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SYNTHESIS OF A NEW N_3S_5-THIAAZACROWNETHER

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ABSTRACT

A new N_3S_5-thiaazacrown ether was synthesized by high dilution method with a good yields. The reaction condition was mild and the products were easy to be purified. The structure of this new crown ether was characterized by IR, MS, NMR, and elemental analysis.

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INTRODUCTION

Macrocycleshave been widely used in the field of coordination (Schneiderand Yatsimirsky, 2014). Among them crown ethers containing nitrogen and sulfur donor atoms (i.e. azathiacrown ethers) are of special interest as they exhibit extremely high affinities towards heavy metal ions (Zhang *et al*. 2011; Sang *et al*. 2012).Manykinds of crown ethers have been synthesized and used as ionophores and fluorophores for the detection of environment targets (Bühlmann *et al*. 1998). Amongthe developed synthetic methods,the high dilution method is most popular (Zhang *et al*. 2010). Kept this in mind, a new N_3S_5-thiaazacrown ether was synthesized and characterized.

Experimental Section

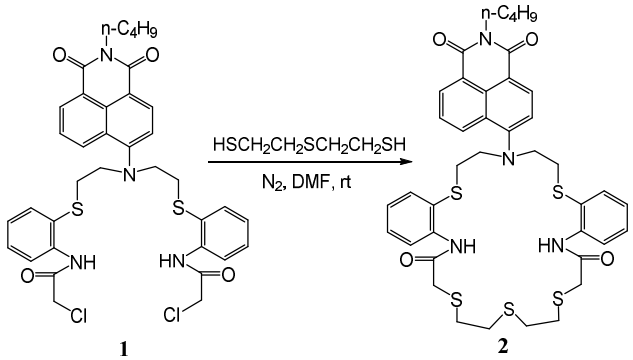
Reagents and Instruments: All of the materials were analytical reagent grade and used without further purification. Infrared (IR) spectra were recorded on KBr pellets using a Perkin–Elmer 1430 spectrometer. Nuclear magnetic resonance (NMR) spectra were measured with a Brucker WM-300 spectrometer, and chemical shifts were given in ppm from tetramethylsilane. Mass spectra (MS) were recorded on a Thermo TSQ Quantum Mass Spectrometer. Elemental analyses were performed with aVarioIII elemental analyzer.

Synthesis

The synthesis route was shown in Scheme 1.

A solution of 1(Zhang *et al*. 2012)(0.5 mmol) in DMF (50 mL) and that of $S(CH_2CH_2SH)_2$(0.5 mmol) in DMF (50 mL) were added simultaneously to a solution of DMF (50 mL) containing 2 mmol anhydrous Na_2CO_3.The reaction was monitored by thin layer chromatography [petroleum ether–ethyl acetate (4:1)]. The whole process was operated under nitrogen atmosphere with vigorously stir for 8 h. The resulting mixture was cooled to room temperature and poured into ice water. The precipitate so obtained was filtered and washed in turn with water, ethanol and diethyl ether and then dried in vacuum. Yields: 81.5 %. MS (ES+) m/z: 805.03 $[M]^+$.IR (KBr tablet, cm^{-1}): 3289.5 (N-H), 2912.6 (Ar-H), 1677.5 (C=O), 1576.4, 1519.4, 1436.7, 765.4; ^{1}H NMR (δ: ppm, $CDCl_3$):^{1}H NMR (d_6-DMSO, δ ppm): 9.68 (s, 2H), 8.54 (d, 1H, J = 7.15), 8.43 (d, 1H, J = 7.95), 8.30 (d, 2H, J = 8.10), 8.22 (s, 1H, J = 8.35), 7.58 (t, 1H, J = 7.82), 7.27 (d, 2H, J = 8.30), 7.25(t, 2H,J = 6.37),7.14(d, 1H, J = 8.05), 6.90 (t, 2H, J = 7.37), 4.16 (t, 2H, J = 7.52), 3.53 (t, 4H, J = 7.35), 3.45 (2, 4H), 2.94 (t, 4H, J = 6.22), 2.91 (t, 4H, J = 4.67), 2.89 (t, 4H, J = 4.07), 1.71 (m, 2H, J = 7.56), 1.44 (m, 2H, J = 7.43), 0.97 (t, 3H, J = 7.35). ^{13}C NMR(δ: ppm, $CDCl_3$): 166.76, 164.29, 163.80,

162.54, 152.17, 138.76, 134.54, 131.54, 131.32, 129.96, 129.84, 129.82, 127.12, 126.13, 124.61, 123.26, 122.49, 120.44, 117.77, 117.49, 53.11, 40.12, 37.78, 36.47, 33.78, 33.36, 32.08, 31.42, 30.22, 20.36, 13.83.



Scheme 1. Synthesis route of the proposed crown ether

RESULTS AND DISCUSSION

The design and synthesis of mixed-donor crown ethers has been developing rapidly because of its applications in the field of coordination chemistry. The reactions proceed to give '1+1' macrocycles or '2+2' macrocycles depending on the chain length of starting materials(Zhang *et al*. 2010).In this experiment, a mixed-donor crown ethers was synthesized in good yields. The cyclizationof the starting materials of 1 with $S(CH_2CH_2SH)_2$ in DMF in the presence of 4-folds anhydrous Na_2CO_3 under nitrogen atmosphere at room temperature produced the corresponding macrocycle2in 81.5% yields.Structure of crown ether 2 was analyzed by MS, IR and NMR spectra. Indeed, the MS spectra data supported the formation of target compound. The IR spectra of macrocyclic compound2 are almost identical to compound 1. The formation of macrocycle is confirmed by the appearance of SCH_2 protons at δ~2.80 ppm in the ^1H NMR spectrum of compound2 in $CDCl_3$.

Conclusions

In summary, a newN_3S_5-thiaazacrown etherwas synthesized in high yields. The product was easy to purify. The conception may expand a promising approach to develop other crown ethers for the detection of environmentally related targets.

Acknowledgments

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