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

$$O_2N \xrightarrow{\qquad \qquad NH_2 \qquad \text{ii, oxalyl chloride} \qquad O_2N \xrightarrow{\qquad \qquad NO_2 \qquad NO$$

0.378g (3mmol) oxalyl chloride was dissolved in 20 mL  $CH_2Cl_2$  solution and 0.183g (1mmol) 2,4-Dinitroaniline in 20 mL  $CH_2Cl_2$  was dripped to the solution. Then methanol was added. The resulted solution was vacuum dried and the residue purified by column chromatography (CHCl<sub>3</sub>) to yield 1 0.2g, 75% yield. Pale-yellow powder. mp=175~177 ;  $^1H$  NMR (300MHz, CDCl<sub>3</sub>, TMS);  $\delta$ 4.065 (s, 3H, -CH<sub>3</sub>), 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<sup>+</sup>), 223 (M-NO<sub>2</sub>), 210 (M-COOCH<sub>3</sub>); Elemental Analysis:  $C_9H_7N_3O_7$ : Calcd: C, 40.16; H, 2.62; N, 15.61. Found: C, 40.22; H, 2.60; N, 15.72.