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Microwave Synthesis of *cis*-Dichlorotetrakis(dimethylsulfoxide)ruthenium(II)

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The *cis*-RuCl₂(dms_o)₄ compound has been shown to be a versatile starting reagent for the preparation of numerous Ru complexes (Evans et al. 1973, Alessio et al. 1991, Landgrafe and Sheldrick 1994). *cis*-RuCl₂(dms_o)₄ is prepared by refluxing RuCl₃·xH₂O in DMSO for 5 min followed by concentration to half volume and subsequent precipitation with acetone (Evans et al. 1973). Due to the high boiling point of DMSO (189 °C), actual reaction time approaches 3 hr. The reduction in volume is also critical, as too little reduction leads to reduced yields. The difficulties in the synthesis of *cis*-RuCl₂(dms_o)₄ and the length of reaction time led us to explore the use of microwave technologies for improving the preparation of the title compound.

The starting materials, RuCl₃·xH₂O and dimethyl sulfoxide, were purchased from Aldrich Chemical Company. All reagents were used as purchased, without further purification. An Agilent 8453 UV-visible Spectroscopy system and a Midac IR spectrophotometer were used for the analysis of the compounds. The microwave synthesis was performed using a CEM Discover microwave reactor.

For the microwave reaction, 0.10 g (0.38 mmol) RuCl₃·xH₂O was added to 2 mL DMSO in a microwave reaction tube under N₂ or Ar and heated to 135 °C for 10 min. Upon standing, a yellow precipitate formed and was isolated by filtration. The product was washed with acetone and air-dried, yield = 49%, melting point = 193 °C. The visible and infrared spectra for our product matched those reported in the literature (Evans et al. 1973, Alessio et al. 1988). Reaction times can be decreased to three min. by increasing the surface area inside the microwave reaction tube.

Though the yield from the microwave reaction is lower than the reported literature yield (Evans et al. 1973), decrease in reaction times, lower energy expenditures and reduction in waste due to the use of smaller solvent volumes makes the microwave synthesis a viable alternative to the conventional preparative method for *cis*-RuCl₂(dms_o)₄.

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Literature Cited

- Alessio E, G Mestroni, G Nardin, WM Attia, M Calligaris, G Sava, and S Zorzet.** 1988. *cis*- and *trans*-dihalotetrakis(dimethylsulfoxide) ruthenium(II) complexes (RuX₂(DMSO)₄; X = Cl, Br): synthesis, structure, and antitumor activity. *Inorganic Chemistry* 27(23):4099-106.
- Alessio E, G Balducci, M Calligaris, G Costa, and WM Attia.** 1991. Synthesis, molecular structure, and chemical behavior of hydrogen *trans*-bis(dimethyl sulfoxide)-tetrachlororuthenate(III) and *mer*-trichloro-tris(dimethyl sulfoxide) ruthenium(III): the first fully characterized chloride-dimethyl sulfoxide-ruthenium(III) complexes. *Inorganic Chemistry* 30(4):609-18.
- Evans IP, A Spencer, and G Wilkinson.** 1973. Dichlorotetrakis(dimethylsulphoxide)-ruthenium(II) and its use as a source material for some new ruthenium(II) complexes. *J.C.S. Dalton Trans* 1973:204-9.
- Landgrafe C and WS Sheldrick.** 1994. Structure and reactions of the thioether half-sandwich ruthenium(II) complexes [Ru(MeCN)₃([9]aneS₃)] [CF₃SO₃]₂ and [Ru(MeCN)₂(PPh₃)([9]aneS₃)] [CF₃SO₃]₂ ([9]aneS₃ = 1,4,7-trithiacyclononane). *J.C.S. Dalton Trans* 1994:1885-93.