Supplementary Material for Chemical Communications

Supplementary Information

Reactivity of a *N*-Heterocyclic Carbene, 1,3-Di-(1-adamantyl)imidazol-2-ylidene, with a Pseudo-Acid: Structural Characterization of Claisen Condensation Adduct

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General Procedures: All reactions were carried out under an atmosphere of dry argon using standard Schlenk techniques or in a MBraun glovebox containing less than 1 ppm of oxygen and water. NMR spectra were recorded using Varian 400 or 300 MHz spectrometers. Methyl acetate anhydrous was purchased from Aldrich and used as received. The NHC, IAd was synthesized according to the literature methods[1]

(I): IAd (100mg, 0.298 mmol) was dissolved in 2mL methyl acetate under inert atmosphere. The resulting solution was stirred at room temperature for 5 days. Methyl acetate was removed under *vacuo* and the residue was washed with toluene. Yield: 209.1 mg (46%); ^{1}H NMR (thf-d_8, 400 MHz): δ= 11.07 (s, 1H), 7.79 (s, 2H), 3.26 (s, 3H), 2.33 (s, 12H), 2.19 (s, 3H), 1.95 (s, 6H), 1.80 (m, 12H). ^{13}C NMR (thf-d_8, 100 MHz): δ = 31.4, 31.6, 31.7, 37.2, 44.5, 62.6, 120.0, 130.1, 135.9.

X-ray structure of (I):

A single crystal was placed in a capillary tube and mounted on a Bruker SMART CCD X-ray diffractometer. Data was collected using Mo Kα radiation at 150 K. The structure was solved using direct methods (SHELXS-97).[2]

Colourless crystals, '$[C_{23}\ H_{33}\ N_2]^+[C_5\ H_7\ O_3]^-$', FW = 452.62;

Monoclinic, space group P2(1)/n,

a = 13.5366(13),

b = 11.6995(12),

c = 15.7153 Å,

α= 90.00,

β = 93.217(2),

γ = 90.00,

V = 2484.9(4)Å^3,

Z = 4,

$\lambda(Mo_{K_\alpha})$ = 0.71073Å,

μ = 0.078,

ρ_{calcd} = 1.097gcm^{-3},

T = 150(2) K,

$F(000)$ = 896.

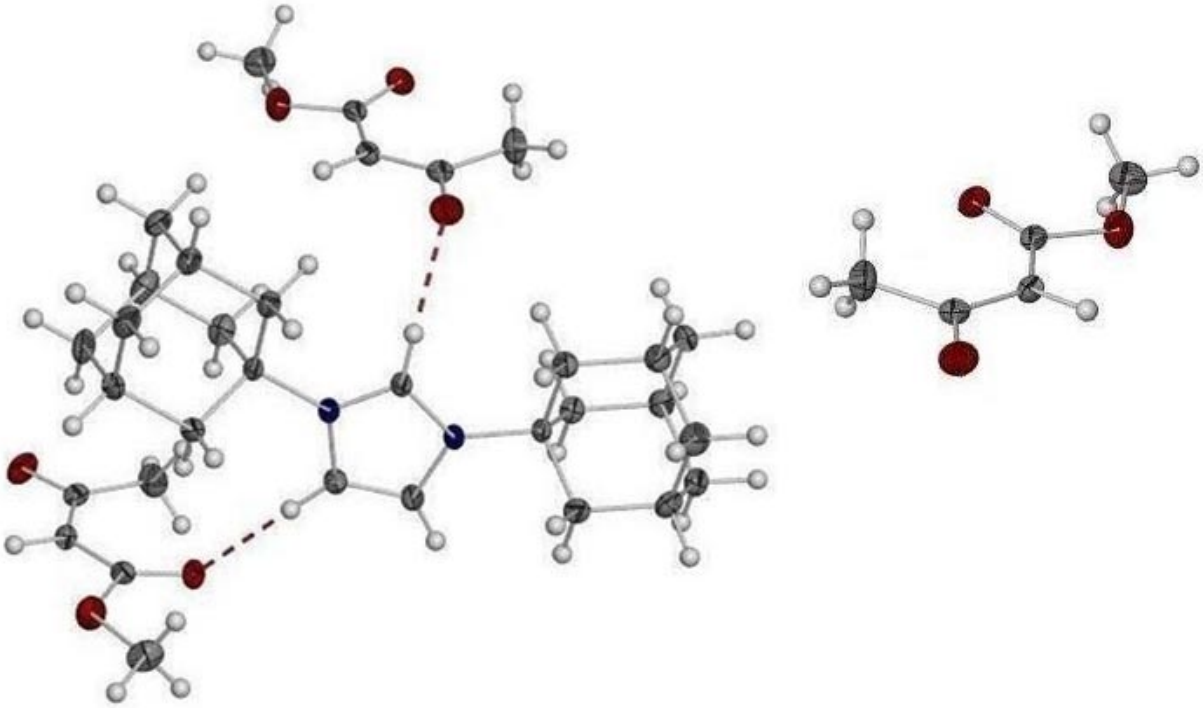
A total number of 32438 reflections of which 7384 independent reflections were collected (θ_{max} = 31.07^0)) and refined by full-matrix least-squares techniques (SHELXL-97), 458 number of parameters, R_1 = 0.0266 wR_2 = 0.0740 (all data).

All H atoms were refined isotropically.

CCDC number: 244497.

Thermal Ellipsoids (with 50% probability level)

The Claisen Condensation Adduct (**I**)



Notes and References

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(1) A. J. Arduengo III, R. Krafczyk, R. Schmutzler, A. Craig, A. Hugh, J. R. Goerlich, J. M. William, M. Unverzagt, *Tetrahedron,* **1999**, *55*, 14523-14534. A. J. Arduengo III, R. L. Harlow, M. Kline, *J. Am. Chem. Soc.,* **1991**, *113*, 361-363. P. L. Arnold, F. G. N. Cloke, T. Geldbach, P. B. Hitchcock, *Organometallics,* **1999**, *18*, 3228-3233. A. J. Arduengo III, S. F. Gamper, J. C. Calabrese, F. J. Davidson, *J. Am. Chem. Soc*. **1994**, *116*, 4391-4393. A. J. Arduengo III, F. Davidson, R. Krafczyk, W. J. Marshall, M. Tamm, *Organometallics,* **1998**, *17*, 3375-3382.

(2) Programs for crystal structure analysis: G. M. Sheldrick, SHELXL-97, University of Gottingen, Gottingen (Germany), 1998.