Indian Journal of Chemistry
Vol. 37B, February 1998, pp. 172 - 173

Note

Microwave assisted preparation of 1,3–dithiolanes under solvent free conditions

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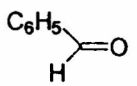
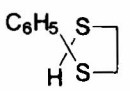
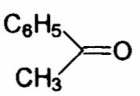
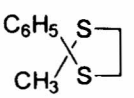
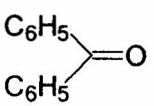
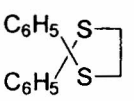
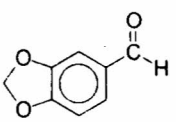
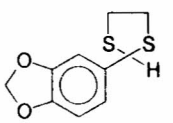
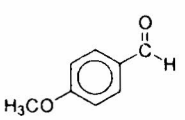
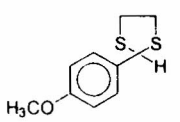
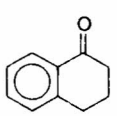
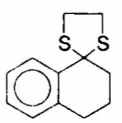
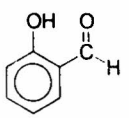
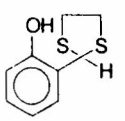
Received 16 October 1997; accepted 22 January 1998

Various carbonyl compounds react efficiently with 1,2-ethylenedithiol by microwave irradiation using mildly acidic K-10 clay within a few minutes and furnish the products in high yield under solvent free conditions.

Protection and deprotection of functional groups[1] are important steps in multiple step synthesis of natural products. For such conversions the hunt for simpler, time saving and environment friendly methods is continuously on by organic chemists. To augment this search use of domestic microwave oven in conjunction with solid support in dry conditions is well documented in literature[2].

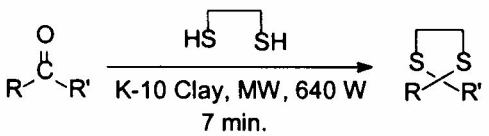
1,3-dithiolanes are widely employed for protection of carbonyl compounds as they are inert upon attack by nucleophiles and reducing agents. Anhydrous iron (III) chloride[3] dispersed in silica gel and sulphonated charcoal[4] under heterogeneous conditions are used as catalyst and are reported by

Table I—Preparation of 1,3-dithiolanes

Entry	Reactant	Rxn time (t/min)	Product	Yield (%)
1.		7		90
2.		7		80
3.		7		75
4.		7		80
5.		7		80
6.		7		70
7.		7		75.2

(a)All the products were characterised by spectral (IR, NMR) and elemental analysis
(b)Purity of compound was determined by TLC analysis

Patney for this conversion. Recently some other catalysts such as H–Y zeolite[5], anhydrous lanthanum trichloride[6] and acidic ion exchange resin[7] have been used for the thioacetalisation of carbonyl groups, but all these have one or the other drawbacks such as long reaction time, lower yields, use of highly carcinogenic solvents, expensive reagents and high temperature. In continuation of our work[8] we herein report a very clean, time saving and high yielding method for preparation of 1,3 dithiolanes from various aldehydes and ketones.



This is a simple procedure for protection of carbonyl groups under very mild conditions with the advantages of (i) high yield, (ii)manifold reduction in reaction time, and (iii) solvent free conditions shall find large application in natural product synthesis.

Experimental Section

^{1}H NMR spectra were recorded in $CDCl_3$ or CCl_4 on a Varian EM–360 (90 MHz) spectrometer using TMS as internal reference (chemical shifts in δ, ppm) and IR spectra as liquid films on a Perkin–Elmer infrared model 1430 spectrophotometer (ν_{max} in cm^{-1}). Unless otherwise stated all organic extracts were dried over anhyd. Na_2SO_4. Purity of all analytical samples was checked by TLC. Microwave reactions were carried out in BPL DMO 700T (640 W) microwave oven.

General procedure. In a 250 mL beaker activated K-10 montmorillonite clay (0.12g) was taken and a mixture of carbonyl compound (1mmole) and 1,2-ethylene-dithiol (1.5mmole) was added. The beaker was covered with a watch glass and exposed to microwave irradiation at 640W for 7 min. The product was extracted with diethyl ether (3×10mL), washed with brine and dried. Evaporation of solvent under reduced pressure gave the desired products in (75-80%) yield as reported in Table1.

Acknowledgement

The authors are thankful to the CSIR & UGC, New Delhi for providing financial support for this work.

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