Electrochemically promoted C-N bond formation from acetylenic amines and CO₂.

Synthesis of 5-methylene-1,3-oxazolidin-2-ones.

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General Experimental Procedure

Constant current electrolyses were performed in a divided glass cell (Pt spiral cathode – apparent area 4.5 cm^2 – volume of the catholyte: 20 mL) using an Amel Model 552 potentiostat equipped with an Amel Model 572 integrator. The counter electrode was a cylindrical platinum gauze - apparent area 1.3 cm^2 , 3.0 cm high - volume of the anolyte: 10 mL; it was separated from the catholyte through a porous glass plug filled with an agar gel (i.e., methyl cellulose 0.5% vol dissolved in DMF- Et_4NClO_4 1.0 mol dm^{-3}). The electrolyses were carried out, under nitrogen atmosphere at room temperature, under galvanostatic control ($J = 16\text{ mA cm}^{-2}$) on solutions of the amine in solvent- 0.1 mol dm^{-3} Et_4NPF_6 . At the end of the electrolysis, CO_2 was bubbled into the catholyte and the solution was heated to reflux for 2 hours. Usual work up (ether extraction and, when necessary, flash chromatography – *n*-hexane/ethyl acetate 8/2) gave oxazolidinones **2a-h** in the reported yields.

GAB 9 PULITO

ppm

7.26214
7.24235
7.22853
7.19599
7.16022

2.77156
2.75339
2.73488
2.71355
2.69965
2.67438
2.63312

2.25560
1.87894
1.84320
1.80332
1.78284
1.77883
1.70315
1.64531
1.63601
1.62247
1.60800
1.58728
1.57036
1.53143
1.51199
1.51592
1.48506
1.46399
1.27063
1.00535
0.98445
0.94922
0.93140

G 151

GAB 13 DOPU PREPARATIVA