

Short Communications

Preparation of C_2N_4 , AzodicarbonitrileBØRGE BAK, ROAR ESKILDSEN and
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Azodicarbonitrile, $NC-N=N-CN$ (I) cannot readily be prepared by published methods.^{1,2} A reproducible yield of controllable purity³ can be obtained as follows: 100 mmol of dry $CICN$ (II) (3.6 l, $p=510$ mm) in an otherwise evacuated system was condensed into a 250 ml flask containing a stirred slurry of 3.6 g (55 mmol) of freshly recrystallized, activated ($N_2H_4 \cdot H_2O$) and vacuum-dried NaN_3 ,⁴ in 50 ml of dry dimethylphthalate (III). The mixture was allowed to attain room temperature. In the total volume of 0.3 l the initial pressure was 229 mm. In 6 h the pressure dropped to 167 mm (consumption of II²). In the next 15 h it rose to 246 mm, most probably due to the evolution of 2-3 mmol of N_2 from the resulting cyanogen azide, $NC-N_3$ (IV).⁴

The reaction mixture was cooled to -10° . Fractions of II were distilled-off and condensed in acetonitrile until $p=2$ mm. A minor quantity of IV is hereby removed. The acetonitrile solution should be discarded at once due to the explosive character of IV. High-purity N_2 was now admitted to the remaining solution of ca. 40 mmol of IV in III until $p=700$ mm. According to seven experiments carried out by us, II + IV + N_2 is non-explosive at $20-25^\circ$. At the subsequent pyrolysis N_2 was bubbled through the flask at atmospheric pressure at a rate of 500-600 ml/min for 8 h. The gases

passed a U-tube (i.d. 3 cm, heated zone-length 30 cm, temperature 200°). Plenty of a yellow reaction product separated in front of and inside two following traps (500 and 100 ml vol.), both cooled in liquid air. The traps contained I and II with the yellow sideproduct. I and II were distilled *in vacuo* into a smaller trap. Here II was removed almost completely (to ca. 97-98 %) by pumping at -80° . Thus, the inconvenient use of $CF_2ClCFCl_2$ ² is avoided. Complete removal of II met with the difficulty that traces of II are occluded in I. These traces can be removed by melting the sample at 35° , but only at the cost of a slight decomposition, manifesting itself in the liberation of N_2 and in the occurrence of a non-volatile decomposition product. Remaining I, with a trace of II, was finally distilled at $<0^\circ$ ($p < 1$ mm) into a receiver cooled in liquid air. At -190° I is stable for at least 3 weeks. Approximate vapor pressure data are: 0° , 8 mm; 10° , 18 mm; 20° , 40 mm. Vapors of I attack mercury and apiezon grease, the compound being truly 'elusive'.¹ Its infrared and Raman spectra have been published by us.³ The yield of purified product was 500 mg, or about 25 % with respect to NaN_3 .

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