Development of New Synthetic Routes for Ebselen in Pursuit of the First Synthesis of Ebtellur

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Abstract

Ebselen, N-phenyl-1,2-benzisothiazol-3(2H)-one, an established antioxidant and cytoprotective agent, has been assessed for its activity for an array of pharmaceutical applications in treating a multitude of disorders and maladies. It has further been identified as an anti-microbial and anti-viral agent against multiple infectious agents and has even been FDA-approved for a variety of these applications. While ebselen has captured pharmaceutical interest, its tellurium analogue, ebtellur, has yet to be successfully obtained, and in only one case was an attempt to obtain ebtellur reported. We have developed multiple new synthetic routes specifically targeting the first synthesis of ebtellur and discuss the viability of each pathway herein.

Methods and Materials

Synthesis of ebtellur dichloride

1. Tellurium tetrachloride (0.337 g) was added to a Schlenk flask under inert atmosphere.
2. Dried THF (~10 mL) was added via needle.
3. Solution was placed into a liquid N2 /acetone bath.
4. Allowed solution to sit for 10 minutes.
5. Solution was warmed to room temperature with occasional venting through Schlenk line.
6. Solution was evaporated in vacuo and resulting solids were filtered using toluene.

Scheme 2: Synthesis of ebtellur dichloride

A catalyzed attempt utilizing potassium tellurocyanate yielded 125Te NMR peaks at 1.59, 1.32, and 1.24 ppt. Products are believed to be ditellurides due to these peak ranges and the product color.

Discussion

The reactions which produced the suspected dimer and ionic species will likely not yield ebtellur, and thus these reactions will require tailoring of starting materials and methods. The catalyzed tellurocyanate route requires further investigation to characterize the products. Trials which failed to yield products also require further reaction tailoring and testing to identify other possible ebtellur routes.

Conclusions

Several routes and attempts to synthesize the tellurium containing analogue of the biactive molecule ebselen, ebtellur, are reported in this project. New tellurium sources and routes were chosen to avoid the formation of previous dead-end products. The novel lithiation route using tellurium tetrachloride yielded a dimer containing two benzanilide and two tellurium centers. The catalyzed tellurocyanate route is believed to have yielded ditellurides. Other attempted routes require further investigation to determine product identities and route feasibilities. Future work will focus on known issues and investigate similar reactions with new tellurium sources.

References


Acknowledgments

I would like to thank the Winona State Chemistry Department for providing the space and supplies to conduct this project, the Winona State Grants & Sponsored Projects Office for supporting this work through the Undergraduate Student Research & Creative Presentations Grant and the Undergraduate Student Research & Creative Presentations Travel Support Grant, and the Winona State Student Senate for funding the lodging for this presentation.