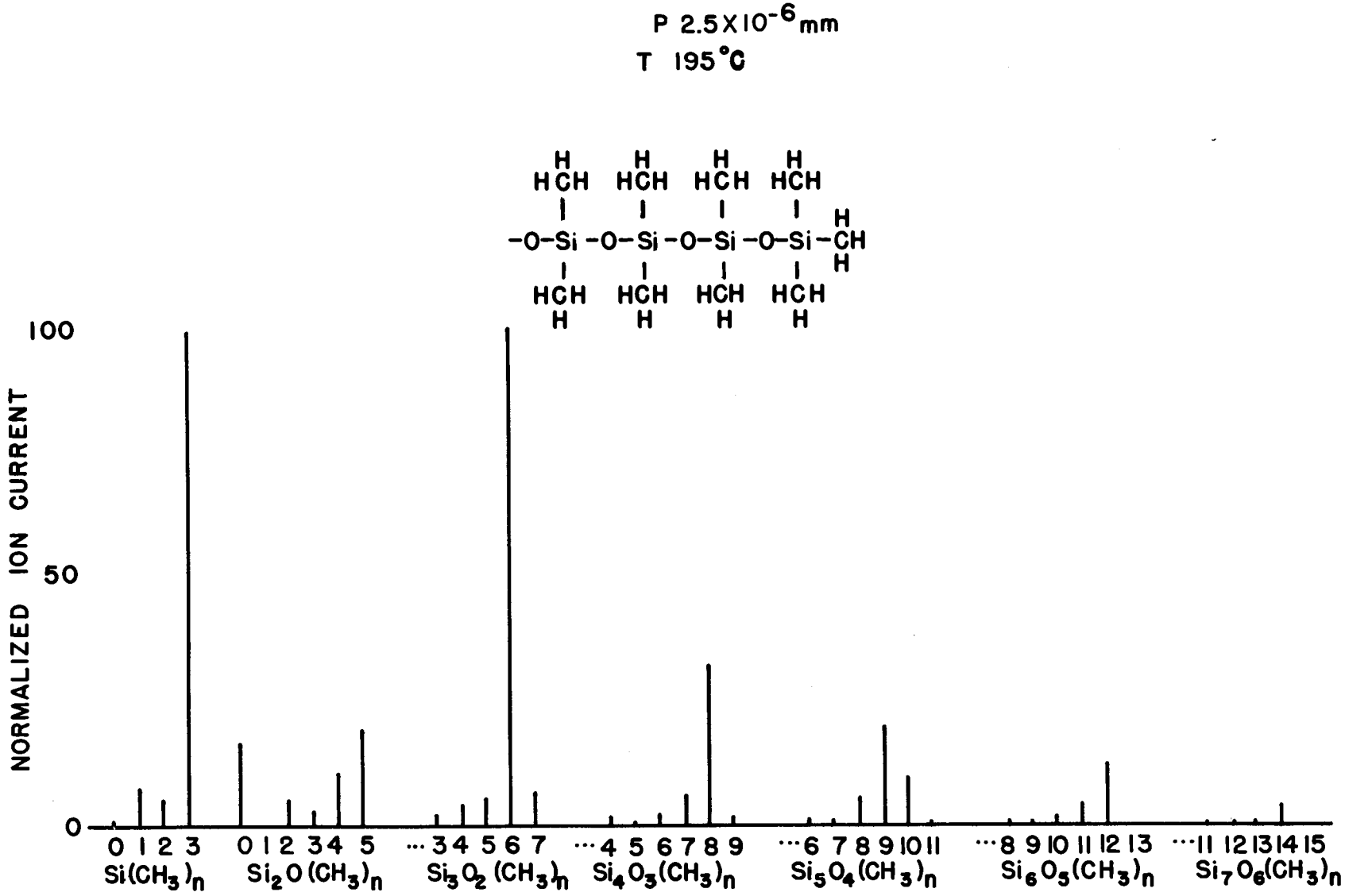

FIGURE 12. MASS CRACKING PATTERN OF SILICONE RUBBER

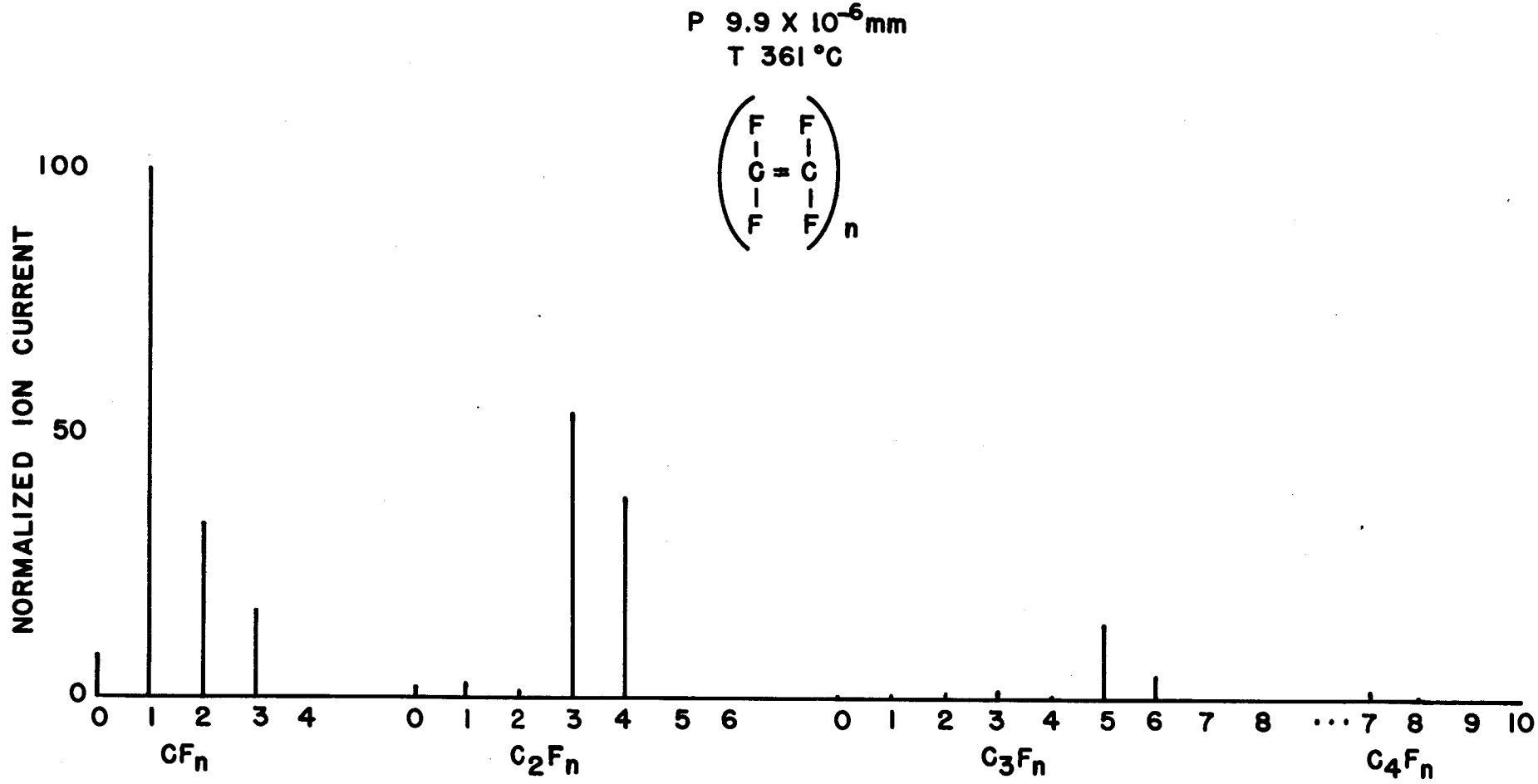

FIGURE 13. MASS CRACKING PATTERN OF TEFLON

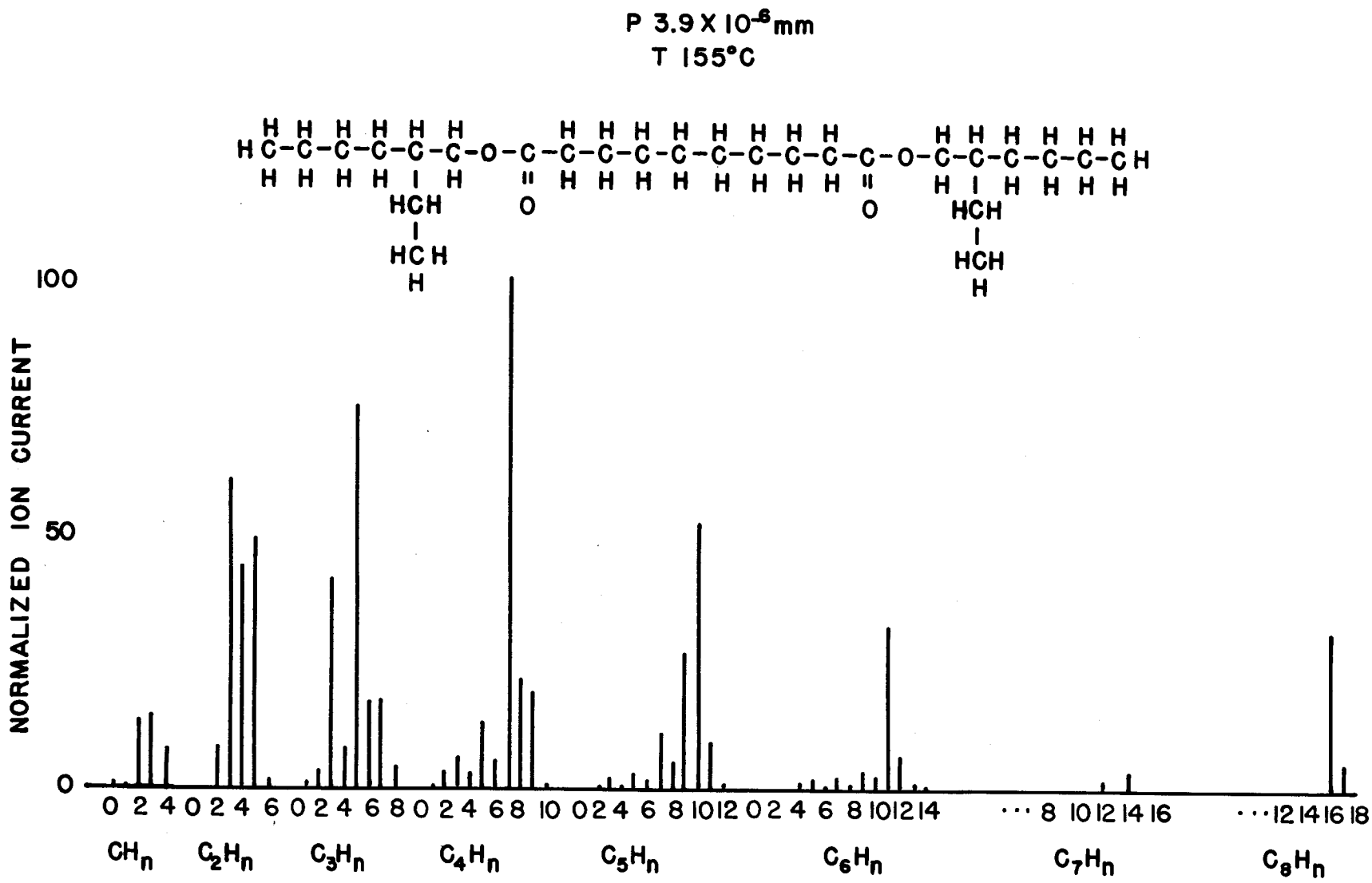

FIGURE 14. MASS CRACKING PATTERN OF OCTOIL-S

Dry Chemistry (L. O. Gilpatrick and J. R. Sites)

Cerous oxide (Ce_2O_3) was mixed with cobaltic fluoride (CoF_3) and heated in the source oven of the mass spectrometer to determine if fluorination of the rare earth oxide would take place and thereby enable one to obtain its mass spectra without external chemistry. The following chemical reaction takes place:

$$12CoF_3 + 2Ce_2O_3 \rightarrow 4CeF_3 + 12\,CoF_2 + 3O_2 \uparrow$$

The reaction proceeds as evidenced by the large quantities of oxygen produced. The source oven could not be heated high enough to vaporize the cerium fluoride. A few small ion peaks of the cobaltic fluoride appeared at about 600°C.

PUBLICATIONS AND PAPERS

Reports in Preparation

1. Robert E. Rummel, John R. Sites, and Russell Baldock, "Mass Spectrometer Study of Silicon Tetrafluoride."

2. L. O. Gilpatrick, Russell Baldock, and John R. Sites, "Mass Spectrometer Investigation of UF_3."

3. Russell Baldock, John R. Sites, and L. O. Gilpatrick, "Mass Spectrometer Study of Technetium."

Papers Presented

1. Robert E. Rummel, John R. Sites, and Russell Baldock, "Mass Spectrometer Study of Silicon Tetrafluoride." Paper presented to the Pittsburg conference on analytical chemistry and applied spectroscopy, March 5-7, 1952.

2. L. O. Gilpatrick, Russell Baldock, and John R. Sites, "Mass Spectrometer Investigation of UF_3." Paper presented to the Pittsburg conference on analytical chemistry and applied spectroscopy, March 5-7, 1952.

3. Russell Baldock, John R. Sites, and L. O. Gilpatrick, "Mass Spectrometer Study of Technetium." Paper presented to the Pittsburg conference on analytical chemistry and applied spectroscopy, March 5-7, 1952.

4. Russell Baldock, "Mass Spectrometry at the Oak Ridge National Laboratory." Paper presented to the Alabama Academy of Science, February 29, 1952.