ISOXAZOLINES FROM BENZYL CYCLOPROPANES

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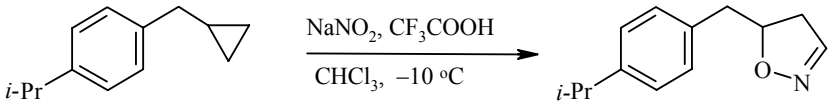
We reported earlier on synthesis of isoxazolines from aryl cyclopropanes when treated with sodium nitrite in trifluoroacetic acid [1].

In [2], the authors were able to obtain alkyl-substituted isoxazole from 2,2-dichlorobicyclo[4.1.0]-heptane (a compound in which the three-membered ring is not joined to an aryl moiety) when treated with $NOBF_4$.

We were able to establish that benzyl cyclopropanes (substrates in which the phenyl and cyclopropane moieties are separated by a methylene group) can be used equally successfully in the reaction with the reagent we used earlier in synthesis of isoxazolines from aryl cyclopropanes:



And in this case, as would be expected, the reaction occurs even more readily than in the case of aryl cyclopropanes, probably because of the lack of conjugation between the small carbocycle and the aryl moiety.

4-Isopropylbenzylisoxazoline. Trifluoroacetic acid (5 ml) was added to a solution of 4-isopropyl benzyl cyclopropane (1.74 g, 0.01 mol) in chloroform (10 ml) at -10°C, and sodium nitrite (0.69 g, 0.01 mol) was added over a 30 min period. The reaction mixture was stirred at the indicated temperature for another 30 min and poured into water, extracted with chloroform, and dried with anhydrous $MgSO_4$. The solvent was evaporated, and the residue was chromatographed on a column with silica gel in the system chloroform–hexane, 1:2. We isolated 1.58 g (78%) of 4-isopropylbenzylisoxazoline, a viscous oil. R_f 0.61, hexane–ether, 1:2. ^{1}H NMR spectrum (400 MHz), δ, ppm: 1.20 (6H, d, $2CH_3$); 2.41 (2H, d, $CH_2C_6H_5$); 2.75 [1H, s, $CH(CH_3)_2$]; 2.90 (2H, m, CH_2); 4.70 (1H, m, CH); 7.02 (1H, –N=CH); 7.15 (4H, s, Ar). Mass spectrum, *m*/*z* (I_{rel}, %): 203 $[M]^+$ (15.58); 133 (100); 119 (40.26); 105 (20.78); 91 (25.97). Found, %: C 78.79; H 8.24; N 7.01. $C_{13}H_{17}NO$. Calculated, %: C 78.84; H 8.37; N 6.97.

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