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Inorganic Experiments, 2nd Edition

edited by J. Derek Woollins

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reviewed by Daniel Rabinovich

There are not a whole lot of manuals aimed at senior-level undergraduate laboratory courses in inorganic chemistry. Only four were published in the 90s: Szafran, Pike, and Singh's *Microscale Inorganic Chemistry* (1), the 3rd edition of Angelici's classic *Synthesis and Technique in Inorganic Chemistry* updated by Girolami and Rauchfuss (2), Tanaka and Suib's *Experimental Methods in Inorganic Chemistry* (3), and the 1st edition of Woollins' *Inorganic Experiments* (4). While this shortage is arguably a consequence of the tendency by many instructors to simply design such courses based on personal experience or experimental procedures reported in this *Journal* or in serials such as *Inorganic Syntheses*, it is clear that the need for innovative, stimulating experiments and texts remains.

The first edition of *Inorganic Experiments* has been completely revised and enlarged (more enlarged than revised, I would say) and it continues to exhibit several features that distinguish it from its current market competitors. It is now a collection of almost 90 experiments written by a group of 63 international contributors (about half of whom are in the UK). While this is not necessarily an advantage over books written by two or three authors, it does reflect an effort to draw experiments from individuals with a diversity of backgrounds and research interests. Perhaps more importantly, it is still the only inorganic laboratory manual that contains separate sections for introductory, intermediate, and advanced experiments. Almost 100 additional pages and 24 new experiments (eight in each section) have been added to this new edition, thus giving instructors even more options to choose from for selected topics or to adjust the course to time or enrollment constraints.

The 2nd edition of *Inorganic Experiments* starts with a brief introduction to the expository (skills-oriented) and investigative (discovery-oriented) approaches to laboratory instruction (5). Since much has been written about the benefits of inquiry-based or discovery-oriented laboratory experiences (6), it is fortunate that Woollins' book follows this model in most of its experiments. Chapter 2 (Introductory Experiments) deals with the preparation of various simple inorganic compounds and coordination complexes, including iron(III) oxalate complexes, interhalogen compounds, and siloxane polymers. Even straightforward experiments have a good pedagogic value when presented in the right context. For example, the preparation of copper(I) iodide from its elements

serves to illustrate fundamental concepts such as stoichiometry and limiting reactants, solubility, and redox reactions. My favorite experiment is probably the preparation of the potassium salt of the tris(3,5-dimethylpyrazolyl)borate anion, one of the most popular "scorpionate" ligands in coordination chemistry. Starting with a molten mixture of KBH_4 and the free pyrazole, the progress of the reaction is monitored by collecting the liberated H_2 gas in an inverted graduated cylinder filled with water and the whole procedure is, in my own experience, very appealing to students.

Chapter 3 contains 31 experiments of intermediate complexity. While "classic" coordination chemistry is still represented by the syntheses of a number of acetylacetonate, dinitrogen, and phosphine complexes, organometallic chemistry is the predominant theme in this chapter. From the syntheses of ferrocene and some of its derivatives to the preparation of a tungsten alkylidyne complex, many procedures in this chapter also require the handling of air-sensitive compounds (e.g., Grignard and organolithium reagents). Among my favorite experiments are the synthesis and multinuclear NMR characterization of $\text{trans-PtHCl(PPh}_3)_2$ and the study of the paramagnetism of iron(III) dithiocarbamate complexes in solution by the Evans method.

Even more challenging are the 33 experiments included in Chapter 4, some of which rely on the use of more specialized techniques (e.g., electron paramagnetic resonance (EPR) spectroscopy) or more esoteric reaction conditions (e.g., liquid ammonia as a solvent). In fact, I suspect that a few of the experiments in this chapter will probably be too complex to implement in most undergraduate settings even if the necessary equipment, a generous budget, and qualified instructors are available. A case in point is the synthesis of the fascinating 17-electron hexacarbonyl V(CO)_6 , which starts with the preparation of a vanadate precursor in an autoclave charged with vanadium trichloride, sodium sand, and 200 atm of CO. Not precisely your everyday synthesis of copper(I) iodide! But there is no question that many of these advanced experiments are aimed at bringing contemporary research topics to the undergraduate arena: the synthesis of a high-valent manganese imido complex, the investigation of metal-containing liquid crystals, and the coordination chemistry of organic free radicals are just three additional examples.

The book has been for the most part carefully produced and is nicely organized, including a five-page subject index, and I've detected only a handful of typographical errors (e.g., "piperidene" instead of "piperidine" [p 144]) or mistaken references (e.g., the year of publication of the fourth reference in p 198 is 1999 not 1998). However, as is often the case with edited monographs, the thoroughness or style of the experimental write-ups is not uniform. For example, not all the experiments contain a list of questions or exercises for further consideration and only a few include suggestions for the preparation of reports. Similarly, whereas some experi-

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ments include numbered references and relevant comments, others show only incomplete citations, and yet others don't mention any references at all.

As much as I personally like this book and believe it is a great resource for instructors, I cannot say it would be my top choice as a lab manual for undergraduate students. I still prefer Girolami, Rauchfuss, and Angelici's book (2), which has a shorter list of experiments to choose from but, unlike Woollins's text, has also good introductory sections on safety, common laboratory practices, and the organization of laboratory notebooks. In addition, it has better guidelines for reports and independent studies and a more comprehensive bibliography associated with each experiment. In the same vein, Szafran, Pike, and Singh's manual (1) also incorporates nice individual chapters on safety, equipment, common microscale techniques, spectroscopic methods (almost 40 pages!), and a more balanced roll of experiments, including several on main group and bioinorganic chemistry. In summary, *Inorganic Experiments* is an eclectic but very attractive compilation of experiments in modern inorganic chemistry, and I highly recommend it to instructors of undergraduate inorganic laboratory courses and to other professionals with a general interest in synthetic inorganic or organometallic chemistry.

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