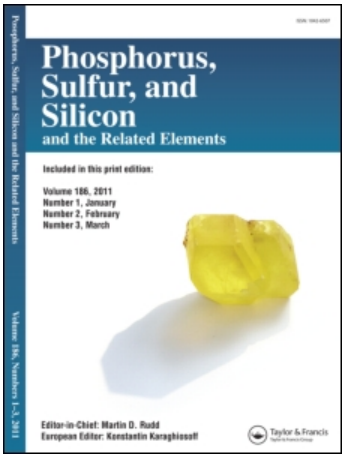

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SYNTHESIS OF THIOAMIDES FROM NITRILES AND HYDROGEN SULPHIDE IN THE PRESENCE OF PHASE-TRANSFER CATALYSTS

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The synthesis of thioamides[1] from nitriles generally involves base catalyzed reaction of nitriles with hydrogen sulphide in a polar organic solvent.

The known preparations are usually accomplished under pressure by heating, in a closed reactor, an alcoholic solution of nitrile and of hydrogen sulphide or of an ammonium hydrogen sulphide otherwise, at atmospheric pressure, by reacting nitrile dissolved in pyridine[2] with hydrogen sulphide in the presence of triethylamine.

We wish[3] to report on the application of phase trasfer method[4] to the reaction of nitriles with hydrogen sulphide.

When an heterogeneous mixture of an aqueous solution of sodium sulphide, a phase transfer catalyst, nitrile dissolved in an organic solvent and hydrogen sulphide (1-2 ata) is stirred vigorously, at 70°, the corresponding thioamide is obtained in high yield.

Generally thioamides are insoluble in the reaction medium and can be conveniently isolated by filtration; the mother liquors (aqueous and organic phases)can be reutilized, after addition of a new amount of nitrile.

Nitriles are generally used dissolved in benzene, 1,2-dichlorobenzene or diphenylether; solvent can be avoided when liquid nitriles are used.

Tricaprilylmethylammonium chloride, tetrabutylammonium chloride and dibenzo-18-crown-6 are used as phase transfer catalysts. In their absence formation of thioamides is very slow.

The convenience and high yields of our phase transfer procedure suggest that this will be a method of choice for routine synthesis of thioamides.

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