The First Observation of Nitrogen-Carbonyl Bonding: Self-Assembly of N-oxalyl Dinitroanilide as Assisted by N…O=C Weak Interaction

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The synthesis and spectroscopic data of 1

Scheme 1

0.378g (3mmol) oxalyl chloride was dissolved in 20 mL CH₂Cl₂ solution and 0.183g (1mmol) 2,4-Dinitroaniline in 20 mL CH₂Cl₂ was dripped to the solution. Then methanol was added. The resulted solution was vacuum dried and the residue purified by column chromatography (CHCl₃) to yield 0.2g, 75% yield. Pale-yellow powder. mp=175~177; ¹H NMR (300MHz, CDCl₃, TMS); δ 4.065 (s, 3H, -CH₃), 8.57 (d, J=9Hz, 1H, ArH), 9.12 (d, J=9Hz, 1H, ArH), 9.21 (s, 1H, ArH), 11.20 (s, 1H, NH); ¹³C NMR (75MHz); 54.6, 122.2, 124.7, 130.2, 137, 139.6, 143.6, 155.6, 160.3; FAB-MS: 269 (M⁺), 223 (M-NO₂), 210 (M-COOCH₃); Elemental Analysis: C₉H₇N₃O₇: Calcd: C, 40.16; H, 2.62; N, 15.61. Found: C, 40.22; H, 2.60; N, 15.72.