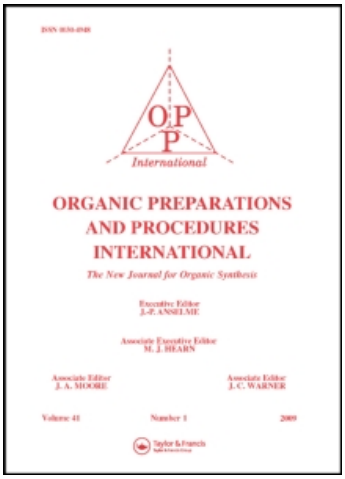

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exo-TRICYCLO[4.2.1.0^{2,5}] NON-7-EN-3-ONE

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exo-TRICYCLO[4.2.1.$0^{2,5}$]NON-7-EN-3-ONE

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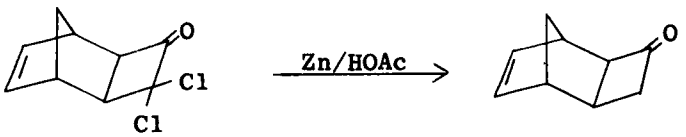
exo-Tricyclo[4.2.1.$0^{2,5}$]non-7-en-3-one (II) has been prepared by dehalogenation of 4,4-dichloro-exo-tricyclo-[4.2.1.$0^{2,5}$]non-7-en-3-one (I) using tri-n-butyltin hydride.[1] We recently required large amounts of II for the synthesis of isopropylidenecyclobutenone[2] and found a much more convenient method of dehalogenation of I using zinc and acetic acid. The latter method has been used successfully in the similar



(I) (II)

dehalogenation of 7,7-dichlorobicyclo[3.2.0]hept-2-en-6-one.[3]

EXPERIMENTAL

exo-Tricyclo[4.2.1.$0^{2,5}$]non-7-en-3-one (II).-To a vigorously stirred mixture of 13.0 g (0.2 g. a.) of zinc dust in 20 ml of glacial acetic acid was added gradually a solution of 5.0 g (0.025 mol.) of I in 10 ml of glacial acetic acid. Slight exothermicity occurred during addition, but external cooling was not required. The resulting mixture was

stirred for 3 hr at 65-70° and overnight at room temperature, filtered, and the filtrate was diluted with ~50 ml of cold water and extracted three times with 25-ml portions of ether. The combined ethereal extracts were washed (25-ml portions each) once with water, twice with saturated $NaHCO_3$ (until basic to litmus), once with saturated NaCl, dried over anhydrous Na_2SO_4, and concentrated at reduced pressure yielding 1.42 g (43%) II with physical properties identical with those reported in reference 1. Final distillation at 28°/0.08 mm gave 79% recovery with no noticeable improvement in purity. When the reaction was scaled up to 13 moles of I, and the zinc residues were washed with acetic acid, the yield increased to 57%.

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