

This article was downloaded by:

On: 28 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713618290>

Reaction of 2,4-(Naphthalene-1,8-Diyl)-1,3,2,4-Dithiadiphosphetane 2,4-with Bases and Acids

Zhaofu Fei; Alexandra M. Z. Slawin; J. Derek Woollins

To cite this Article Fei, Zhaofu , Slawin, Alexandra M. Z. and Woollins, J. Derek(2001) 'Reaction of 2,4-(Naphthalene-1,8-Diyl)-1,3,2,4-Dithiadiphosphetane 2,4-with Bases and Acids', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 169: 1, 69 – 72

To link to this Article: DOI: 10.1080/10426500108546592

URL: <http://dx.doi.org/10.1080/10426500108546592>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Reaction of 2,4-(Naphthalene-1,8-Diyl)-1,3,2,4-Dithiadiphosphetane 2,4-with Bases and Acids

ZHAOFU FEI, ALEXANDRA M. Z. SLAWIN
and J. DEREK WOOLLINS

Department of Chemistry, St. Andrews University, Fife, Scotland KY16 9ST, UK

The compound 2,4-(naphthalene-1,8-diyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide **1** reacts with different bases **2a-2d** HNR(HNR = N,N,N',N'-tetramethyl-guanidine, morpholine, pyrrolidine or NH₃) to give the corresponding ammonium salts of **3a-3d**. The reaction of **1** with Et₃N·3HF lead to the formation of **4a**. The new compounds formed from the reactions have been studied spectroscopically and by X-ray crystallography.

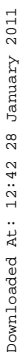
Keywords: P-S bond; NMR; X-Ray crystal structure

Introduction

Organophosphorus-sulfur heterocycles with the P₂S₂ systems have attracted great interest in recent studies^{1,2}. In continuation of this research³, we studied the properties of 2,4-(naphthalene-1,8-diyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide **1** towards different bases and acids (HF, HCl, HBr) in organic solvents.

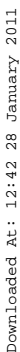
Downloaded At: 12:42 28 January 2011

Downloaded At: 12:42 28 January 2011



Downloaded At: 12:42 28 January 2011

Downloaded At: 12:42 28 January 2011



Downloaded At: 12:42 28 January 2011

Compounds **3b-3d** were initially soluble in the dichloromethane but the solubility decreased after removal of the solvent. In the $^3\text{P}\{\text{H}\}$ NMR the $^2J(\text{PP})$ are in the range 11.24-13.12 Hz. All **3a-3d** are very stable in inert atmosphere, but they seem to undergo slow decomposition in dimethyl sulfoxide.

Treatment of **1** with morpholine in the presence of excess triethyl amine lead to the formation of a mixture of several compounds, from which **3e** (Fig. 2) could be isolated by recrystallisation. The rest of the material which was poorly soluble in common solvents like diethyl ether, dichloromethane, chloroform, acetonitrile and dimethyl sulfoxide and could not be completely identified.

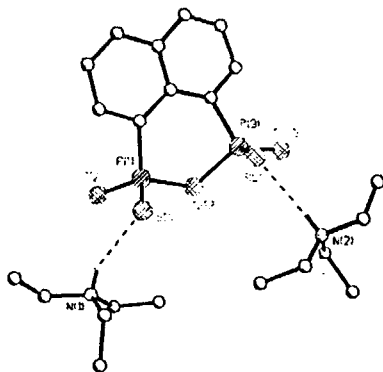


Figure 2: X-Ray crystal structure of **3e**

If **1** was treated with $\text{Et}_3\text{N} \cdot 3\text{HF}$, **4** (Fig.3) could be obtained in high yield; however, the treatment of **1** with HCl and HBr under the same condition did not lead to the similar results.

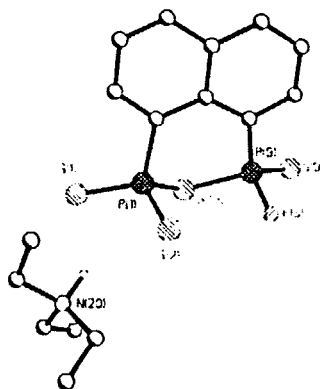


Figure 3: X-Ray crystal structure of **4**

Acknowledgements

We are grateful to the EPSRC for funding of the research and to the JREI for an equipment grant.

References

- [1] Foreman, M R. SJ.; Novosad, J; Slawin, A M. Z.; Woollins, J. D; *J. Chem. Soc., Dalton Trans.*, **1997**, 1347.
- [2] Kilian, P; Marek, J; Marek, R; Touin, J; Humpa, O; Novosad, J, Woollins, J. D; *J. Chem. Soc., Dalton Trans.*, **1998**, 1175.
- [3] Kilian, P; Slawin, A M.Z.; Woollins, J. D; *Eur. J. Inorg. Chem.*; **1999**, 2327.