

https://dspace.org/

Solventless substitution chemistry of iron and ruthenium metal carbonyl complexes

Munyaneza, Apollinaire

2008-03-06T10:24:52Z
http://hdl.handle.net/10539/4560

Downloaded from a DSpace repository. https://dspace.org/

CHAPTER 3. EXPERIMENTAL PROCEDURES AND TECHNIQUES USED IN THE STUDY

3.1. GENERAL WORKING CONDITIONS

The reactions were conducted using a dual manifold line with nitrogen and vacuum lines connected by 2-way ground glass taps. Nitrogen was passed through a glass tube containing 4 Å molecular sieves and blue copper sulfate grains serving as an humidity indicator. When the colour of the copper sulfate turned from blue to yellow, the grains were regenerated by heating them in an oven until the blue colour was recovered. The flow rate of nitrogen was monitored by a paraffin oil bubbler that was connected to a nitrogen line [1] (figure 3.1).



Figure 3.1. Manfold nitrogen line

A = Nitrogen line

B = Glass tube containing molecular sieves and copper sulfate

C = Paraffin oil bubbler

D = Tube connecting the system to a vacuum pump

Generally a three-necked round bottomed flask was used for synthesis purposes while NMR tubes were used in the experiments involving the monitoring of a reaction.

3.2. SOLVENT PURIFICATION

Solvents used in the synthesis of the starting materials or in the isolation of the products were dried and deoxygenated before use. Solvents were first pre-dried using 4 Å molecular sieves for at least 24 hours in a reagent bottle. The solvent was then distilled from a suitable drying medium under nitrogen flow [2].

3.3. CHARACTERIZATION TECHNIQUES

The following techniques have been used to characterize compounds throughout the study.

3.3.1. Nuclear Magnetic Resonance (NMR) spectroscopy

Nuclear Magnetic Resonance spectroscopy is a useful tool for the characterization of chemical compounds [3]. NMR spectroscopy was used to confirm the success of the synthesis of the starting material, to identify the products formed and to monitor the reaction in order to get information about a particular system under study.

^{1}H- and ^{31}P-NMR spectra were recorded on a Bruker AC 300 spectrometer at 300 MHz and 121.5 MHz respectively. All spectra were recorded in chloroform-d (99.8 atom % D, containing 0.03 % v/v trimethylsilane) and referenced to tretramethylsilane (TMS), on the δ-scale. Mestrec application was used to process the spectra.

3.3.2. Infrared (IR) spectroscopy

This technique gives information about the structure of a compound [4]. The spectra were recorded on a Bruker Vector 27 FTIR spectrometer using OPUS software. Generally, dichloromethane was used to dissolve the material. The transmission spectra were recorded on the samples. The pure solvent was used as a blank and its spectrum recorded before that of the actual sample.

3.3.3. Differential Scanning Calorimetry (DSC)

Depending on the material under study, DSC can be used in a number of applications: phase diagram of polymers, heats of reaction measurements of metal salt hydrates, melting point measurement for most compounds, purity determination for biological materials, to name a few [5]. The melting points of our compounds were determined using a Mettler Toledo DSC822e instrument. Between 4-8 mg of the sample were loaded into an aluminium pan and heated at a heating rate of 10°C/minute. Indium was used in the calibration processes.

3.3.4. Mass Spectrometry (MS)

Mass spectrometry is a technique used to generate ions from either inorganic or organic compounds by any suitable method, to separate these ions by their mass-to-charge ratio (m/z) and to detect them qualitatively and quantitatively by their respective m/z values and abundance [6]. A VG 70 SEQ mass spectrometer with a resolution of 1000, at a scan rate of 2 secs/decade (external), was used. The Fast Atomic Bombardment (FAB) method was found to be suitable for our compounds.

3.3.5. X-ray Diffraction (XRD).

XRD is the technique which gives the most credible information about the structure of a particular compound. Crystallography provides the most direct way of forming three dimensional images of molecules [7, 8]. Crystal structures have been determined using a Bruker SMART CCD area – detector difractometer. The SHELXTL program was used to solve the structure while SHELXL97 was used in the refinement processes.

3.3.6. Optical microscopy

In order to obtain information about the phenomenon taking place at the microscopic level between reactants, optical microscopic experiments have been undertaken. Pictures were taken using a microscope directly connected to a digital camera and a PC (figure 3.2).

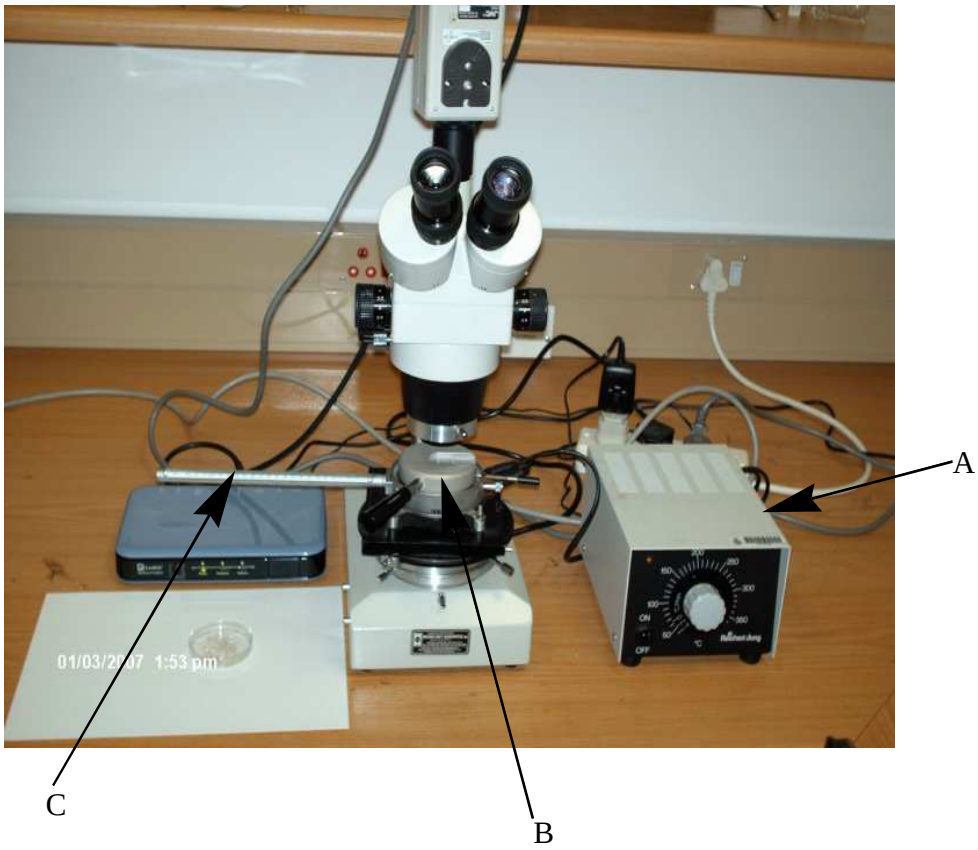

Figure 3.2. Optical microscope

A = Heat controller

B = Sample holder

C = Thermometer

3.4. REFERENCES

1. R. J. Errington, *Advanced Practical Inorganic and Metalorganic Chemistry*, Backi Academic & Professional, London, 1997
2. D. D. Perrin, W. L. F. Armarego and D. R. Perrin, *Purification of Laboratory Chemicals*, Pergamon Press, Oxford, 1980
3. J. W. Akitt, *NMR and Chemistry, An Introduction to Modern NMR Spectroscopy*, Chapman & Hall, London, 1992
4. P. R. Griffiths and J. A. de Haseth, *Fourier Transform Infrared Spectrometry*, John Wiley & Sons, New York, 1986
5. W. W. M. Wendlandt, *Thermal Analysis, John Wiley & Sons*, New York, 1986
6. J. H. Gross, *Mass Spectrometry*, Springer, Berlin, 2004
7. D. Blow, *Outline of Crystallography for Bbiologists*, Oxford University press, New York, 2002
8. J. P. Glusker, M. Lewis and M. Rossi, *Crystal Structure Analysis for Chemists and Biologists*, VCH, New York, 1994