This article was downloaded by: [Michigan State University]

On: 07 February 2015, At: 16:04

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



Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information: http://www.tandfonline.com/loi/lsyc20

Envirocat EPZG, R a New Catalyst for the Conversion of Aldoximes Into Nitriles

B. P. Bandgar ^a , S. R. Jagtap ^a , S. B. Ghodeshwar ^a & P. P. Wadgaonkar ^b

^a Dept. of Chem. , P. G. and Research Centre, R. B. N. B. College, Shrirampur , 413709, Dist Ahmednagar, Maharashtra, India

^b Polymer Chem. Div., National Chemical Laboratory, Pune, 411008, M. S. Published online: 16 Feb 2007.

To cite this article: B. P. Bandgar , S. R. Jagtap , S. B. Ghodeshwar & P. P. Wadgaonkar (1995) Envirocat EPZG, R a New Catalyst for the Conversion of Aldoximes Into Nitriles, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 25:19, 2993-2998, DOI: 10.1080/00397919508011431

To link to this article: http://dx.doi.org/10.1080/00397919508011431

PLEASE SCROLL DOWN FOR ARTICLE

Taylor & Francis makes every effort to ensure the accuracy of all the information (the "Content") contained in the publications on our platform. However, Taylor & Francis, our agents, and our licensors make no representations or warranties whatsoever as to the accuracy, completeness,

or suitability for any purpose of the Content. Any opinions and views expressed in this publication are the opinions and views of the authors, and are not the views of or endorsed by Taylor & Francis. The accuracy of the Content should not be relied upon and should be independently verified with primary sources of information. Taylor and Francis shall not be liable for any losses, actions, claims, proceedings, demands, costs, expenses, damages, and other liabilities whatsoever or howsoever caused arising directly or indirectly in connection with, in relation to or arising out of the use of the Content.

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden. Terms & Conditions of access and use can be found at http://www.tandfonline.com/page/terms-and-conditions

ENVIROCAT EPZG,^R A NEW CATALYST FOR THE CONVERSION OF ALDOXIMES INTO NITRILES¹

B.P. Bandgar, *a S.R. Jagtap, a S.B. Ghodeshwara and P.P. Wadgaonkarb

 a) Dept. of Chem., P.G. and Research Centre, R.B.N.B. College, Shrirampur - 413709, Dist Ahmednagar, Maharashtra, India.
b) Polymer Chem. Div., National Chemical Laboratory, Pune-411008, M.S.

ABSTRACT: The conversion of aldoximes into nitriles was carried out at 100°C in the absence of a solvent using Envirocat EPZG^R as a new solid supported catalyst.

The conversion of aldehydes into nitriles via aldoximes represents an important reaction.² For dehydration of aldoximes to nitriles a great variety of reagents have been used such as selenium dioxide-chloroform,³ diphosporus tetraiodide,⁴ trifluoroacetic anhydride-pyridine,⁵ Chlorosulphonyl isocynate,⁶ 4,6-diphenyl-2-methylthio-pyrilium tetrafluroborate,⁷ Copper (II) acetate- acetonitrile,⁸ trichloroacetyl chloride-triethylamine,⁹ triphenylphosphinecarbon tetrachloride in acetonitrile¹⁰ and montmorillonite KSF.¹¹ However many of these methods are deficient in some respect. The reagents are expensive or not readily available or work-up is tedious. The preparation of reagents like

^{*} To whom correspondence should be addressed

2994 BANDGAR ET AL.

triethylamine-sulphur dioxide¹² and sulphuryl chloride fluoride is inconvenient (at -70°C). ¹³ Dehydration with zeolites requires high temperature (350°C). ¹⁴ The reagents like phosgene, ¹⁵ diphosgene¹⁶ and triphosgene¹⁷ are hazardous to use. Therefore, there is still a need for convenient and generally applicable method for this conversion.

There has been a considerable growth in interest in recent years in the catalysis of organic reactions by inorganic reagents supported on high surface area inorganic materials. ¹⁸ Envirocats^R a new family of supported reagents ¹⁹ are a significant breakthrough in environmentally-friendly chemistry. These reagents are capable of catalysing Friedel-Craft's alkylation and acylation, sulphonylation, oxidation and other related process. ¹⁹ These supported reagents are non-toxic powders, which can be filtered off from the process and may be reused several times before it is exhausted. There is no aqueous effluent and the HCl emmission is limited to that given off from the envirocrat process.

In continuation of our work on solid supported reagents and reactions, ²⁰ we wish to report that Envirocat EPZG^R is an useful, easily handled catalyst for the conversion of aldoximes into nitriles.

ArCH=N.OH
$$\frac{\text{Envirocat EPZG}}{100^{0}\text{C}}$$
 > Ar-C = N

A mixture of (E)-and (Z)-isomers of aldoxime and catalytic ammount of Envirocat EPZG^R were mixed together and heated at 100°C in absence of a solvent with constant stirring for the specified time (Table). The reactions proceeded smoothly giving the corresponding products in good yields and purity.

Table. The Conversion of Aldoximes into Nitriles Using Envirocat $EPZG^R$ as the Catalyst.

ENTRY	Ar	TIME, h	YIELD,%	M .P./B.P.°C,
1	cı—(O)—	16	83	91-92
2	CI	20	92	64
3	NO ₂	12	79	111
4	QN QN	14	76	118
5	н ₃ со	24	92	61 – 62
6	н ₃ со-Осн ₃	24	67	90
7		23	78	144/760 mm Hg

2996 BANDGAR ET AL.

In conclusion, Envirocat EPZG^R has been found to be a convenient and very effective catalyst for the conversion of aromatic aldoximes into nitrites. The obvious advantages of Envirocat in terms of easy seperation, good yields and recyclability are noteworthy. Currently work is in progress to study the reactions catalyzed by Envirocats.

Experimental

All aromatic oximes were prepared from aromatic aldehydes using standard synthetic method. ²¹ Envirocat EPZG^R was obtained from Contract Chemicals, England and activated 1 h prior to use by azeotropic drying or by heating at 300-350°C and cooling in a flow of nitrogen. The products were characterised by their physical properties and spectral characteristics (¹H NMR, IR etc.). PMR spectra were recorded in CDCl₃ on 60 MHz or FT 90 MHz instrument using TMS as internal reference. IR spectra were recorded in nujol on Perkin Elmer IR spectrometer: model PE-883.

General Procedure

The aromatic aldoxime (10 mmol) and Envirocat EPZG^R (200 mg) were mixed together and heated at 100°C in the absence of a solvent with constant stirring for the specified time (Table). After completion of the reaction, the reaction mixture was cooled to room temperature and treated with dichloromethane (10 mL). The catalyst was removed by filtration and washed with dichloromethane (2 x 10 mL). Removal of the solvent under reduced pressure afforded products in good yields and almost pure form.

Acknowledgement. We thank Contract Chemicals, England for the generous gift of Envirocat EPZG and Prof. R.S. Mali for encouragement.

Downloaded by [Michigan State University] at 16:04 07 February 2015

ENVIROCAT EPZG^R 2997

References and footnotes.

R: Registered trade mark of Contract Chemicals, England.

- Solid supported Reagents and Reactions Part 6, for part 5, see Bandgar, B.P., Shahapurkar, R.G. and Wadgaonkar, P.P. Catsymp 1994 (accepted for publication).
- Friedrich, K. and Wallenfels, K. "The Chemistry of the Cyano Group" Rappoport, Z. Ed., Interscience, New York, 1970, PP. 92-93.
- 3. Sosnovsky, G. and Krogh, J.A. Synthesis 1978,703.
- 4. Suzuki, H., Fuchita, T., Iwasa, A. and Mishina, I. Synthesis 1978, 905.
- 5. Carroti, A. and Campaga, F. Synthesis 1979, 56.
- 6. Olah, G.A., Vankar, Y.D. and Garcia Luna, A. Synthesis 1979, 227.
- 7. Molina, P., Alajarin, M. and Vilaplana, M.J. Synthesis 1982, 1016.
- 8. Attanasi, O., Palma, P. and Serra-Zanetti, F. Synthesis 1983,741.
- 9. Santaniello, E., Milani, F. and Casati, R. Synthesis 1983, 749.
- 10. Kim, J.N., Chung, K.H. and Pyn, E.K. Synth. Commun. 1990, 20, 2785.
- 11. Meshram, H.M. Synthesis 1992, 943.
- 12. Olah, G.A., Vankar, Y.D. Synthesis 1978, 702.
- 13. Olah, G.A., Narang, S.C. and Garcia-Luna, A. Synthesis 1980, 659.
- Narayan Rao, M., Kumar, P. and Garyali, K. Org. Prep. Proced. Int. 1989, 21, 230.
- 15. Babad, H. and Zeiler, A.G. Chemical Review 1973, 73, 75.
- Mai, K. and Patil G. Synthesis 1986, 1037; Skorna, G. and Ugi, I Angew. Chem. Int. Ed. Eng. 1977, 16, 259.
- 17. Sahu, D.P. Ind. J. Chem. 1993, 32B, 385.
- 18 Villemin, D. and Martin, B. J. Chem. Res. (S) 1994, 146 and references cited therein.

2998 BANDGAR ET AL.

19. Contract Chemicals, England, "Envirocats - supported Reagents: Product Information" 1993.

- Bandgar, B.P., Unde, S.D., Unde, D.S., Kulkarni, V.H. and Patil, S.V. Ind. J. Chem. 1994, 33B, 782; Bandgar, B.P., shrotri, N.S., Ghorpade, P.K. and Patil, S.V. Ind. J. Chem. 34.B 1995, 152; Bandgar, B.P., Kshirsagar, S.N., and Wadgaonkar, P.P. Synth. Commun. (accepted for publication, SC:94138); Bandgar, B.P., Nikat, S.M. and Wadgaonkar, P.P. Synth. Commun. (in press, SC: 1198); Bandgar, B.P., Shahapurkar, R.G. and Wadgaonkar, P.P. Catsymp 1994 (accepted for publication).
- 21. Vogel, A.I. "A Text Book of Practical Organic Chemistry" ELBS, 3rd Ed., 1975, P. 721.

(Received in The Netherlands 24 February 1995)