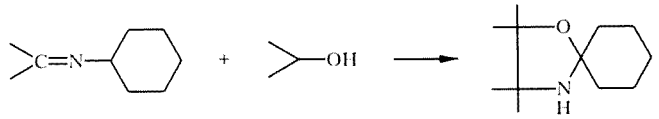
PREPARATION OF 2,5-PENTAMETHYLENE-4,4,5,5-TETRAMETHYLOXAZOLIDINE BY RADICAL CYCLOALKYLATION OF N-ISOPROPYLIDENECYCLOHEXYLAMINE

E. V. Pastushenko and G. I. Safiulova

Oxazolidines are known to be formed when aryl ketones and aliphatic imines are photolyzed [1].

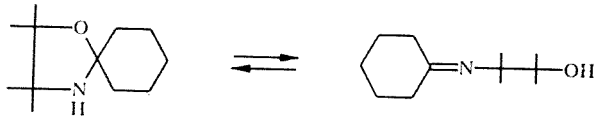
The radical alkylation of an aliphatic ketimine was studied to discover the behavior of Schiff's bases under free radical conditions. We have shown that 2,2-pentamethylene-4,4,5,5-tetramethyloxazolidine (II) can be obtained in one step by the reaction of N-isopropylidenecyclohexylamine (I) with isopropanol in the presence of *tert*-butyl peroxide. The conditions are the same as those reported earlier [2].



I II

It is proposed on the basis of the experimental results and literature data [3] that the radicals generated from isopropanol add to the azomethine carbon atom. The oxazolidine is formed from the α-aminoalkyl radical by recombination or disproportionation. α-Aminoalkyl radicals are not stabilised so that reduction of the C=N double bond cannot occur concurrently with cycloaddition.

For the spirocyclohexane derivative the possible equilibrium between the oxazolidine and the Schiff's base (cf. the data in ref. [4]) is shifted towards formation of the oxazolidine.



The reaction was carried out for 4 h at 140°C with the mole ratio of Schiff's base–*tert*-butyl peroxide–alcohol equal to 1:0.6:6. The alcohol and low boiling constituents were evaporated in vacuum. The amorphous product was washed with ether and chromatographed on a silica gel (40/100) column. The process was monitored by TLC on Silufol UV-254 strips with 20:4:3 hexane–ethyl acetate–chloroform as eluent. Conversion of the Schiff's base reached 38% with a 90% yield of 2-cyclohexyl-4,4,5,5-tetramethyloxazolidine based on that conversion.

N-Isopropylidenecyclohexylamine (I). ^{1}H NMR Spectrum ($CDCl_3$): 1.82 (6H, s, $2CH_3$), 1.98-2.3 ppm (11H, m, cyclohexyl).

2,2-Pentamethylene-4,4,5,5-tetramethyloxazolidine (II). ^{1}H NMR Spectrum ($CDCl_3$): 1.15 (6H, s, $2CH_3$), 1.23 (6H, s, $2CH_3$), 1.5-1.95 (10H, m, $5CH_2$), 5.3 ppm (1H, br s, NH).

C, H and N elemental analysis for the compounds synthesized agreed with calculated values.

Bashkir State Institute of Medicine, Ufa 450000. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 6, pp. 846-847, June, 1994. Original article submitted May 31, 1994

0009-3122/94/3006-0741$12.50

REFERENCES

1. A. A. Baum and L. A. Karnischky, J. Amer. Chem. Soc., **95**, 3072 (1973).
2. G. I. Safiulova, D. É. Kruglov, E. V. Pastushenko and R. A. Karakhanov, Khim. Geterotsikl. Soedin., No. 6, 847 (1991).
3. A. Padva, Chem. Rev., **77**, 37 (1977).
4. E. D. Bergmann, Chem. Rev., **53**, 332 (1953).