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UPDATED INORGANIC AND ORGANOMETALLIC LABORATORY COURSE FOR JUNIOR CHEMISTRY STUDENTS

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ABSTRACT: An advanced inorganic and organometallic laboratory programme has been worked out for junior students. The laboratory exercises were selected to provide an introduction for the students to the different techniques and compound classes of inorganic and organometallic chemistry. Emphasis was also put on the improvement of the students' verbal and written communication skills through laboratory reports and short talks. The student responses to the programme consisting of twelve groups of experiments were favourable. [*Chem. Educ. Res. Pract. Eur.*: 2000, 1, 179-182]

KEYWORDS: *university education; inorganic chemistry; organometallic chemistry; laboratory practice*

INTRODUCTION

In the chemistry curriculum at the Eötvös University (Budapest, Hungary) an advanced inorganic and organometallic laboratory course is included in the educational programme. This has been motivated by the fact that in the inorganic chemistry laboratory course of the second semester emphasis is put only on basic preparations and laboratory techniques. The course is generally taken by junior students in the sixth semester and it extends over seven laboratory periods (each of eight hours length). Previous studies in inorganic and organic chemistry, as well as lectures in structural and organometallic chemistry provide sufficient background to this laboratory course.

The substantial increase in the number of chemistry students entering our Faculty prompted us to reorganise the course. The modernisation was also motivated by the fact that organometallic chemistry as one of the most rapidly growing area of chemistry should play pronounced role in the university education (Miessler, 1991; Elschenbroich, 1992; Spessard, 1996). Special attention was paid both to professional and technical details. Orienting principles were as follows: a) experiments should draw the students' attention to the synthesis and application of modern materials; b) the programme should provide the students with a detailed insight into the different laboratory manipulation techniques; c) exposure to chemicals should present only minimal safety and environmental hazard; d) verbal and written communication skills of the students should be strengthened; e) the programme should be economic. The updated programme is based on the experience we have collected by guiding the course during the previous academic years [Borossay, 1990], on literature search, and on our own research experience in the field of organometallic chemistry.

The main features of the programme consisting of 12 groups of experiments are summarised in Table 1.

RATIONALE

When selecting the exercises we had the following factors to consider: scale of the preparation, applied techniques, the involved compounds and the practical (industrial) aspects of the process.

1) Because the students already acquired the basic laboratory skills and techniques at an earlier stage, we usually chose microscale preparations. Besides the advantage of teaching the students the skills associated with microscale preparations and forming an environmental awareness in the student, these processes are cheaper to perform and also produce less potentially harmful waste and product.

2) Special emphasis was put on broadening the student's synthetic arsenal by the introduction of novel techniques such as: electrochemical synthesis, conventional inert techniques, vacuum line preparations, photochemistry, microwave irradiation assisted transformations and chemical vapor deposition.

3) When selecting the exercises, we aimed to utilize and prepare a broad spectrum of chemicals in order to broaden the student's experience. Reagents included dissolved metal in liquid ammonia, Grignard reagents, organolithium compounds, sandwich compounds and reactive metal powders. The products consisted of transition metal complexes, complex inorganic salts, sandwich compounds, hard carbide layers and functionalised organic molecules.

4) In a number of cases, the organometallic reagents were used to transform organic molecules. Some of the cases (e.g. lithiation) demonstrated the applicability of these reagents, while in other cases processes of industrial relevance were carried out on a small scale (Wilkinson hydrogenation, Wacker oxidation). In each case, the students' attention was brought to the advantage of the applied reaction compared to the alternative classical transformations.

In addition to extending the laboratory skills of the students, we also aimed to improve their communication skills. Before each laboratory session, the students had to give a brief summary of the day's planned work, proving their preparation and in depth understanding of the task ahead, as well as being familiar with potential hazards of the operations involved. After each session, the students had to write a report on the exercise, which (along with their performance in the lab) was assessed by their supervisor.

At the beginning of the semester, the students were assigned an individual problem (usually preparation of a compound). They had to devise a synthetic route based on literature procedures or their analogies, and carry out the outlined synthesis. They had to write a report on their project (describing the background of the problem and their practical work) and also to give a short talk (10 min) on the subject, which was always followed by discussion.

The laboratory course was accompanied by a series of talks on the background of the different synthetic methodologies.

TABLE 1. *The list of experiments involved in the programme*

Topic	Title	Related studies	Equipment
Electrochemical reactions – cathodic reduction	Preparation of $\text{NH}_4\text{V}^{\text{III}}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ and $(\text{NH}_4)_2\text{V}^{\text{II}}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	electrochemical fundamentals; oxidation states of V	electrochemical cell, inert gas (CO_2)
Study of molecular oxygen fixation	Preparation and study of a cobalt(II) oxygen adduct complex	structure and reactivity of cobalt–salen complex	microscale glassware
Synthesis of metal carbonyls	Synthesis and study of nickel tetracarbonyl	preparation and properties of metal carbonyls	standard laboratory glassware
Reduction by using dissolving metal	Reduction of anisol by lithium – Birch-reduction	synthesis in liquid NH_3 ; reduction of aromatic systems	basic inert atm techn.
Synthesis of organometallic compounds by using anionic alkylating agent	Synthesis of triaryl phosphines	Grignard reaction	standard laboratory glassware
Transition metal „sandwich” compounds	Synthesis of ferrocene and acetyl ferrocene	electronic structure and reactivity of sandwich complexes	microscale glassware
Use of organometallic reagent in organic synthesis	Synthesis of thiophene carboxylic acid	reactivity of organolithium compounds	basic inert atm. techn. (balloon)
Preparation and reactions of transition metal organyls	Organometallic chemistry of molybdenum,	properties of the Mo–Mo bonds	vacuum line Schlenk techn.
	Synthesis and study of cobalt clusters	structure and reactivity of cobalt clusters	vacuum line Schlenk techn.
Transition metal catalysis	Synthesis and Use of Wilkinson’s Catalyst	organometallic catalysis: hydrogenation; the chemistry of rhodium	microscale glassware
	Modelling of Wacker Process	organometallic catalysis: olefin oxidation	standard laboratory glassware
Chemical Vapour Deposition	Production of chromium carbide hard coating	CVD, OMCVD; decomposition of organometallic compounds	vacuum system hot wall CVD reactor
Organometallic photochemistry	Synthesis and reactivity of cis- $\text{Cr}(\text{CO})_4(\text{CH}_3\text{CN})_2$	organometallic photochemistry; ligand exchange reactions of Cr-, Mo- and W-carbonyls	UV reactor. inert atm., Schlenk tech.
Reactions in MW field	Heck reaction	MW assisted chemical reactions	MW oven
	Preparation of ferrocenyl oximes		Teflon reactor

CONCLUSIONS

We believe that the laboratory course broadens substantially the chemical knowledge and manipulative skills of our students and draws their attention to the different aspects and applications of modern inorganic and organometallic chemistry. For example, the exercises related to catalytic cycles (such as the Wacker process or homogeneous hydrogenation) clearly demonstrate that organometallic compounds form useful catalysts which are of significant industrial interest. Furthermore, there are several instances where organometallic chemistry is involved in organic synthesis and biochemical reactions. Finally, coverage of applications of inorganic and organometallic precursors to thin surface film depositions is an exciting aspect of the programme for students who are interested in the study of materials science.

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