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Christian Klixbull Jørgensen (1931–2001): inorganic spectroscopist extraordinaire

Peter Day *

Davy Faraday Research Laboratory, The Royal Institution of Great Britain, 21 Albemarle Street, London W1S 4BS, UK

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1. Spectroscopist extraordinaire

By devising simple models for correlating electronic spectra of inorganic compounds and through his inimitable style in expounding them, Christian Klixbull Jørgensen, who died on 9 January 2001, exerted a powerful influence on the development of inorganic chemistry from the 1950s onwards, and which continues (often unacknowledged) up to the present day. The style was the man, and the style consisted of recording and assimilating enormous quantities of experimental data, much obtained by himself (he rarely had students or co-workers) and the remainder garnered from endless intense reading in the frequently obscure scientific literature. Indeed, the image of him that stays most readily in the mind is of a large domed head bent low over a tome in the library, for he was decidedly short sighted. It is, too, the archetypal image of a savant, for that he undoubtedly was, as well as, perhaps less kindly, a mad professor.

2. The making of a spectroscopist

It seems right to begin this brief appreciation of the life and work of Klixbull (as he was known to all but his closest friends and colleagues) on such a personal note, not only because it was my privilege to know and work with him over many years, but because, in an age where scientific knowledge is increasingly regarded as a commodity and its practitioners as ciphers, Klixbull was notably individualistic in his approach to the search for order in nature. He was born at Allborg, in the Danish province of Jutland on 18 April 1931. His father was an officer on a training ship but the family soon moved to Copenhagen, where he went to school and to university. He was a precocious pupil, with an early interest in science fostered by experiments at home with a chemistry set. It was there that began his lifelong fascination with the rare earth elements, which he tried

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* Tel.: +44-20-7670-2928; fax: +44-20-7495-7395.
E-mail address: pday@ri.ac.uk (P. Day).

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to separate by fractional crystallisation. His interest in optical spectroscopy was also kindled by contact with a member of the staff of the Neils Bohr Laboratory in Copenhagen.

Arriving at the University of Copenhagen he quickly extended the reading materials set by his teachers to include, somewhat surprisingly, the doctoral dissertation of Professor Jannik Bjerrin, who was later to become Klixbull's research supervisor. It is not hard to imagine the consternation of his already eminent professor on being relentlessly quizzed by a determined undergraduate about the details of his own thesis work. Perhaps it was Bjerrum's friendly response to this onslaught that persuaded Klixbull to take up graduate work in that laboratory. More likely, though, it was the combination of inorganic chemistry (which at that time was beginning to emerge from an empirical and fact-collecting phase into a search for more theoretically based explications) with the opportunity to exploit what was then an emerging new physical tool, visible absorption spectroscopy.

3. Absorption spectroscopy of complexes in the 1950s

In order to do justice to Klixbull's abiding influence on the development of models for chemical bonding in inorganic compounds (not only metal complexes) it is necessary to recreate the circumstances prevailing at that point in the evolution of the subject. Of course, in a general sense, the colours of inorganic compounds had been a source of interest to chemists for a very long time, often being the only physical property that could easily be reported, but the earliest optical spectroscopy concerned lanthanide compounds, in which the narrow intrasubshell transitions were susceptible to study by spectroscopies with photographic recording. Connecting these sharp spectroscopic features with chemical bonding we now know to be quite a subtle business, because to quite a good approximation the orbitals concerned do not play much of a role in bonding. However, following the Second World War it became much easier to record the broad and diffuse absorption spectra that most transition metal complexes show in solution at room temperature through the introduction of photocell detectors. This technical innovation also coincided with a surge of interest in the thermodynamics of forming metal complexes in solution and quantitative spectrophotometry, often at a single wavelength, when using a manual spectrophotometer such as the Beckman DU, came to the fore. Bjerrum's laboratory, being one of the world's leading centres for determining and rationalising complex formation constants, was equipped with the new technique and this, together with the young Klixbull's introduction to atomic spectra through his mentor in the Neils Bohr Institute, kindled the flame.

4. Methods and aims

It is true that Klixbull's very earliest publications were on thermodynamic properties in collaboration with his mentor and thesis advisor Jannik Bjerrum [1,2], but apart from one or two theoretical papers co-authored with Carl Ballhausen [3–5], practically all his publications (and there were no fewer than 44 in the first five years of his career) carry his name alone. As one who had the privilege of seeing him in action in the laboratory just a few years later, I can vouch personally for his prodigious rate of production. From the preparative laboratory, where quite often the product came directly from the supplier's bottle into the sample cuvette, to the spectrometer room where the Cary 14 ran more or less continuously, and on to the office, where the results were transcribed immediately and a manuscript written out in immaculate longhand a few hours later, scarcely can the path from conception to publication have been shorter. The famous phrase attributed to Michael Faraday, 'work, finish, publish', applied precisely to Klixbull. There was an exhilaration, too, in being at the opening of a new subject, when almost any new data, even on the simplest of compounds, raises questions and widens horizons. Of course, in this headlong rush mistakes were made—compounds wrongly identified, especially when multiple complexes were formed in solution or spectral bands overlapped. But the insights which moulded a discipline were not obtained by tireless study of the minutiae in single spectra, but through more or less empirical comparison of groups of related compounds: the classic inorganic chemist's approach, in fact. Fortunately, in the presence of such an avalanche of data, the approach was benignly fault tolerant and few conclusions from these years (and none of much consequence) have been overthrown by subsequently more careful mopping up operations.

Despite the fact that Klixbull's approach to collecting spectroscopic data could appear rather cavalier at first sight, he was by no means neglectful of detail. In addition to correlating energies of spectral transitions [6], he was closely interested in features of the band profiles themselves, in particular intensities [7] and lineshapes [8]. Indeed, as early as 1955 we find him commenting on vibrational fine structure in one of the rare cases (actually $ReCl_6^{2-}$) where such a feature is seen in a room temperature solution spectrum [9]. It is fair to say, though, that his attention was not captured strongly by such fine detail so that, for example, when it became possible to record polarised electronic spectra of metal complexes in crystals and hence apply symmetry selection rules, or to observe vibronic structure at low temperature, he did not deploy these techniques in his own work. Still less was he seized by the opportunities presented by magneto-optical spectroscopy (and in particular magnetic circular dichroism) from the early 1970s to define excited state wavefunctions. Klixbull's driving purpose was to use knowledge of the energy levels in inorganic compounds to derive models for their chemical bonding. The very first sentence of the Preface of his first book 'Absorption Spectra and Chemical Bonding in Complexes' [10] proclaims his approach (just as the title itself does):

> Our knowledge of the electron clouds of gaseous atoms and ions is based on the energy levels, studied in atomic spectroscopy. It has not been generally realised among chemists that the study of energy levels, i.e. the absorption spectra, is equally fruitful in helping our understanding of the chemical bonding, not only in transition group complexes, but in every type of molecule.

Such a breadth of purpose led him, right from the outset, both to attempt correlation of spectroscopic parameters with other chemically relevant properties [11] and to go beyond the simplest high symmetry metal complexes like the hexa-aquo-ions into lower symmetry and non-transition-metal compounds. For example, in 1956 he wrote on the influence of the spin-pairing energy and the consequent hump in the ionisation potentials of the 3d elements [12]. He also wrote extensively about 4d and 5d complexes, and their striking differences from the corresponding 3d ones [13,14]. At the same time, he was concerned, with Jannik Bjerrum, by what was then called crystal field stabilisation and the thermodynamics of complex formation in 3d elements [15,16].

5. The nephelauxetic effect

Possibly as a result of his conditioning in early years by an interest in atomic spectra, especially of the 4f-elements, throughout the 1950s Klixbull was greatly preoccupied by the role of electron repulsion in determining the band energies in ligand field spectra [17]. In particular, he drew attention to the fact that the values of the Racah–Cordon–Shortley parameters that gave the best fits to the observed spectra were invariably smaller than in the spectra of the corresponding gaseous ions. In a pseudo-atomic formalism, this implies that the average radius of the shell in question is greater in the coordination complex. Furthermore, for a given metal in a specific oxidation state, the effect varied with the coordinated ligands [18]. With Claus Schaeffer, he placed all the common ligands in a series of increasing propensity to expand the metal d-shell and, after consulting a Professor of Classics at the University of Copenhagen, named it the nephelauxetic (Greek 'cloud expanding') series [19]. In some ways that series, found in all undergraduate textbooks, is his most enduring monument. Earlier, in a nice piece of empirical ordering, he had shown that the spectrochemical series, which measures the ligand field splitting, could be factored into terms due to both the metal and the ligand [20]. Now that a molecular orbital approach has long since taken over from crystal field or hybrid ligand field (let alone Pauling valence bond) models of coordination complexes, it is hard to recreate the confusion and controversy that reigned in this matter through the 1950s and 1960s. There can be no doubt, however, that Klixbull's insights, gained by correlating large numbers of spectra, greatly clarified the picture.

6. Charge (electron) transfer spectra

Harking back to the question above, which clearly announced the breadth of Klixbull's intentions as a spectroscopist, it is no surprise to learn that, in parallel with his immersion in what might appear to be the minutiae of electron repulsion in partly-filled d-shells, he was taking a wide view of molecular bonding by starting out on the first ever exploration into the detailed structure of charge transfer spectra. Looking back at the era in question, it seems somewhat strange that so much intellectual energy was being expended in understanding spectra that resulted from electric-dipole-forbidden transitions while at the same time the transitions that are fully allowed (and hence, in many cases, determine the colours of compounds themselves) were being strenuously ignored. All that can be said is that, at the time, there was a generalised perception that a correlation existed between dark colours and ready oxidation–reduction; assignment of the individual absorption bands to transitions between levels of defined (and verifiable) symmetry had never been attempted. In that context, Klixbull's 1959 paper on the electron transfer spectra of octahedral hexahalogeno-complexes of 4d and 5d elements represented a clear breakthrough [21]. Not only did he lay out in an entirely convincing way how the bonding and non-bonding molecular orbitals of ligand type were ordered, but he assigned the complicated succession of absorption bands to transitions from these levels to the partly-filled d-shells. To do so, he invoked not only electron repulsion effects on multi-centre configurations (apparently for the first time) but also the influence of spin–orbit coupling on the ligands, laying a foundation for much future theoretical work, especially in photoelectron spectroscopy.

Yet, it is entirely characteristic of Klixbull that he followed up this remarkable extension of quantitative theory into new territory, not by further refining the details, which was left to others using the novel techniques of low temperature high resolution spectroscopy and magnetic circular dichroism, but by using the results to derive a set of simple chemical parameters that could be applied to a wide range of compounds, giving insight to non-theorists and the general run of inorganic chemists. The concept of 'optical electronegativity' was founded on Mulliken's definition of electronegativity, but quantified using the electron transfer energies found in the absorption spectra, corrected for electron repulsion [21]. For the halogens, for example, the resulting numbers mapped precisely on to the Pauling scale, but with the big advantage that parameters could be derived precisely, not just for elements but for individual oxidation states, and also for polyatomic groupings such as NCS. Later, in his book 'Orbitals in Atoms and Molecules' the concept was broadened to orbital electronegativity since analysis of the electron transfer spectra enabled the defining parameters to be determined orbital by orbital [22].

7. The angular overlap model

This brief survey of Klixbull's work has taken a more or less chronological approach rather than concentrating on the different classes of spectra that attracted his attention. Thus it is that, following his pioneering venture into charge transfer (or, as he always insisted on calling them, electron transfer) spectra, he returned to his earlier love, ligand field spectra, and embarked on a sustained effort to recast ligand field theory into a model comparable with the molecular orbital model that he believed provided the physically correct basis for rationalising the properties of partly-filled d- and f-shells. One of the great strengths of the electrostatic crystal field model, taken over to a considerable degree by the more flexible ligand field approach, was its ability to treat low symmetry perturbations or mixed ligand environments by adding extra terms to the expansion of the central field around the metal in spherical harmonics. Values of the coefficients describing the contributions of these terms could be found by fitting the spectra, and interpreted through the chosen physical model of the central field. However, given that it was widely recognised by the 1960s that the orbitals concerned were not purely atomic, or even expanded atomic, but actually molecular orbitals containing a non-negligible ligand component, the question arose as to how this physical perception could be built on to the conventional model which retained as much as possible of its elegant simplicity. The result was the 'angular overlap model'.

Worked out and promulgated in close partnership with his long time friend and colleague Claus Schaeffer, the model took as its central simplifying hypothesis the idea that the group metal–ligand overlap integral associated with each symmetry-based molecular orbital could be factored into a product of a radial and an angular part [23]. This was tantamount to assuming that the angle subtended by the ligand orbitals at the metal centre could be neglected—a surprisingly good and exceptionally useful approximation. Because of its relative simplicity in treating a very diverse range of problems that were directly interesting to inorganic chemists, the angular overlap model was taken up widely and enthusiastically. To this day, it continues to be a most helpful component in the coordination chemist's toolbox.

8. The Cyanamid Institute in Geneva

The body of work that we have just been surveying occupied essentially the decade of the 1960s. It was carried out, following Klixbull's departure from the University of Copenhagen, in the highly congenial and productive environment of the Cyanamid European Research Institute (CERI) just outside Geneva. This was such an unusual, and remarkably effective unit for generating new science, that it deserves a few words of description. During the later 1950s, a number of US chemical companies found it both convenient and cost effective to open small basic research laboratories in Europe, for example Union Carbide in Brussels and Monsanto in Zurich in addition to CERI. The concept of CERI, set up by American Cyanamid Company in a refurbished and extended banker's villa on the shores of the Lac Leman, was especially innovative. The Institute was diverse but overall quite small: six groups, each with only three scientists, covering fields where, the company's research management felt, there could be future opportunities for product development. Thus, organophosphorus chemistry rubbed shoulders with crystal growth of new compound semiconductors, organometallic chemistry and organic electrochemistry. It is not quite clear what product area Klixbull's expertise matched, but most probably it was inorganic pigments and luminescent materials. In any case, looking back, the institute was clearly intended at least in part as an advertisement for the company as an organisation operating at the boundaries of current science.

Klixbull's group was labelled 'theoretical inorganic chemistry', although much experimental work was undertaken, and coordination chemists of the older generation who found themselves on the CERI mailing list will recall the steady rain of envelopes through their mailboxes, bearing weighty preprints that carried the serial identification CERI-TIC followed by a rapidly rising number. In such diverse company, and with few administration constraints, Klixbull was in his element.

The Institute was a few miles outside the centre of Geneva, effectively in the countryside, so most members of staff stayed on site at lunchtime to use the small cafeteria converted from what had been a gatekeeper's cottage. Such lunches were an invaluable and (at least to an impressionable young graduate student) unforgettable forum for scientific discourse and speculation, starting with the soup and often lasting well into the afternoon. Whilst Klixbull's technique at a blackboard was to cover every inch of its surface systematically with formulae and sketched spectra (often in numerous colours), his custom of writing on the Formica-topped cafeteria tables with a pencil was broadly similar. Indeed, from time to time he found it necessary to migrate with the company between courses to another table, having filled the surface of the first one with diagrams.

But nemesis was at hand. In the late 1960s Cyanamid became involved in a legal battle that proved expensive for the company, so economies were sought. As is so often the case, long-range research was the first to suffer and CERI closed after 10 years of extraordinarily productive lifetime. Several senior scientists, including Klixbull, became Professors in the University of Geneva and began new careers as academics. For him, it was undoubtedly a scientific watershed.

9. The University of Geneva: photoelectron spectroscopy, luminescence and other topics

Coincident with his new beginning in the University, Klixbull began to develop an interest in the then new field of photoelectron spectroscopy which, he felt, offered insights into chemical bonding in metal complexes analogous to, but potentially more extensive than, those furnished by visible–UV absorption spectroscopy 20 years earlier. Having purchased the equipment, he attacked the subject with the same vigour that he had brought to bear on ligand field spectroscopy, and with a similar approach. That is, he quickly recorded large numbers of spectra of sets of related compounds and began to correlate them. Data cascaded out, as evidenced by the title of one publication 'Photoelectron Spectra Induced by X-rays of Above 600 Non-metallic Compounds Containing 77 Elements' [24]. Chemical shifts due to change of oxidation [25] and fine structure due to electron repulsion of the core hole state and partly filled d-shell [26] were identified. Unfortunately, however, there were problems. Certainly these were known to Klixbull, but perhaps were not treated by him with the seriousness that less imaginative and more pedantic experimenters brought to them. The first was surface charging. The coordination complexes and simple inorganic compounds of greatest interest to him were insulators, and while efforts were made to extrapolate results back to zero irradiation [27], doubts remained about precise energy calibrations. Another difficulty that makes photoelectron spectra more difficult to interpret than visible–UV spectra is the effect of the X-ray beam on the sample surface. Degradation is common and needs careful evaluation, only with difficulty compatible with the 'broad brush' approach preferred by Klixbull. Nevertheless, a large volume of publications attests to his efforts in this field; those that have had the greatest influence are probably the ones concerned more with concepts than data [28].

Towards the end of his research career Klixbull returned to his first love in optical spectroscopy, the lathanides and actinides, especially through a long and fruitful collaboration with Renata Reisfeld, concentrating especially on luminescence [29] and energy transfer [30], the latter frequently connected with issues relating to solar energy conversion [31]. The actinides (in particular the uranyl ion) preoccupied him [32], while a flash of his old enthusiasm for data collection can be discerned in an article on Mn^{II} emission in no fewer than 24 different phosphate glasses [33]. The frontiers of his erudition continued to broaden, and he attempted to apply some of the principles of atomic spectroscopy that he had learned first many years before, including arguments based on symmetry, to the structure of nuclei and elementary particles [34]. However, it is open to doubt whether any of these unconventional ideas found their way into the mainstream thinking of particle theory, although one at least found a place in the pages of Nature [35].

Throughout his life Klixbull had always taken a close interest in the history of science, not just as a further exercise in recondite fact finding, but as an essential tool for understanding how present concepts and approaches have developed and, above all, as a source for his ever present love of analogy. He was particularly delighted to draw attention to the discovery of Gd and Yb by Marignac in the laboratories of the University of Geneva [36], but in the title of his contribution on Marignac to the volume marking the century of the Geneva Chemistry Department [37], perhaps one may catch an echo of Klixbull's own situation: 'the solitary pioneer'. There can be no doubt that in the latter part of his life Klixbull, a naturally loquacious (indeed garrulous) and gregarious figure became increasingly isolated.

The early death of his wife Micheline in 1978 was not only a deep personal tragedy in itself but, for one not much concerned with everyday domestic matters, made life difficult. In the University, his unusual style and manner of working did not attract many graduate students, and while his pedagogical approach was rich in insights for those who were already familiar with the subjects he taught, less motivated students found his lectures too challenging to be easily accessible. In his books and articles, his arguments often developed through sequences of quite convoluted analogy, buttressed by dense arrays of references, frequently to obscure sources. The latter was the result of voracious reading and a remarkably retentive memory. Colleagues soon found that, when searching for a reference, it was often quicker to ask Klixbull for the volume and page number than to go to the library. In the end, perhaps it could be said that erudition was his greatest enemy in making contact with the wider community of scientists.

Klixbull's lasting memorials to science are the nephelauxetic series, the angular overlap model and optical electronegativity. To those who knew him, or saw him in action, however, the lasting impression is of one consumed by an urge to know; to gather, sift and synthesise facts and extract from them models of how the world works. Is there a better epitaph for a scientist?

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