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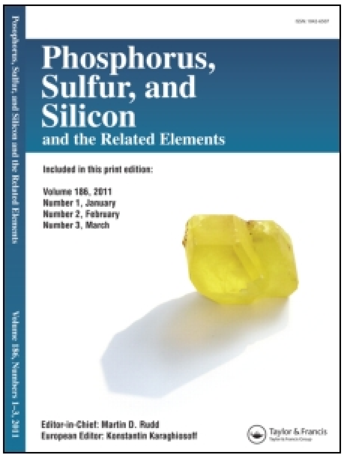

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
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New Cages Derived from 1,2,4-Triphosphol

Daniel A. Rajão[a]; Vinicius Caliman[a]; Maria Helena Araujo[a]; Antônio C. Doriguetto[b]; Eduardo E. Castellano[b]; Javier Ellena[b]; Peter B. Hitchcock[c]
[a] Universidade Federal de Minas Gerais, Brazil [b] Universidade de São Paulo, Brazil [c] University of Sussex, United Kingdom

Online publication date: 27 October 2010

To cite this Article Rajão, Daniel A. , Caliman, Vinicius , Araujo, Maria Helena , Doriguetto, Antônio C. , Castellano, Eduardo E. , Ellena, Javier and Hitchcock, Peter B.(2002) 'New Cages Derived from 1,2,4-Triphosphol', Phosphorus, Sulfur, and Silicon and the Related Elements, 177: 8, 2005 — 2006

To link to this Article: DOI: 10.1080/10426500213365

URL: http://dx.doi.org/10.1080/10426500213365

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Phosphorus, Sulfur and Silicon, 2002, Vol. 177:2005–2006

1042-6507/02 $12.00 + .00
DOI: 10.1080/10426500290094233

NEW CAGES DERIVED FROM 1,2,4-TRIPHOSPHOL

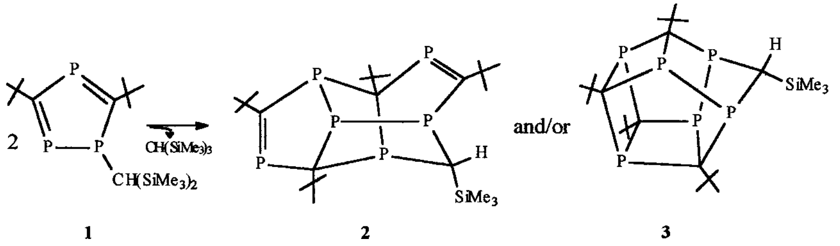
Daniel A. Rajão,[a] *Vinicius Caliman,*[a] *Maria Helena Araujo,*[a]
Antônio C. Doriguetto,[b] *Eduardo E. Castellano,*[b] *Javier Ellena,*[b]
and Peter B. Hitchcock[c]
Universidade Federal de Minas Gerais, Brazil;[a] *Universidade de São Paulo, Brazil;*[b] *and University of Sussex, United Kingdom*[c]

(Received July 29, 2001; accepted December 25, 2001)

The synthesis of the first 1,2,4-triphosphacyclopentadiene, **1** has been reported[1] and its coordination chemistry widely studied. In this work, we present the synthesis of two new cages, **2** and **3**, formed from the coupling of two molecules of **1** with looses of $CH(SiMe_3)_3$. Compound **2** has an open structure with two double bonds and a $CHSiMe_3$ bridging two phosphorus atoms. The structure of compound **3** is more symmetrical and two five-membered rings are joined side by side by a $CHSiMe_3$ bridge. The formation of these two isomers is confirmed by multinuclear (^{31}P, ^{13}C, and ^{1}H) NMR spectroscopy, MS, and x-ray diffraction studies.

The two isomers were synthesized from the anionic ring $(P_3C_2But_2)^-$ and the organic group $BrCH(SiMe_3)_2$ in a reflux temperature of dimethoxyethane via formation of the triphosphole (**1**). After purification by chromatography, single crystals were obtained from hexane solutions.

Both compounds **2** and **3** shown a 548 peak at the mass spectra with a different fragmentation pattern. $^{31}P\{^1H\}$ NMR of compound **2** present



SCHEME 1

Address correspondence to Vinicius Caliman, Departamento de Química, ICEx, Universidade Federal de Minas Gerais, Belo Horizonte, MG 31270-901, Brazil. E-mail: vcaliman@dedalus.lcc.ufmg.br six phosphorus resonances being 2 typical of sp^2-hybridized and 4 sp^3. Compound **3** also shows six phosphorus resonance at the $^{31}P\{^1H\}$ NMR and all of then appears at the sp^3-hibridized region.

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[1] V. Caliman, P. B. Hitchcock, J. F. Nixon, L. Nyulaszi, and N. Sakarya, *Chem. Comun.*, 1305 (1997).