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# Transition Metal and Rare Earth Compounds

Excited States, Transitions,  
Interactions I

213 Topics in Current Chemistry



Springer

# **Transition Metal and Rare Earth Compounds**

## **Excited States, Transitions, Interactions I**

**Volume Editor: Hartmut Yersin**

With contributions by

K. L. Bray, M. Glasbeek, H. Kunkely, A. Vogler



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## Preface

For a long time, the properties of transition metal and rare earth compounds have fascinated chemists and physicists from a scientific view-point, and more recently also their enormous potential as new materials has been explored. Applications in different fields have already been realized or are under current investigation, for example, new laser materials, IR to visible upconversion systems, compounds for photolithographic processes, systems involving photo-redox processes for solar energy conversion, new photovoltaic devices, chemical sensors, biosensors, electroluminescent devices (OLEDs) for flat panel display systems, supramolecular devices with wide-range definable photophysical properties, materials for energy harvesting, optical information and storage systems, etc. Metal complexes are also highly important in biology and medicine. Most of the applications mentioned are directly related to the properties of the electronic ground state and the lower-lying excited states. Metal complexes with organic ligands or organometallic compounds exhibit outstanding features as compared to purely organic molecules. For instance, metal compounds can often be prepared and applied in different oxidation states. Furthermore, various types of low-lying electronic excitations can be induced by a suitable choice of ligands, for example, such as metal-centered transitions (MC, e.g. d-d\* transition), ligand-centered (LC, e.g.  $\pi$ - $\pi^*$ ), metal-to-ligand-charge transfer (MLCT, e.g. d- $\pi^*$ ), intra-ligand-charge-transfer (ILCT) transitions, etc. In particular, the orbitals involved in the resulting lowest excited states determine the photophysical and photochemical properties and thus the specific use of the compound. It is of further interest that the lowest excited electronic states can be shifted over the large energy range from the U.V. to the I.R. by chemical variation of the ligands and/or the central metal ion. Moreover, these excited states have mostly spin-multiplicities different from those of the electronic ground states. In contrast to organic molecules, spin-orbit coupling induced by the metal center is of crucial importance for the splitting and the population and decay dynamics of these multiplets as well as for transition probabilities. In summary, it is of outstanding importance that this class of compounds provides the possibility of tuning excited state properties by chemical variation. Thus, compounds with user-defined excited state properties can be prepared.

In view of the fascinating potential of these compounds, it is of great interest to develop a deeper understanding of their photophysical properties. In this volume, leading scientists present modern research trends in comprehensive reviews which not only provide a deep insight into the specific subjects, but are



also written in a style that enables researchers from related fields and graduate students to follow the interesting subjects. In particular, in the present volume Kevin L. Bray presents effects that are observed under the application of high pressure. Max Glasbeek introduces us to optically detected magnetic resonance (ODMR) techniques as applied to transition metal complexes and Arnd Vogler and Horst Kunkely give a summary concerning the diversity of excited states as found in various compounds. In a companion volume (Topics in Current Chemistry 214, in press) Daniel R. Gamelin and Hans U. Güdel review upconversion processes in luminescent transition metal and rare earth systems. Mark J. Riley relates geometric and electronic properties of copper(II) compounds. Finally, Hartmut Yersin and Dirk Donges explain on the basis of case studies, the ways in which photophysical properties of organometallic and related compounds depend on the metal character of the low-lying electronic states and how these properties can be tuned by chemical variation.

I hope that the contributions in the present and in the subsequent volume demonstrate the attractiveness and the enormous potential of metal compounds and that a more detailed understanding of the photophysical properties will open pathways to new developments.

Regensburg, Germany  
October 2000

Hartmut Yersin

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# High Pressure Probes of Electronic Structure and Luminescence Properties of Transition Metal and Lanthanide Systems

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Recent work has demonstrated that unique insight into the electronic structure and optical properties of solid state transition metal and lanthanide systems is achievable through high pressure studies. In this review, we present selected examples that illustrate the advances in understanding possible from high pressure luminescence experiments. The ability of pressure to continuously vary energy level structure and coordination environment is emphasized and related to variations in luminescence properties. Pressure is shown to influence luminescence properties through crystal field, covalency, and spin-orbit coupling effects. The effect of pressure on luminescence energy, intensity, and decay properties will be considered. Additional phenomena to be discussed include electronic crossovers, vibrational coupling, admixing of electronic states, energy transfer and site selective spectroscopy. High pressure luminescence properties of transition metal and lanthanide ions in crystalline solids, metal complexes, and glasses will be reviewed.

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## 1

### Introduction

The ability to predict and control the luminescence properties of transition metal and lanthanide systems has long been an objective of the optical materials community. Solid state lasers, solar energy conversion, optical sensing, optical data storage, and phosphors are examples of areas where precise control over the wavelength, lineshape, and efficiency of luminescence is essential for optimizing performance and extending the range of applications. Luminescence properties of transition metal and lanthanide ions are ultimately controlled by the chemical and physical interactions they experience in a given material. The most important interactions occur in the nearest neighbor coordination shell where factors such as bond lengths, bond angles, coordination number, and covalency determine the energy, mixing, and splittings of the electronic states involved in luminescence. Secondary effects associated with more distant neighbors are also frequently important and depend on the system in which a luminescent ion is found. When transition metal and lanthanide ions are incorporated as dopants in crystalline or amorphous solids, ligands in the first coordination shell are strongly bonded to second nearest neighbors. As a result, the spatial distribution and electronic interactions of ligands in the first coordination shell are constrained. In molecular inorganic or organometallic complexes, intramolecular interactions normally dominate intermolecular interactions and the influence of atoms outside the first coordination shell occurs secondarily through ligand-ligand interactions or chelation effects.

The design of luminescent materials for new and more exacting applications requires a systematic understanding of the relationship between the bonding environment of transition metal and lanthanide centers and absorption and emission properties. The customary approach for investigating the relationship between bonding environment and optical properties is through variations of chemical composition. By changing chemical composition, it is possible to systematically vary the local bonding environment of luminescent centers and gain information useful in developing a predictive capability. Examples of this approach include luminescence studies of a given metal ion as a dopant in a series of host lattices or of complexes of a given metal ion with a series of ligands.

In this review, we present an alternative approach to gaining structure-bonding-property information for luminescent transition metal and lanthanide ions in solids and complexes. The approach is based on using hydrostatic pressure to systematically influence the bonding environment of luminescent centers. We will show that with pressure it is possible to directly influence the bonding interactions that are responsible for determining the luminescence properties of the electronic states of transition metal and lanthanide ions. By correlating pressure-induced changes in electronic states, structure, and bonding with pressure-induced changes in luminescence properties, we create new opportunities for generalizing the behavior of transition metal and lanthanide ions.

High pressure studies are complementary to the conventional chemical composition approach and offer several potential advantages. First, with high pressure we can continuously vary structure, coordination environment, and luminescence properties. Variations in chemical composition, on the contrary, lead to discrete changes. The continuous tuning capability of pressure provides new opportunities for correlating structure, bonding, and luminescence properties. The finer resolution offered by high pressure is particularly important at conditions such as electronic crossovers or phase transitions where optical properties vary abruptly. Second, with high pressure we have the ability to study a given chemical composition over a wide range of structure and bonding conditions. As a result, the need to synthesize a range of host lattice compositions or complexes with a variety of ligands is minimized. Complications such as differences in impurities or defects that may accompany a series of systems are also avoided. Third, high pressure provides the potential for achieving structures, molecular configurations, phases and electronic states that are not achievable through other means. Variations in pressure alter the relative thermodynamic stability of different states of a system and offer the possibility of creating new states with new properties. Finally, high pressure often allows us to resolve overlapping or competing excited state processes. Frequently several optical processes and/or electronic states contribute to ambient pressure luminescence properties. Since competing effects are typically perturbed to differing extents by pressure, it becomes possible to stabilize one effect relative to others. Characterization of dominant effects at high pressure, followed by back-extrapolation to ambient pressure, frequently leads to new understanding of ambient pressure luminescence properties.

High pressure and variable chemical composition studies share the goal of striving to identify the chemical and physical factors necessary for controlling

optical properties. The ultimate objective is to progress toward the rational design of new materials with new ambient pressure properties. The following review attempts to illustrate the unique insight into luminescence properties achievable through high pressure studies. We restrict our attention to the luminescence properties of transition metal and lanthanide ions in insulating inorganic solids and metal complexes. The effects of pressure on the luminescence properties of pure organic systems [1–18], inorganic systems that do not contain transition metal or lanthanide ions [19–25], semiconductors [26–38], and liquid phase systems [39–50], as well as related techniques such as uniaxial stress [51–54] and shockwaves [55, 56], will not be discussed. We begin by describing the methods of high pressure luminescence spectroscopy and the phenomenological effects of pressure on basic luminescence properties. We then present several examples from the recent literature that illustrate the range of phenomena observable at high pressure and conclude with a few remarks concerning the future.

## 2

### Methods of High Pressure Luminescence

Studies of material properties as a function of pressure have received less attention historically than studies as a function of temperature. As a result, most investigators have a more intuitive sense of the effect of temperature on materials and routinely consider temperature-dependent experiments during the course of their work. This state of affairs is unfortunate because pressure is an equally valuable variable that not only complements temperature, but also provides an opportunity to gain insight not attainable through temperature studies. Whereas temperature studies are valuable probes of competing states and processes that are separated by an energy barrier, pressure can alter the heights of energy barriers and is much more likely to stabilize new states and phases.

The underutilization of high pressure methods is partly due to the perception that the laboratory facilities and procedures needed to create high pressure conditions are cumbersome and time consuming. Contrary to this perception, the tremendous advances that have been made in high pressure experimentation over the past decade have greatly increased the accessibility of high pressure methods to the scientific community and have prompted growing interest in research at high pressure conditions. The range of experiments that can now be conducted as a function of pressure includes [57–60] X-ray diffraction, Brillouin scattering, EXAFS, optical absorption, luminescence, non-linear spectroscopy, Raman scattering, NMR, and conductivity. High pressure experiments can also be completed at both low and high temperature.

In this section, we present basic aspects of modern high pressure experimentation. We begin with a few introductory comments about pressure, its effect on materials, and the range of pressure normally needed for studying solids. We continue by discussing the diamond anvil cell technique used by many groups to generate high pressure in the laboratory and conclude with a discussion of luminescence experiments at high pressure.

## 2.1

### High Pressure as an Experimental Variable

High pressure has traditionally been viewed as a macroscopic, thermodynamic experimental variable. Classic applications of pressure have involved equation of state studies of liquids and solids and measurements of the variation of physical properties as a function of pressure [57, 59–66]. The basic effect of pressure on a system is a consequence of the thermodynamic stability requirements of the second law [67] and can be expressed most generally as

$$\left(\frac{\partial V}{\partial P}\right)_T < 0 \quad (1)$$

Equation (1) indicates that, in order to comply with the second law, the volume of a material must decrease upon isothermal compression. The precise manner, however, in which a material reduces its volume in response to an applied pressure is unspecified by the second law and requires consideration on a molecular level. Molecular attributes such as bond angles, bond lengths, covalency, coordination number, and intermolecular forces can be influenced by pressure. Since these attributes are responsible for defining chemical, electrical, optical, and magnetic properties, pressure is a potentially powerful probe of the properties of materials.

When considering the potential effect of pressure on a system, it is useful to recognize the magnitude of pressure required to significantly alter molecular and bulk properties. The isothermal compressibility,  $\kappa$  (or its reciprocal  $K$ , the bulk modulus) (Eq. 2), gives an indication of the sensitivity of a system to pressure:

$$\kappa = -\frac{1}{V} \left(\frac{\partial V}{\partial P}\right)_T = \frac{1}{K} \quad (2)$$

Systems with large compressibilities (“soft” or “highly compressible” systems) exhibit large volume decreases per unit applied pressure and as a result are expected to show significant changes in properties with pressure. Pressures of a few bars, for example, can significantly alter the behavior of gases. Condensed phases experience much stronger intermolecular interactions and are much less compressible. As a result, pressures in the kbar and even mbar range are generally required to measurably perturb the properties of solids and liquids.

Our focus in this review is on the luminescence behavior of solid state lanthanide and transition metal systems over a pressure range extending up to ~300 kbar. Since this magnitude of pressure is well beyond everyday experience, it is beneficial to consider how these pressures compare to those encountered in the physical world. Table 1 presents selected examples from a more comprehensive compilation presented by Jayaraman [68]. The pressures in Table 1 range from  $10^{-19}$  bar in outer space to  $10^{11}$  bar at the center of the sun. The unit of pressure of relevance to this review is the kbar. From Table 1, we see that 1 kbar corresponds approximately to the pressure at the deepest point in the ocean. A pressure of 50 kbar would result if one were to invert the Eiffel tower and place it on



**Table 1.** Pressures encountered in several physical situations

Situation	Approximate pressure (bar)
Pressure in outer space	$10^{-19}$
High vacuum chamber	$10^{-14}$
Sound waves	$10^{-10}$ - $10^{-4}$
Inside light bulb	$10^{-1}$
Sea level	1
Engine cylinder	10
Charged scuba tank	100
Marianas Trench (37,800 ft)	1000
Freezing of H <sub>2</sub> O at 100 °C	$3 \times 10^4$
Center of Earth	$6 \times 10^6$
Center of Jupiter	$10^8$
Center of Sun	$10^{11}$

a 12 cm square plate [69]. Static pressures well above 1 Mbar have been achieved and are routinely used in many laboratories [57–60, 70–73].

**2.2**  
**Diamond Anvil Cell**

The generation of high pressure in the laboratory is typically accomplished by placing a sample between opposing anvils and forcing the anvils together to produce pressure. The strength of the anvil material ultimately determines the maximum attainable pressure. In the early days, large hydraulic presses based on diametrically opposed hardened steel or carbide anvils were used in high pressure research. Over the years, several revisions occurred which led to the development of devices based on multiple anvil and toroidal geometries. These devices are capable of generating ~300 kbar and continue to be widely used today [59, 60, 74].

A new era of high pressure research began with the advent of the diamond anvil cell [57–60, 68, 75, 76]. The use of diamond as an anvil material has two important advantages over steel or carbide anvils. First, diamond is the hardest known substance and is capable of reaching higher pressures than steel or carbide anvils. Second, diamond is optically transparent and therefore permits a wider range of experimental probes at high pressure. Photon spectroscopies, in particular, become possible in diamond anvil cells. Although diamond anvil cells were recognized to be versatile devices for generating pressure, they were not widely used initially because of the difficulty in accurately determining the pressure exerted on a sample. It was not until the development of the ruby fluorescence pressure calibration method (see Sect. 2.2.1) in the 1970s that the diamond anvil cell became the leading device for generating static high pressures.

A schematic depiction of a diamond anvil cell is shown in Fig. 1. The principle of operation of the cell is straightforward [77]. A sample is placed between the two parallel diamonds. By moving the diamonds together, a force is trans-