

This article was downloaded by:

On: 27 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



Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

2,1-Benzisothiazoles

M. Davis^a; E. Homfeld^a; T. Paproth^a

^a Department of Organic Chemistry, La Trobe University, Bundoora, Victoria, Australia

To cite this Article Davis, M. , Homfeld, E. and Paproth, T.(1973) '2,1-Benzisothiazoles', Organic Preparations and Procedures International, 5: 4, 197 – 199

To link to this Article: DOI: 10.1080/00304947309355567

URL: <http://dx.doi.org/10.1080/00304947309355567>

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.

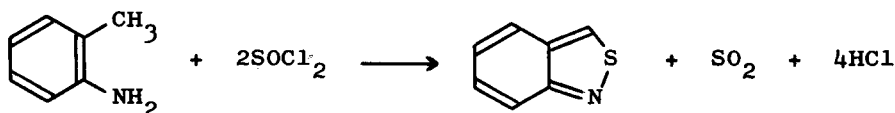
The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any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

2,1-BENZISOTHIAZOLES

Submitted by M. Davis, E. Homfeld and T. Paproth
(5/7/73)

Department of Organic Chemistry
La Trobe University
Bundoora, Victoria 3083, Australia

The scaled-up synthesis of 2,1-benzisothiazoles^{1,2} has been worked out. The lower molecular weight members (chloro, methyl, methoxy, etc.) are isolated as described for the parent compound below. For the less volatile ones (bromo, nitro and polysubstituted), it is best to extract the crude reaction mixture with conc. hydrochloric acid, followed by dilution of the acidic extract with excess water to precipitate the weakly basic 2,1-benzisothiazoles. The conversions vary from 20-70%, the best yield being obtained with a methoxy substituent and



the worst with the nitro group present. The starting amines can be recovered if desired and the yields then become almost quantitative in many cases.

EXPERIMENTAL

CAUTION: Since sulfur dioxide and hydrogen chloride are evolved in large quantity, the reactions should be conducted in a

J. A. MOORE

good hood and the gases trapped by passing them into a sodium hydroxide solution through an upturned funnel. A large gas washing bottle should be interposed between the top of the condenser and the funnel to obviate the possibility of a suck-back of the sodium hydroxide solution into the reaction flask.

2,1-Benzisothiazole.- To a mixture of 107g. (107 ml., 1.00 mole) and 150 ml. of xylene (mixed isomers, bp. 138-142°) in a 3 l. flask equipped with a stirrer, a dropping funnel and a reflux condenser, was added dropwise 160 ml. (264 g., 2.22 moles) of thionyl chloride with stirring. After the addition, the mixture was heated under reflux for 24 hrs (Since the initial yellow crystals dissolve quickly, the mixture becomes homogeneous within 2 hrs and stirring need not be continued beyond this point). The reaction mixture was cooled, water (500 ml.) was added cautiously and the whole was steam distilled until about 3 l. of distillate (consisting of xylene, o-toluidine, 2,1-benzisothiazole and water) had been collected. The organic layer was separated and the aqueous phase extracted with 100 ml. of xylene. The combined xylene extracts were washed free of o-toluidine (which is more basic than 2,1-benzisothiazole) by repeated shaking with 0.5M hydrochloric acid, followed by a final washing with water. The dried (sodium sulfate) organic phase was concentrated on a rotary evaporator to remove xylene. The residual orange liquid was distilled under reduced pressure, to afford 18-22 g. of 2,1-benzisothiazole as an almost colorless oil, bp. 70-72°/0.5 mm., 110-112°/1 mm.

2,1-Benzisothiazole as prepared above is 98% pure or better; it may be further purified by conversion into the

picrate salt, best prepared by treatment with excess saturated methanolic picric acid followed by decomposition of the pure picrate, mp. 123^o, with aqueous base.

REFERENCES

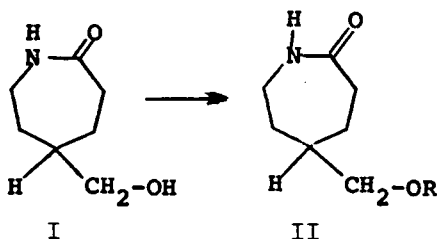
- 1.- M. Davis and A. W. White, Chem. Comm., 1547 (1968).
- 2.- M. Davis and A. W. White, J. Org. Chem., 34, 2985 (1969).

ESTERS OF (±)HEXAHYDRO-5-HYDROXYMETHYL-2H-AZEPIN-2-ONE

Submitted by C. G. Overberger and J. H. Kozolwski
(6/7/73)

Department of Chemistry and
The Macromolecular Research Center
The University of Michigan
Ann Arbor, Michigan 48104

Five new derivatives (II) of (±)hexahydro-5-hydroxymethyl-2H-azepin-2-one (I)¹ have been prepared and characterized.



II, R =

- a) 1-Menthoxyacetyl
- b) Phthaloyl-1-phenylalanyl
- c) D-10-Camphorsulfonyl
- d) Phthaloyl-H
- e) Succinoyl-H

EXPERIMENTAL⁵

(±)Hexahydro-5-(1-menthoxyacetoxymethyl)-2H-azepin-2-one (IIa).

A solution of 6.6 g (0.028 mole) 1-menthoxyacetyl chloride² [α]_D²⁴ -83.7^o (c, 2.0 chloroform), (bp. 67^o/0.04 mm),