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PARAMAGNETIC RESONANCE ABSORPTION

Final Report

Clyde A. Hutchison Jr.

The Department of Chemistry and

The Enrico Fermi Institute

The University of Chicago

Chicago, Illinois, 60637

1954 March 1 - 1973 February 28

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RESEARCH ON
PARAMAGNETIC RESONANCE ABSORPTION
FINAL REPORT

CONTRACT NO. AT(11-1)-294
1954 MARCH 1 THROUGH 1973 FEBRUARY 28

Clyde A. Hutchison Jr., Principal Investigator
Enrico Fermi Institute and Department of Chemistry
University of Chicago
Chicago, Illinois, 60637

1. Introduction.

The research in our laboratory supported by the United States Atomic Energy Commission during the period 1954 March 1 to 1973 February 28 may be categorized under two main headings:

- (1) f-electron molecules and ions (and transition element ions); this work has been described in those publications on the attached list numbered 1-16, 18-20, 26, 27, 33, 39, 51, 53, and 58, a total of 26 publications;

and

- (2) organic molecules in triplet electronic states; this work has been described in those publications on the attached list numbered 17, 21-25, 28-32, 34-38, 40-50, 52, 54-57, 59, and 60, a total of 34 publications.

2. Summary of the research on f-electron molecules and ions.

2.1. f-electron configurations.

During the period of AEC support of our research the earliest work on f-electron systems was concerned with the demonstration by EPR (electron paramagnetic resonance) methods and magnetic susceptibility methods of the occurrence of f-electron configurations in ions and molecules containing uranium, neptunium, and plutonium, and with the detailed study of the electronic states of these ions and molecules in crystals. This work is described in publications, 3, 6, 8, 10, 12, 18, 19, and 26, on the attached list.

During this same period we also studied by EPR methods the electronic states of both the trivalent ions of all of the ordinary rare earth elements and also of trivalent uranium ions in the previously relatively little investigated lanthanum trichloride single crystal host. This work is described in publications 1, 3, 6, 10, 11, 16, and 39, on the attached list.

Related work on transition element and rare earth ions is described in publications 2, 4, 5, 7, 14, 15, 20, and 33, on the attached list.

The outstanding accomplishments during this period were (1) the unambiguous establishment of the fact of the occurrence of f-electron configurations in the ions and molecules containing uranium, neptunium, and plutonium; (2) the first thorough study of the electronic states of f-electron ions in the LaCl_3 host crystal, a host which has turned out to be of great importance for both optical and magnetic studies of such ions; and (3) the obtaining of precise information on the nature of the electronic states of the NpF_6 molecule.

2.2. Nuclear properties.

Concurrently and subsequently our interest turned to various aspects of the study, by EPR and ENDOR (electron nuclear double resonance) methods, of the nuclear spins, magnetic moments, and electric

moments of rare earths, uranium, neptunium, and plutonium. This work is described in publications 3, 6, 10, 13, 19, 27, and 53, on the attached list.

The outstanding accomplishments of this work were (1) the first determination of the nuclear spin of ^{235}U ; (2) the demonstration that by ENDOR methods the magnetic dipole moments of nuclei of f-electron ions in crystals could be determined relatively accurately without any knowledge of the f-electron wave functions; (3) the measurement of the nuclear magnetic dipole moments and relative moments of the nuclides ^{235}U , ^{143}Nd , ^{145}Nd , ^{147}Sm , ^{149}Sm ; and (4) the demonstration that ENDOR studies of f-electron ions in crystal fields could give important information on the shielding and antishielding of f-electrons and ion nuclei from crystal fields by outer closed shell electrons.

2.3. Electronic states.

With the development of ENDOR methods in our laboratory we were able to obtain very detailed information on ground and excited states of f-electron ions and molecules in crystals. This work is described in publications 27, 53, and 61, on the attached list.

The outstanding accomplishments of this work are the obtaining of very detailed and precise information on the electronic states of Nd^{+3} and Sm^{+3} ions and of NpF_6 molecules in single crystals.

2.4. Photoexcited states.

Finally our research effort has turned to the study of photoexcited electronic states of trivalent rare earth ions in single crystals by EPR and ENDOR methods. The published results of this work to date are given in publications 51 and 58, on the attached list.

The outstanding accomplishments of this work are (1) the first magnetic resonance studies of any trivalent rare earth ion in a photoexcited electronic state; and (2) the demonstration of the existence of interesting and efficient energy transfer processes between f-electron ions in single crystals.

3. Summary of research on organic molecules in triplet electronic states.

3.1. Mechanism of phosphorescence.

During the period of AEC support of our research the earliest work on photoexcited triplet states of aromatic organic molecules was concerned with the demonstration of the validity of the Lewis-Terenin mechanism as an explanation of the phosphorescence of these molecules. This work is described in publications 17 and 22, on the attached list.

The outstanding accomplishments of this period were the conclusive demonstration that the Lewis-Terenin mechanism was in fact responsible for the phosphorescence of aromatic organic molecules by the use of EPR methods to demonstrate that the phosphorescing species was paramagnetic and was in an electronic state of triplet spin multiplicity.

3.2. EPR spectroscopy of organic molecules in electronic states of triplet spin multiplicity.

Subsequently our attention turned to the detailed study of the fine structure and hyperfine structure of the magnetic resonance spectra of organic molecules in both photoexcited and ground triplet electronic states. This work is described in publications 22-24, 28-32, 35, 36, 38, 40, 42-46, 48-50, 54, 55, 57, and 59, on the attached list.

The outstanding accomplishment of this work was the attainment of a detailed understanding of the distribution of electron spin angular momentum and of the electron-electron, electron-proton, and electron-carbon nucleus interactions in triplet state organic molecules.

3.3. Transfer of triplet excitation from one molecule site to another in organic crystals.

EPR methods were used to study the triplet state energy transfer processes which occur in organic crystals. This research is described in publications, 25, 34, 42, 47, 48, 56, and 60, on the attached list.

The outstanding accomplishment of this work was the demonstration by EPR methods that there was transfer of triplet excitation from one molecule site to another several molecules away and the detailed elucidation of the mechanisms by which this transfer occurs.

3.4. Deuteration effects.

Closely related to the work described in 3.3. was our observation and investigation of the effects of deuteration on lifetimes and energy transfer processes of photoexcited triplet state molecules in crystals. This work is described in publications 21, 34, 41, 42, 52, 56, and 60, on the attached list.

The outstanding accomplishments of this research were (1) the observation of the remarkable effect of deuteration on the lifetime of the phosphorescent triplet states of aromatic organic molecules in single crystals and (2) the obtaining of detailed information on the nature of various triplet state energy transfer processes in organic crystals.

3.5. Shapes and structures of organic molecules in triplet electronic states.

EPR and especially ENDOR techniques used in our laboratory led to useful methods of studying triplet state molecule shapes, orientations, and local structure distortions in organic crystals. This research is described in publications 32, 35 - 38, 42 - 45, 49, 50, 54, 55, 57, and 59, on the attached list.

The outstanding accomplishments of this work were (1) the determination of the shapes and geometrical conformations of substituted methylenes in organic crystals; (2) the acquisition of detailed knowledge of the orientations of chemical impurity species in organic host crystals and of the local disruptions of host crystal structure which are caused by them; and (3) the distortions in crystals from the ideal symmetries of triplet state organic molecules.

3.6. Chemical reactions in organic crystals.

Finally, EPR and ENDOR methods have been used in our laboratory to study the kinetics and the mechanisms of chemical reactions occurring in organic single crystals without disruption of the host crystal structure. This research is described in publications 49 and 54, on the attached list.

The outstanding accomplishment of this research was the use of ENDOR (and EPR) methods to determine the detailed mechanism and pathway of two chemical processes occurring in an organic single crystal.

4. Ph.D. students whose research was supported by the AEC during the period 1954 March 1 to 1973 February 28.

NAME	THESIS TITLE
1. John H. Anderson	Paramagnetic Resonance Absorption in Praseodymium Trichloride. Phys. Rev. <u>97</u> , 76 (1955) co-authored with Clyde A. Hutchison Jr.
2. Nicholas A. Ashford	Triplet State Paramagnetic Resonance Absorption and Energy Transfer in the Single Crystal System β -Methylnaphthalene in Naphthalene.
3. Henry C. Brenner	Electron Paramagnetic Resonance Studies of Triplet State Energy Transfer in Single Organic Molecular Crystal.
4. J. Philip Bromberg	Magnetic Susceptibilities of Uranium Dioxide in Thorium Dioxide and of Osmium and Iridium Hexafluorides in Uranium Hexafluoride.
5. James E. Butler	Electron Paramagnetic Resonance and Electron Nuclear Double Resonance of $^{237}\text{NpF}_6$ in Single Crystals of UF_6 .
6. George A. Candela	Magnetic Susceptibilities of Uranium (IV) and Plutonium (IV) Ions in Cubic Crystal Fields.
7. Iu-Yam Chan	ENDOR Spectra of Sm^{+3} in LaCl_3 .
8. David C. Doetschman	A Study of Chemical Reactions of Diphenyldiazomethane and Diphenylmethylene in Single Crystals of 1,1-Diphenylethylene by Magnetic Resonance Methods.
9. Lynden E. Erickson	Electron-Paramagnetic-Resonance Absorption by Trivalent Neodymium Ions in Single Crystals by Lanthanum Trichloride and Lanthanum Ethyl Sulphate in Zero Magnetic Field. Phys. Rev. <u>143</u> , 295 (1966).

NAME	THESIS TITLE
10. Antonio M. Ponte Goncalves	Hyperfine Interactions in Organic Triplet States Phenanthrene and Benzene.
11. Carl A. Gottlieb	Electron Paramagnetic Resonance in Thorium Oxide Single Crystals Containing Uranium.
12. Donald Halford	Electron-Nuclear Double Resonance, Nuclear Moments, and $\langle r^{-3} \rangle$ of Neodymium-143 (III) and Neodymium-145 (III) in Lanthanum Trichloride. Phys. Rev. <u>127</u> , 1940 (1962).
13. Paul M. Handler	The Magnetic Susceptibilities of Some Uranium Compounds.
14. Arthur H. Heiss	La ENDOR LaCl_3 : Sm^{3+} .
15. Tex L. Horning	Present graduate student.
16. Soon Sam Kim	Present graduate student.
17. John Swinton King Jr.	Magnetic Resonance Studies of the Triplet State of a Juxtaposed Pair of Molecules.
18. Bryan E. Kohler	Polarized Optical Absorption Spectra of the Ground State Triplet Molecules Fluorenylidene and Diphenylmethylene Oriented in Single Crystals.
19. Arthur G. Kowalsky	Analysis of Organic Free Radicals by Paramagnetic Resonance Absorption.
20. Narcinda R. Lerner	Electron Nuclear Double Resonance, Nuclear Moments and $\langle r^{-3} \rangle$ in Uranium-235 (III) in LaCl_3 .
21. En-Den Liu	Present graduate student.
22. Billy W. Mangum	Paramagnetic Resonance Absorption in Naphthalene in Its Phosphorescent State.

NAME	THESIS TITLE
23. Valda H. McCann	Electron Nuclear Double Resonance by Triplet State Molecules of Phenanthrene- d_{10} in Single Crystals of Diphenyl.
24. Gordon A. Noble	Paramagnetic Resonance in Additively Colored Alkali Halides.
25. Donald E. O'Reilly	Paramagnetic Resonance Absorption Studies of Solutions of Potassium in Liquid Ammonia and Deutero-Ammonia.
26. Gerald A. Pearson	Electron Nuclear Double Resonance and Spin Densities in Ground Triplet State Fluorenylidene in Diazofluorene Single Crystals.
27. Paul J. Peckar	Electron Nuclear Double Resonance and Nuclear Moments of Uranium-233 Tripositive Ions in Single Crystals of Lanthanum Trichloride.
28. Gary W. Scott	Zero Field Magnetic Resonance Spectroscopy of the Lowest Photo-excited Triplet State of Aromatic Molecules.
29. Tsang Tung	Magnetic Susceptibilities of Neptunium Hexafluoride in Uranium Hexafluoride and of Uranium Dioxide in Thorium Dioxide.
30. John A. Weil	1. A Study of the Equilibrium between Hexaphenylethane and Triphenylmethyl by Use of the Paramagnetic Resonance Technique. 2. A Search for the Paramagnetic Resonance Absorption of an Organic Dye in Its Triplet State.
31. Eugene Wong	Paramagnetic Resonance of Rare Earth Ions in $LaCl_3$.
32. Yung-Tsai Yen	Present graduate student.

5. Postdoctoral research associates whose research was supported by the AEC during the period 1954 March 1 to 1973 February 28.

NAME

1. Elliot R. Bernstein
2. Roy W. Brandon
3. J. Philip Bromberg
4. Richard H. Clarke
5. Henry H. Dearman
6. Paul B. Dorain
7. Arthur Forman
8. R. Peter Frosch
9. Roger E. Gerkin
10. James A. Hebden
11. Jan P. Hessler
12. Noboru Hirota
13. Marvin D. Kemple
14. Bryan E. Kohler
15. Peter M. Llewellyn
16. William Low
17. John V. Nicholas
18. Charles G. Wade
19. Eugene Y. Wong

6. Undergraduate students whose research was supported by the AEC during the period 1954 March 1 to 1973 February 28.
(This list does not include the names of undergraduates who worked mainly as technicians in our laboratory but only of those who engaged in active research programs.)

1. Michael N. Hadad
2. Patrick E. Palmer
3. Bilin Tsai

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1. John H. Anderson and Clyde A. Hutchison Jr.
Paramagnetic Resonance Absorption in Praseodymium Trichloride.
Phys. Rev. 97, 76 (1955).
2. W. Low.
Paramagnetic Resonance Spectrum of Manganese in Single Crystals of Alkali Halides Grown from Solutions.
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Proc. Phys. Soc. B LXIX, 1169 (1956).
5. W. Low.
Paramagnetic Resonance Spectra of Some Ions of the 3d and 4f Shells in Cubic Crystalline Fields.
Phys. Rev. 101, 1827 (1956).
6. Clyde A. Hutchison Jr., P. M. Llewellyn, Eugene Wong, and Paul Dorain.
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Phys. Rev. 102, 292 (1956).

7. W. Low.
Hyperfine Structure and Nuclear Moments of Gadolinium
from Paramagnetic Resonance Spectrum.
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Magnetic Susceptibilities of Uranium (V) and Uranium (III)
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Paramagnetic Resonance Absorption in Uranium (III)
Chloride and the Nuclear Spin, Magnetic Dipole Moment,
and Electric Quadrupole Moment of Uranium-233.
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11. Clyde A. Hutchison Jr., B. R. Judd, and D. F. D. Pope.
Paramagnetic Resonance Absorption in Gadolinium
Trichloride.
Proc. Phys. Soc. B LXX, 514 (1957).
12. Clyde A. Hutchison Jr. and George A. Candela.
Magnetic Susceptibilities of Uranium (IV) Ions in Cubic
Crystalline Fields.
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13. Donald Halford, Clyde A. Hutchison Jr., and Peter M. Llewellyn.
Electron Nuclear Double Resonance of Neodymium.
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19. Clyde A. Hutchison Jr. and Bernard Weinstock.
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Kohler, and J. W. Stout.
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Ion in Cadmium Dichloride.
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(preprint not yet available).
60. Henry C. Brenner.
Electron Spin Polarized Triplet State Energy Transfer in Mixed Organic Single Crystals at Low Temperatures.
(preprint not yet available).
61. James E. Butler and Clyde A. Hutchison Jr.
EPR and ENDOR of NpF_6 in Single Crystals of UF_6 .
(preprint not yet available).

Several papers which are still in preparation
will be forthcoming on work supported
by the Atomic Energy Commission.