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Synthesis of Ethyl [4-(2-chlorobenzyl)-3-methyl-6-oxopyridazin-1(6H)-yl]acetate

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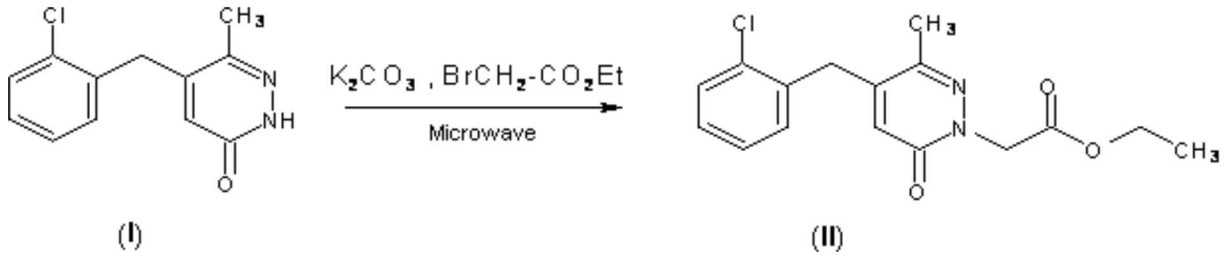
Pyridazines are of chemical and biological interest. They have been reported to be anticonvulsive agents [1], Furthermore, Gehrlein et al. have described the antihypertensive effects of novel hydroxyethylpyridazine compounds [2]. In continuation of this line of investigation, we have synthesized compound (I); it will be subjected to further pharmacological investigations, especially tests of its anticancer activity.



The product (II) was prepared from 5-(2-chlorobenzyl)-6-methylpyridazin-3(2H)-one (I) in situ by the solid-liquid PTC conditions without solvent [3]. To pyridazin (I) (1.4 g, 6 mmol) was added (2.75 g, 9 mmol) of potassium carbonate, (0.3 g, 1 mmol) of TBAB and (1 g, 6 mmol) of 2-ethyl bromoacetate. The mixture was placed in a pyrex tube which was then introduced into a Maxidigest MX 350 Prolabo microwave monomode reactor fitted with a rotational system. At the end of the irradiation time (10 min on 90 w as irradiation power), the mixture was cooled to ambient temperature. After elution with ethyl acetate (30 ml) and subsequent filtration on florisil, the organic product was purified by chromatography on silica gel using CH_2Cl_2 as eluent, yield: 92 % of (II) solid.

Melting point: 89-90°C

IR (KBr, cm^{-1}): 1747 (CO_2Et), 1670 (C=O), 1605, 1470, 1210 (C = N)

^{1}H-NMR (300.14 MHz, $CDCl_3$) d (ppm): 1.28 (t, J = 7.5 Hz, 3H, CH_3), 2.31(s, 3H, CH_3), 3.38 (s, 2H, CH_2), 4.25 (q, J = 5 Hz, 2H, CH_2), 4.82 (s, 2H, CH_2), 6.32 (s, 1H, H4), 7.20 (m, 4H, aromatic protons).

^{13}C-NMR (75 MHz, $CDCl_3$) d (ppm):14.11 (CH_3), 19.03 (CH_3) 35.77 (CH_2), 52.68 (CH_2), 61.66 (CH_2), 127.71 (CH o), 127.42(CH aromatic), 129.02 (CH aromatic), 130.02 (CH aromatic), 131, 12 (CH aromatic) 134.43, 144.97, 145.17, 160.30 (C3), 167.72 (C=O).

MS m/z (%) $(M+1)^+$ = 321.5, 279, 275,219.

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