

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>

Synthesis of M_xO_y and Related Rings

M. R. St. J. Foreman; R. A. Howie; M. J. Plater; J. M. S. Skakle; A. M. Z. Slawin

To cite this Article Foreman, M. R. St. J. , Howie, R. A. , Plater, M. J. , Skakle, J. M. S. and Slawin, A. M. Z. (2001) 'Synthesis of M_xO_y and Related Rings', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 169: 1, 297 – 300

To link to this Article: DOI: 10.1080/10426500108546647

URL: <http://dx.doi.org/10.1080/10426500108546647>

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.

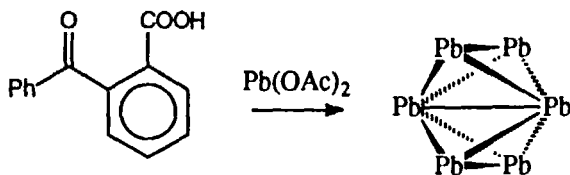
The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any 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.

Synthesis of M_XO_Y and Related Rings

M.R. St J. FOREMAN^{a*}, R.A. HOWIE^a, M.J. PLATER^a,
J.M.S. SKAKLE^a and A.M.Z. SLAWIN^b

^aDepartment Of Chemistry University of Aberdeen, Meston Walk, Aberdeen, AB24
3UE, Scotland, UK and ^bSchool of Chemistry, University of St Andrews,
St Andrews, Fife, KY16 9ST, Scotland, UK

By the hydrothermal reaction of lead (II) acetate with 2-benzonylbenzoic acid, colourless crystals of a hexanuclear lead carboxylate with bridging oxide ligands was obtained. The structure of the molecule could be described as being like a tennis ball of lead atoms at the centre of a soccer ball of carboxylate ligands. For clarity the carboxylate and oxide ligands have been omitted.

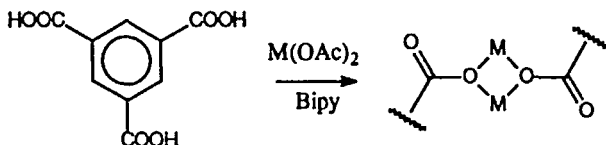


Crystal structures of other lead (II) carboxylates, which are polymeric, have been obtained.

* Corresponding author. E-mail: M.J.Plater@abdn.ac.uk or Foreman_mark@hotmail.com

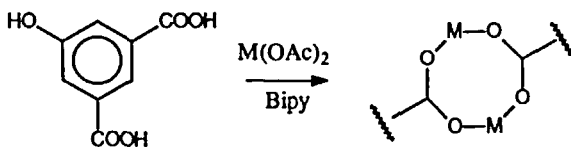
The hydrothermal reaction of cadmium acetate and 1,3,5-benzene tricarboxylic acid, formed a polymer which consists of layers of $\text{Cd}_3\text{C}_3\text{O}_6$ rings. This hydrothermal synthesis like the other reactions forming polymeric carboxylates was done inside a PTFE Parr bomb.

The hydrothermal reactions of zinc, manganese and cobalt acetates with 1,3,5-benzene tricarboxylic acid and 2,2'-bipy forms polymeric compounds which contain M_2O_2 rings. These compounds contain layers of carboxylates separated by layers of bipy ligands. This layered structure can be thought of as a stack of jam sandwiches.



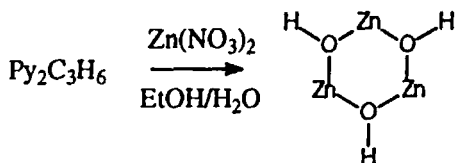
In our more recent work, Mn_2O_2 rings have been formed in solids where the bipy has been replaced with pyridyl pyrazoles.

The hydrothermal reaction of zinc or manganese (II) acetate, 2,2'-bipy and 5-hydroxyisophthalic acid forms polymeric materials containing $\text{M}_2\text{C}_2\text{O}_4$ rings.



When the synthesis is repeated with 1,10-phenanthroline instead of 2,2'-bipy, similar compounds, which also contain $\text{M}_2\text{C}_2\text{O}_4$ rings, were obtained.

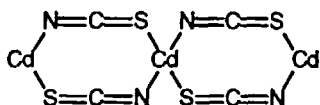
The room temperature reaction of zinc nitrate with 2 equivalents of 1,3-bis-(4-pyridyl) propane, formed crystals of a polymeric compound, with the formula $[\text{Zn}(\text{OH})(\text{Py}_2\text{C}_3\text{H}_6)\text{NO}_3]_n$ which contains Zn_3O_3 rings. Attempts to form this compound using one equivalent of $\text{Py}_2\text{C}_3\text{H}_6$ only formed traces of a fine powder.



The reaction of manganese (II) chloride, 1,3-*bis*-(4-pyridyl) propane and potassium thiocyanate forms colourless crystals of a polymer, which consists of parallel layers of two interpenetrated grids, made of $\text{Mn}_4\text{C}_{28}\text{N}_8$ rings. Above and below each layer are the thiocyanate ligands.

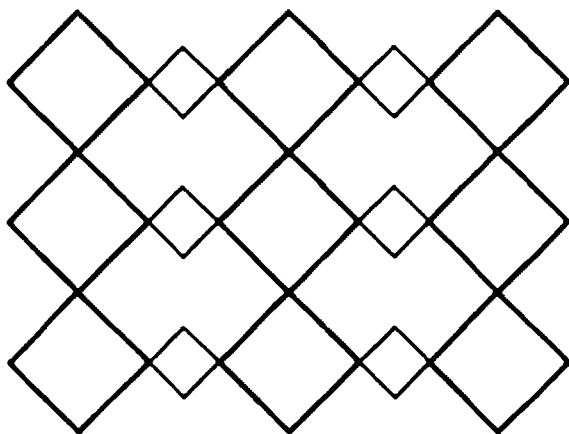
The reaction of cadmium nitrate, 1,3-*bis*-(4-pyridyl) propane and potassium thiocyanate forms a mixture of at least two solids. The crystal structure of one of these solids has been obtained.

$\text{Cd}_2\text{N}_2\text{C}_2\text{S}_2$ rings are linked at the cadmiums into infinite chains. These chains are crosslinked by the 1,3-*bis*-(4-pyridyl) propane ligands that bind to cadmium atoms in different chains.



By means of a Wittig reaction followed by hydrogenation (H_2 1 Atm at room temperature using 5% Palladium on Carbon) 1,2-*bis*-(2-(4-pyridyl) ethyl) benzene was obtained in good yield. The Wittig reaction is conducted in a conical flask using sodium ethoxide in ethanol as the base.

The reaction of cadmium nitrate with 1,2-*bis*-(2-(4-pyridyl) ethyl) benzene formed crystals of a polymeric compound with the formula $[\text{Cd(NO}_3)_2(\text{C}_{20}\text{H}_{20}\text{N}_2)_2]_n$. This compound contains $\text{Cd}_2\text{C}_{24}\text{N}_4$ and $\text{Cd}_4\text{C}_{48}\text{N}_8$ rings which are interlinked at the cadmium atoms. In the following diagram the larger rings are show in bold.



ACKNOWLEDGEMENT

We are grateful to the Leverhulme Trust for financial support (MRSJF), and to the University of Aberdeen for X-ray instrumentation.