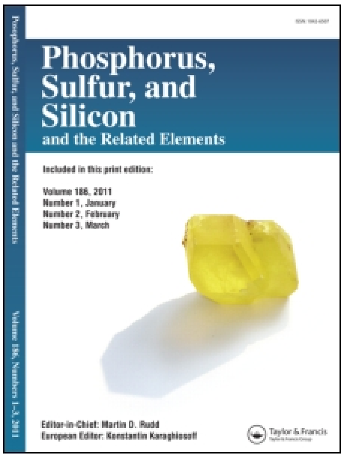

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Chromium Tricarbonyl Compounds of Phenylcyclopolysilanes

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CHROMIUM TRICARBONYL COMPOUNDS OF PHENYLCYCLOPOLYSILANES

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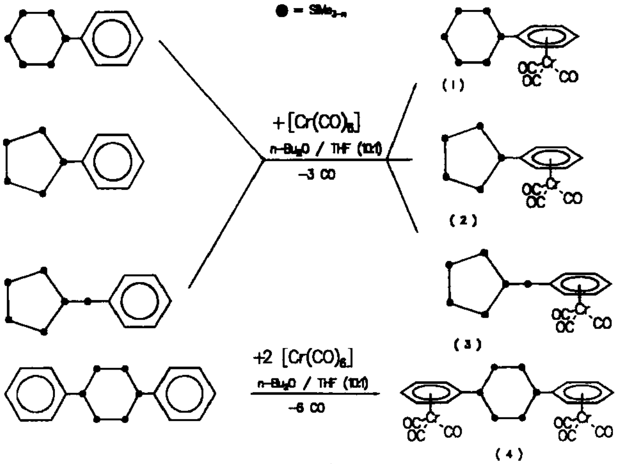
Abstract Syntheses of tricarbonyl chromium complexes of phenylcyclopolysilanes and their application to separate isomers.

Introduction

Besides some examples of chromium silicon complexes with direct Cr-Si bond there exists a variety of tricarbonyl chromium η^6-phenyl silanes. Most of these derivatives have monosilyl groups at the phenyl ring [1] but there are also a few species with linear polysilanyl framework [2]. We prepared some novel cyclopolysilanyl derivatives and established their physical properties. In the course of this study we discovered a possibility for separating the isomeric cyclopentasilanyl compounds by means of fractional crystallization of their tricarbonyl chromium complexes.

Syntheses

All starting materials were synthesized by standard methods [3,4,5]. The chromium complexes were prepared by thermal replacement of the carbonyl groups in a refluxing solvent mixture of THF/n-Bu$_2$O (1:10) (bp =110°C) during three days [6]. The final products were recrystallized from n-pentanes.



^{29}Si-NMR

A comparison of the chemical shifts of starting compounds (Ph) with the chromium complexes exhibits a small but noticeable deshielding effect caused by the electron withdrawing tricarbonyl chromium group. (illustrated by the shift difference)
All compounds were measured using an INEPT pulse sequence. The chemical shifts are related to TMS in dry THF.

Separation of Isomers

A mixture of the two isomers 1,3- and 1,2-diphenyl-dimethylsilyloctamethylcyclopentasilane was reacted with [$Cr(CO)_6$] (fig.2). Multiple crystallization from n-pentanes gave pure crystalline 5A, whereas the 1,3 derivative was accumulated in the extracts. We were able to determine the position of the Si-atoms of 5A by the connectivities of Si-Si coupling constants applying an INEPT-INADEQUATE pulse sequence. Since the chromium complexes can easily be removed by oxygenative cleavage this method seems to be a promising new way to the not easily accessible 1,2 isomer of diphenyldimethylsilyl-octamethyl-cyclopentasilane.

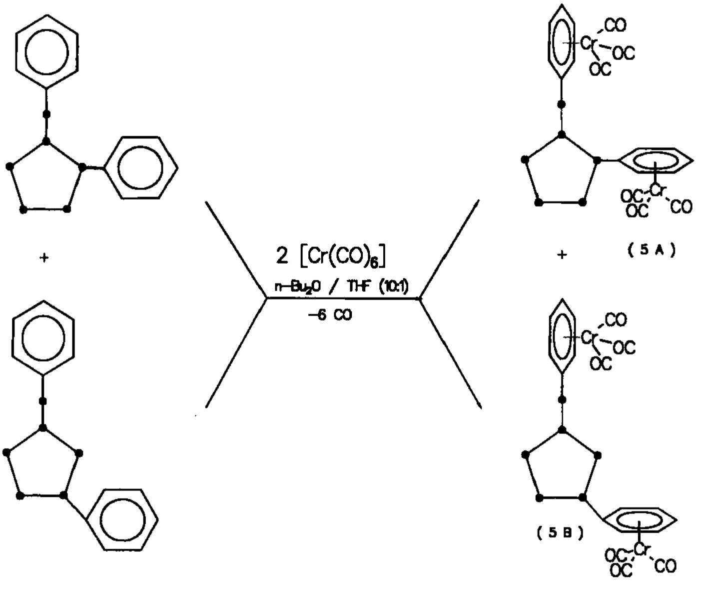


fig.2

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