FORMATION OF ACYCLIC BIS (REISSERT COMPOUNDS)

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References Cited
U.S. PATENT DOCUMENTS
4,242,273 12/1980 Shepard 564/91

OTHER PUBLICATIONS
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ABSTRACT

Bis Reissert compounds can be formed by reaction of a cyanohydrin, formed by reaction of an aldehyde and a diamine, with benzoyl chloride in the presence of an amine acid acceptor.

5 Claims, No Drawings
FORMATION OF ACYCLIC BIS (REISSERT COMPOUNDS)

BACKGROUND OF THE INVENTION

Recently, it has been proposed that Reissert compounds be synthesized to develop novel heterocyclic polymers for high performance applications (see H. W. Gibson et al., Amer. Chem. Soc., Polymer Preprints, 29(1), 154, 1988). Bis (Reissert compounds) have been synthesized in excellent yields by the use of a trimethylsilylcyanide reagent and can be used to develop such polymers (see A. Pandya et al., Amer. Chem. Soc., Polymer Preprints, 30(1), 206, 1989). Several novel 4,4'-coupled bis-isoquinolines have also been synthesized (see H. W. Gibson et al., Amer. Chem. Soc., Polymer Preprints, 30(1), 208, 1989).

W. E. McEwen et al., in J. Org. Chem. 1980, 45, 1301—1308 discuss the synthetic uses of open-chain analogues of Reissert compounds by first preparing an aminonitrile by condensation of a primary amine with a cyanohydrin followed by reaction of the aminonitrile with an acid chloride to form the Reissert compound. Chemical Abstracts, Vol. 87, 202161k reports on work by N. Voznesenskaya et al. in 1977 relating to the preparation of poly(phenyleneimidazolones) by polymerization of bis(alpha-aminonitriles) with aromatic dicarboxylic acid chlorides and subsequent cyclization of the prepared poly(alpha-cyanamides) by isomerization.

DESCRIPTION OF THE INVENTION

The starting cyanohydrin reagent of the formula

\[ RCH(CN)NH(R')NHCH(CN)R \]

where R is alkyl (e.g., ethyl) and R' is alkylene (e.g., \(-(CH_2)_{6}-\)) can be initially formed by reaction of an aldehyde (RCHO) and diamine (H2N—R'——NH2) in the presence of a cyanide source (e.g., NaCN) and bisulfite (e.g., sodium bisulfite). The molar ratio of aldehyde to diamine may vary from about 1:2 to about 1:3. The molar ratio of cyanide and bisulfite to aldehyde may vary from about 1:1 to about 1.6:1 in both cases. The reaction may be conducted in aqueous solution at temperatures of from about 0° C. See Organic Synthesis, Col. Vol. 4, p. 437.

Reaction of the cyanohydrin described above with benzoyl chloride in the presence of an appropriate amine acid acceptor (e.g., pyridine) yields the desired bis Reissert compound of the formula

\[ RCH(CN)NH(R')NCH(CN)R \]

where Ar represents phenyl. When purified by crystallization from ethanol it had a melting point of 110°—111° C.

IR (KBr): 3065—2860 (C—H), 2243 (weak CN), 1633 (N—CO), 1494, 1466, 1435, 1413, 1382, 1370, 1352, 1324, 1316, and 1283 cm⁻¹.

\[ ^1H \text{ NMR (DMSO-\text{d}_6)}: \delta 7.55—7.35 (m, 10H, COC_6H_5), 4.84 (s, 2H, CHCN), 3.3—3.1 (m, 4H, NCH_2), 2.1—1.85 (m, 4H, CH_2CH_2), 1.1—0.8 (m, 14H, CH_3 and 4 CH_2). \]

We claim;

I. A process for the formation of a bis Reissert compound of the formula
where R is alkyl, R' is alkylenic, and Ph is phenyl, which comprises reaction of
\[
\text{RCH(CN)NH(R')NHCH(CN)R}
\]
with benzoyl chloride in the presence of pyridine as amine acid acceptor.

2. A process as claimed in claim 1 where R is ethyl.
3. A process as claimed in claim 1 wherein R' is (CH₂)₆.
4. A bis Reissert compound of the formula
\[
\text{RCH(CN)NH(R')NHCH(CN)R}
\]
where R is alkyl, R' is C₆ alkylenic and Ph is phenyl.
5. A compound as claimed in claim 4 where R is ethyl.

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