



donating effect of the substituent amino group, so there were no multiamino compounds observed.

In conclusion, a new method has been developed for the synthesis of *N*-alkyl-substituted 3,5-dinitroanilines. The materials are inexpensive and the yields are high. The method described herein compares very favourably with known methods and should be a valuable addition to amination methodology.

### Experimental

<sup>1</sup>H NMR spectra were obtained in CDCl<sub>3</sub> with a Bruker AC-400 instrument. Chemical shifts are given in ppm, with respect to internal TMS; *J* values are quoted in Hz. IR spectra were obtained with a Nicolet NEXUS-670 spectrophotometer; only the most significant absorptions in cm<sup>-1</sup> are indicated. Melting points were determined by using a Gallenkamp heated-block apparatus.

**CAUTION:** 1,3,5-trinitrobenzene is an explosive compound and should be stored and handled using appropriate precautions for the amounts involved.

#### General procedures for preparation of *N*-alkyl-3,5-dinitroanilines **3a–f**.

1,3,5-Trinitrobenzene (6.4 g, 30 mmol) and amine (240 mmol) **2a–f** were taken up in 15 ml water in a 25 ml autoclave. The mixture was heated at 180°C for 4 h after which time the starting material had been consumed as evidenced by thin layer chromatographic (developing solvent, EtOAc/hexanes = 3:1, V/V) analysis. The mixture was then cooled, filtered and the solvent removed under reduced pressure to give the crude product. Recrystallisation with ethanol:water = 3:1 (V/V) afforded *N*-alkyl-3,5-dinitroanilines **3a–f**. The yields obtained were in the range of 84–91%.

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