

References

1. H. G. Adolph, U. S. Pat. № 3531534, 1984.
2. B. S. Fedorov, N. I. Golovina, M. A. Fadeev, V. V.

Arakcheeva, G. V. Strukov, G. V. Shilov, R. F. Trofimova, and L. O. Atovmyan, *Izv. Akad. Nauk, Ser. Khim.*, 1994, 2207 [*Russ. Chem. Bull.*, 1994, **43**, №11 (Engl. Transl.)].

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Formation of 1-amino-1,2,3-triazole in the oxidation of 1,1,1,5,5,5-hexafluoro-4-trifluoromethylpentane-2,3-dione dihydrazone

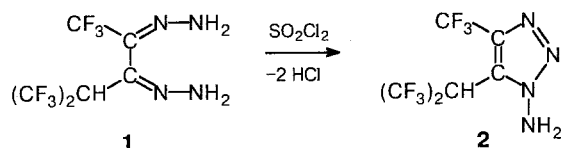
G. G. Bargamov* and M. D. Bargamova

A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences,
28 ul. Vavilova, 117813 Moscow, Russian Federation.
Fax: +7 (095) 135 5085

Dihydrazones of aliphatic α -diketones are known to be converted into dialkylacetylenes *via* the intermediate formation of aminotriazole,¹ which then entirely loses hydrogen and nitrogen.

Unexpectedly, it turned out that oxidation of dihydrazone of 1,1,1,5,5,5-hexafluoro-4-trifluoromethylpentane-2,3-dione (**1**) (see Ref. 2) with sulfuryl chloride under mild conditions affords 1-amino-4-trifluoromethyl-5-(1,1,1,3,3,3-hexafluoro-2-propyl)-1,2,3-triazole (**2**) in a yield of ~80 % (Scheme 1).

Scheme 1



SO₂Cl₂ has not been used hitherto for the oxidation of aliphatic α -diketone dihydrazones.

Polyfluoroalkyl substituted 1-amino-1,2,3-triazoles have not been described.

Compound **2**. m.p. 90–92 °C (from CCl₄). IR, ν/cm^{-1} : 1370–1380 s (N=N); 1480–1500 m (N=N); 1600–1630 m, 2930 and 3010 w (CH); 3280 m, 3370 s (NH₂). ¹H NMR (CDCl₃), δ : 5.8 (s, 2 H, NH₂); 9.4 (hept, 1 H, $J = 10$ Hz, CH(CF₃)₂). ¹⁹F NMR (CDCl₃), δ : -18.2 (s, 3 F, CF₃); -12.7 (d, 6 F, $J = 10$ Hz, (CF₃)₂CH). Found (%): C, 23.71; H, 1.14; F, 55.80; N, 18.98. C₆H₃F₉N₄. Calculated (%): C, 23.84; H, 0.99; F, 56.62; N, 18.64. MS, m/z : 303 [M+1 H]⁺; 274 [M-N₂]⁺; 255 [M-N₂, F]⁺; 235 [M-N₂, F, HF]⁺; 225 [M-F, N₂H₂]⁺; 206 [M-2 F, N₂H₂]⁺; 185 [M-2 HF, F, N₂H₂]⁺; 157 [M-3 F, N₄H₂]⁺; 137 [M-N₂]⁺; 113 [C₃F₄H]⁺; 93 [C₃F₃]⁺; 69 [CF₃]⁺; 30 [H₂N-N]⁺.

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References

1. H. J. Bestman, K. Kumar, and L. Kisielowski, *Chem. Ber.*, 1983, **116**, 2378.
2. M. D. Bargamova and L. S. German, *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1991, 1463 [*Bull. Acad. Sci. USSR, Div. Chem. Sci.*, 1991, **40**, 1308 (Engl. Transl.)].

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