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A New Approach to [60]Fullerene Ferrocenyl Derivative

Yuan Yin CHEN*, Xin Hong LI

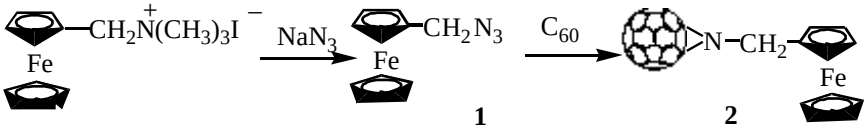
College of Chemistry and Environmental Science, Wuhan University, Wuhan 430072

Abstract: A new approach to fullerene ferrocenyl derivative has been made. [60]Fullerene reacts with ferrocenylmethyl azide to give N-ferrocenylmethyl imino[60]fullerene in 42% yield.

Keywords: C_{60}, ferrocenyl, azide, synthesis.

It is well known that fullerene (C_{60}) possesses strong electron acceptor characters[1]. It can accept reversibly up to six electrons[2-3]. Accordingly, many investigators try to link covalently different electron donors to C_{60} to design molecular electron devices [4-9]. Ferrocene is a rich electron-donor, it is not surprise that fullerene ferrocenyl derivative plays a relevant role in the design of molecular electron devices. A series of fullerene ferrocenyl derivatives have been synthesized *via* the cycloaddition of azomethine ylides to C_{60}, [6-9]. We wish to report a new approach to synthesize fullerene ferrocenyl derivative. Compound **2** was synthesized by reacting of fullerene with ferrocenylmethylazide **1** in toluene as shown in the **scheme**:

Scheme



1 **2**

Ferrocenylmethyl azide **1** was prepared according to literature[10]. A mixture of 54 mg of C_{60} (0.075 mmol) and 18 mg (0.075 mmol) of ferrocenylmethyl azide **1** was stirred in 35 ml of dry toluene under argon at reflux temperature for 42 h. After removing the solvent under reduced pressure, the residue was passed through a silica column (petroleum ether/toluene, 2:1, V/V). 23 mg of target compound **2** was obtained in 42% yield. UV-Vis: λ_{max}（cyclohexane）: 217.5，235.5，276，266, 428 nm; IR ν (cm^{-1},KBr): 3100, 2918.1, 1429.2 (C_{60}), 1190.0 (C_{60}), 1103.2 (Fc), 999.1 (Fc), 571 (C_{60}), 522.7 (C_{60}). ^{1}HNMR (300MH$_Z$, $CDCl_3/CS_2$): δ4.08-4.30 (m, 9H), 1.42 (s, 2H); Anal calcd for $C_{71}H_{11}FeN$: C 91.47, H 1.12, N 1.42; found: C 92.02, H 1.20, N 1.51; *m/z* (FAB): 720 (C_{60}); Mn (VPO): 882±50（The calculated molecular weight of monoadduct is 933）.

We only observed the peak of the pieces of C_{60} but no molecular ion peak in the

FAB-MS spectra. Maurizio P. *et al*[6] reported the same result. We tried to use the VPO for determining the molecular weight of compound **2**. The result fitted the calculated value of monoadduct within the limit of error. Thus, VPO can be used as a complement tool to determine the molecular weight of fullerene derivative.

The TG analysis of compound **2** was carried out under nitrogen flow at 30 ml / min rate. It was observed that the weight increased 1% at 235°C. A possible explanation is that the nitrogen was inclused into the cage of C_{60} at high temperature. It losed 13% of weight from 265 to 275° C, and then losed other 62% of weight with increasing the temperature. So the conclusion can be made that the decomposition temperature of compound **2** is 265° C.

In summary, we have synthesized a new ferrocenyl derivative of C_{60} by a new approach and its structure has been confirmed by FT-IR, UV-VIS, FAB-MS, ^{1}HNMR spectra and VPO. Its themostability is moderate. Its electrochemical activities would be investigated on progress.

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