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

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ENVIROCAT EPZG,[R] A NEW CATALYST FOR THE CONVERSION OF ALDOXIMES INTO NITRILES[1]

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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- aceto-nitrile,[8] trichloroacetyl chloride-triethylamine,[9] triphenylphosphinecarbon tetra-chloride in acetonitrile[10] 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

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triethylamine-sulphur dioxide[12] and sulphuryl chloride fluoride is inconvenient (at -70°C).[13] Dehydration with zeolites requires high temperature (350°C).[14] The reagents like phosgene,[15] diphosgene[16] and triphosgene[17] are hazardous to use. Therefore, there is still a need for convenient and generally applicable method for this conversion.

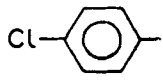
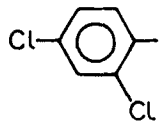
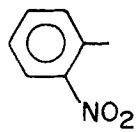
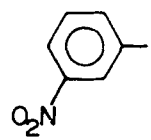
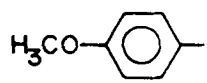
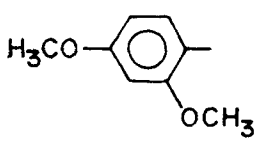
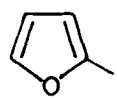
There has been a considerable growth in interest in recent years in the catalysis of organic reactions by inorganic reagents supported on high surface area inorganic materials.[18] Envirocats[R] a new family of supported reagents[19] are a significant breakthrough in environmentally-friendly chemistry. 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.

$$\mathbf{ArCH{=}N.OH \xrightarrow[100^0C]{\text{Envirocat EPZG}} Ar\text{-}C{\equiv}N}$$

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		16	83	91–92
2		20	92	64
3		12	79	111
4		14	76	118
5		24	92	61–62
6		24	67	90
7		23	78	144/760 mm Hg

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 (^{1}H NMR, IR etc.). PMR spectra were recorded in $CDCl_3$ 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.

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References and footnotes.

R : Registered trade mark of Contract Chemicals, England.

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