SYNLETT

Accounts and
Rapid Communications in
Synthetic Organic Chemistry

SUPPORTING INFORMATION

Supporting Information
for DOI: 10.1055/s-0034-1379026

Thieme

A Practical, Large-Scale Synthesis of Pyrene-2-Carboxylic Acid

Juan M. Casas-Solvas,[1] Tiddo J. Mooibroek, Shugantan Sandramurthy, Joshua D. Howgego and Anthony P. Davis*

School of Chemistry, University of Bristol, Cantock's Close, Bristol, BS8 1TS, UK
[1]New address: Department of Chemistry and Physics, University of Almería, Carretera de Sacramento s/n, 04120, Almería, Spain.
E-mail: Anthony.Davis@bristol.ac.uk

Supporting Information

(15 Pages)

Table of Contents

General methods. Proton and carbon NMR spectra were recorded at 500 MHz on a Varian System 500A spectrometer. Chemical shifts for proton and carbon are reported in ppm downfield from tetramethylsilane or the residual solvent signal. Mass spectra (electron impact (EI) and electrospray ionization (EIS)) were recorded on VG Analytical Autospec (EI) and VG Analytical Quattro (ESI) spectrometers. IR spectra were measured on a Perkin Elmer Spectrum One FT-IR spectrometer equipped with a Perkin Elmer universal ATR sampling accessory. All commercially available compounds were purchased from Aldrich and were used without further purification. Solvents for synthesis were dried by passing through a modified Grubbs system,[1] manufactured by Anhydrous Engineering. Routine monitoring of reactions was performed using pre-coated silica gel TLC plates (Merck silica gel 60 F254). Spots were visualized by either UV light or ethanolic solution of phosphomolybdic acid. Flash column chromatography[2] was performed using silica gel (Fisher brand silica 60Å particle size 35-70 micron) as the stationary phase.

[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.
[2] Still, W. C.; Khan, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

[1]H-NMR spectra for compounds 2-4

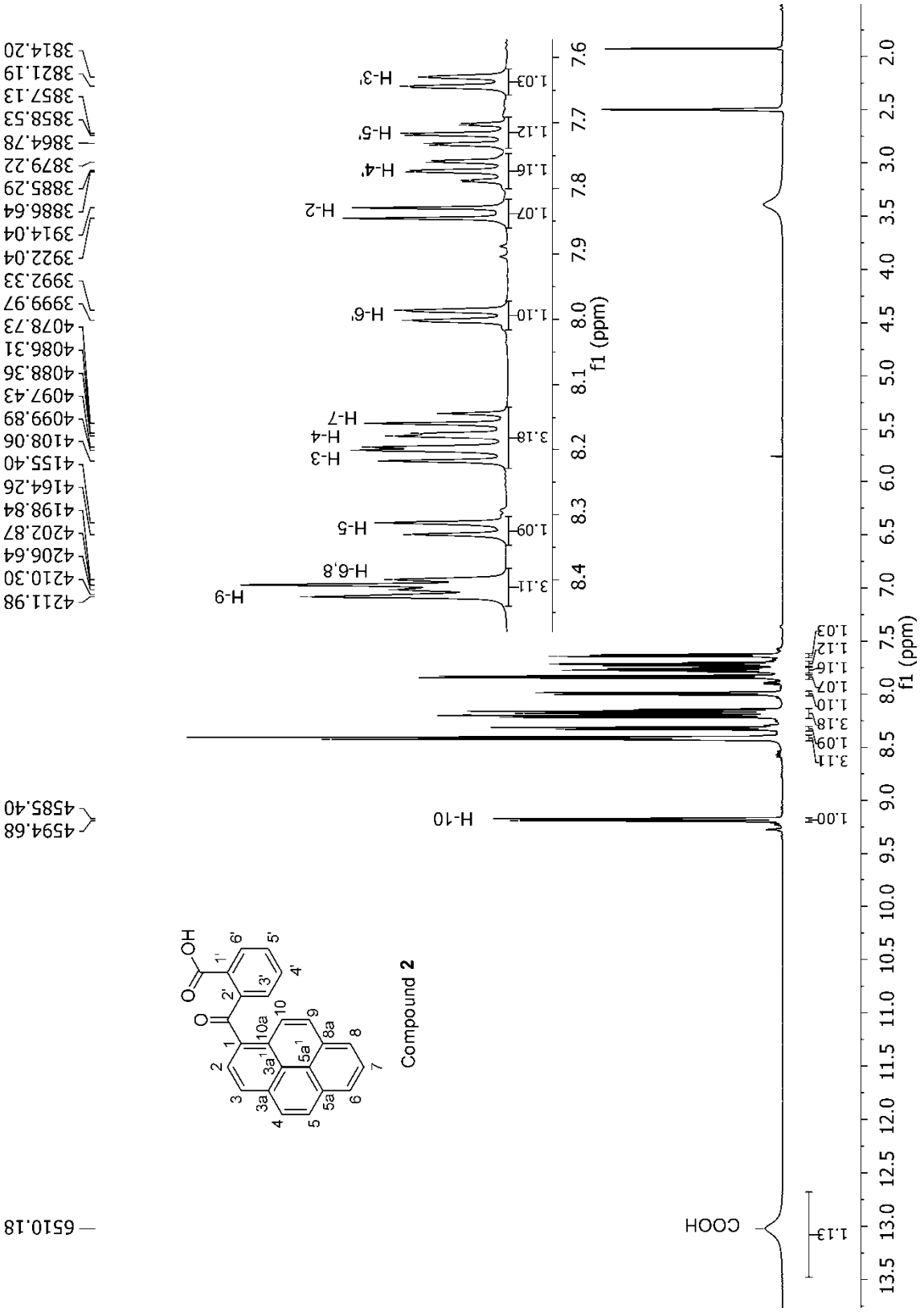

Figure S1. [1]H NMR spectrum of 1-(*o*-carboxybenzoyl)pyrene **2** in DMSO-d^6.

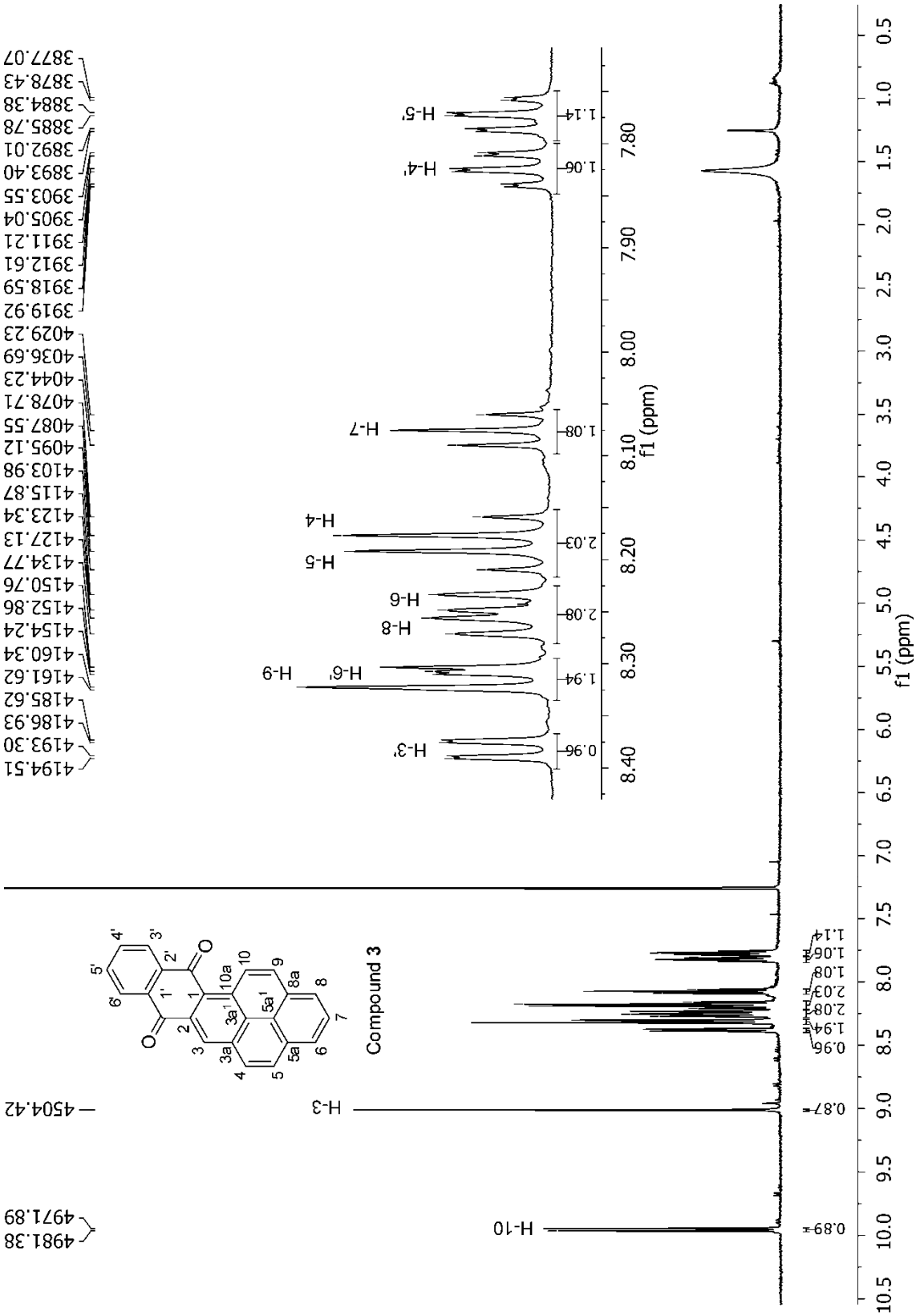

Figure S2. ^{1}H NMR spectrum of crude 1,2-phthaloylpyrene **3** as used in the preparation of **4** (see paper refs 27, 28). Solvent = $CDCl_3$.

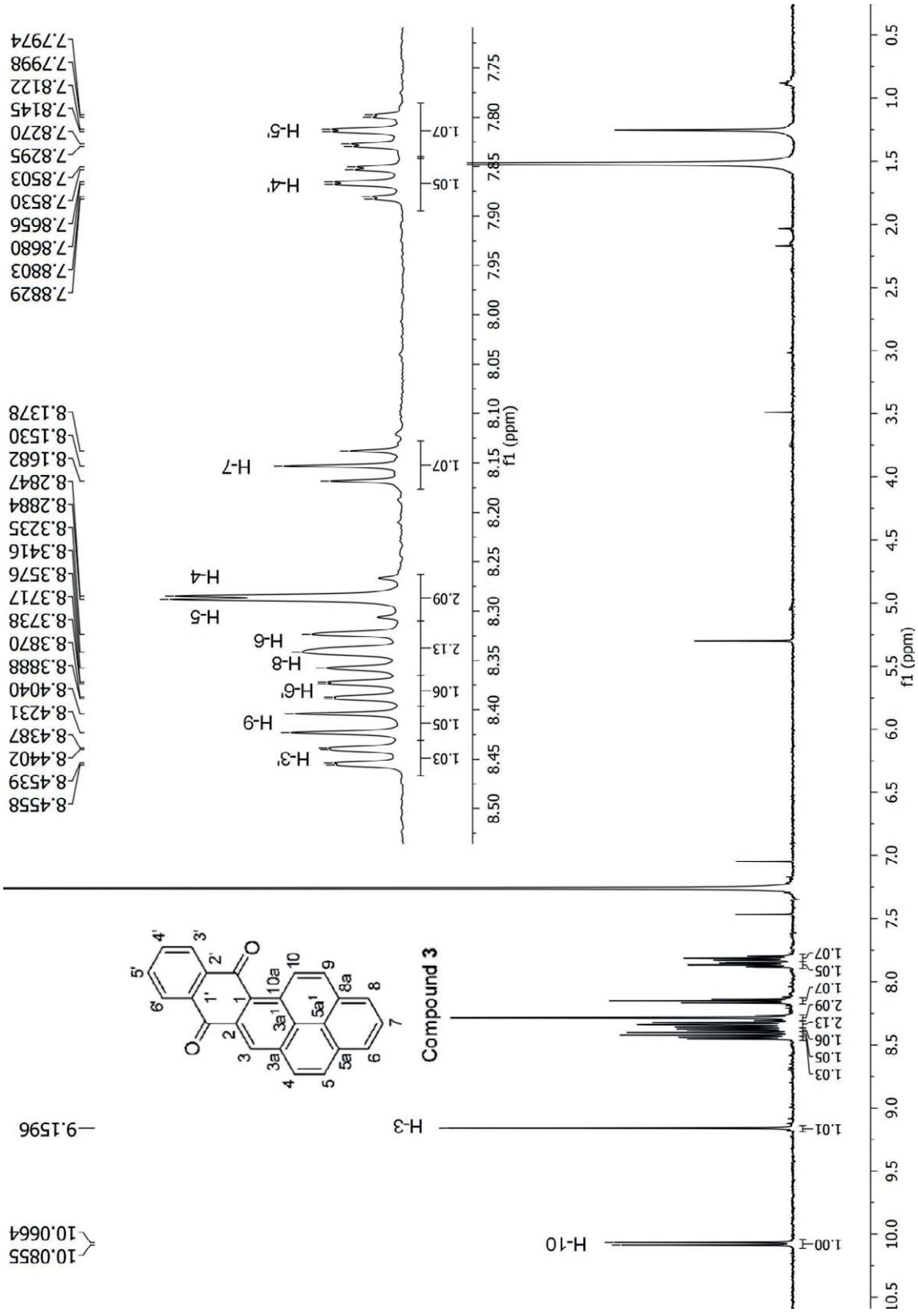

Figure S3. ^{1}H NMR spectrum of 1,2-phthaloylpyrene **3** after purification by chromatography. Solvent = $CDCl_3$.

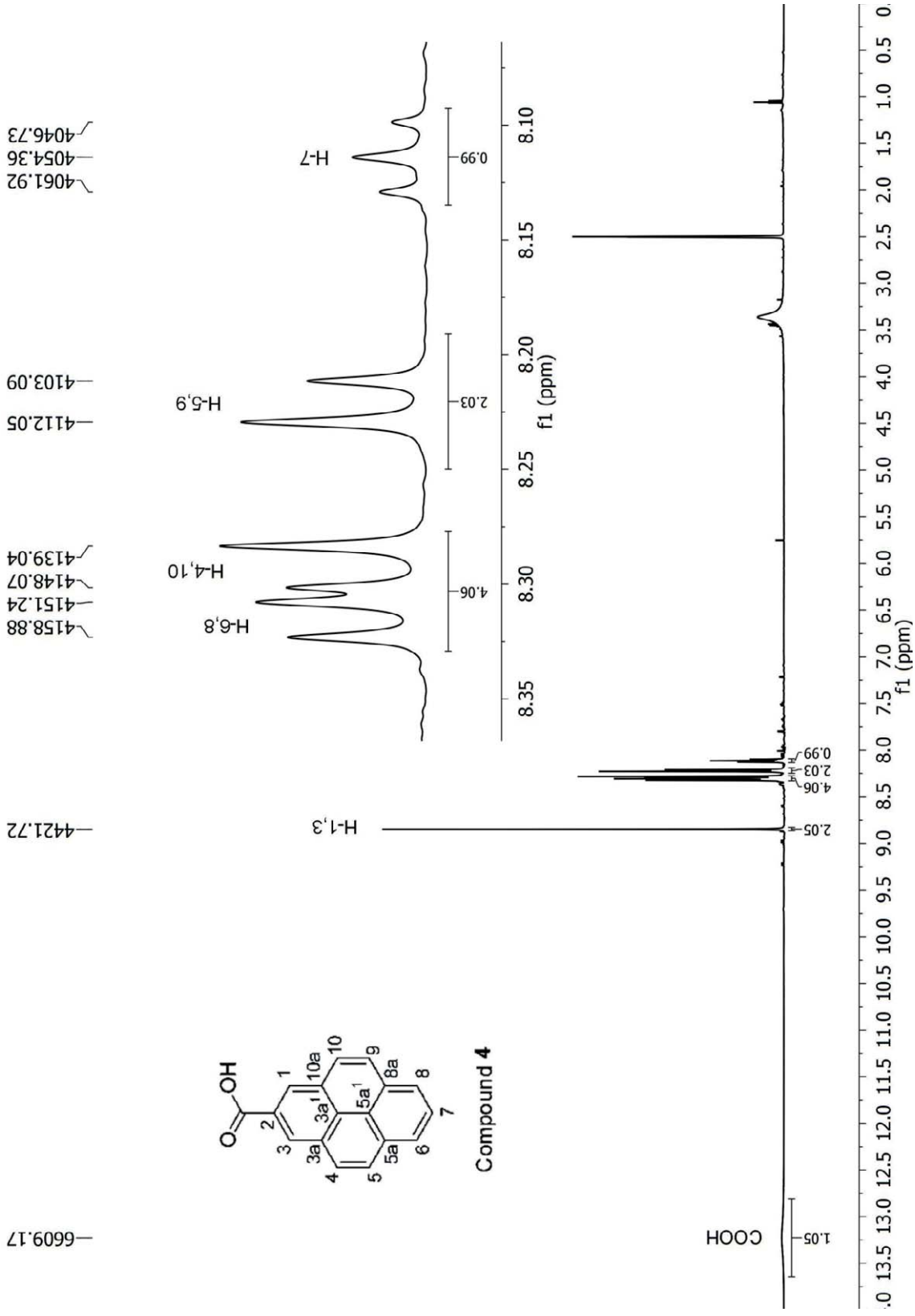

Figure S4. ^{1}H NMR spectrum of pyrene-2-carboxylic acid **4** after short column chromatography (see paper ref 28). Solvent = DMSO-d^{6}.

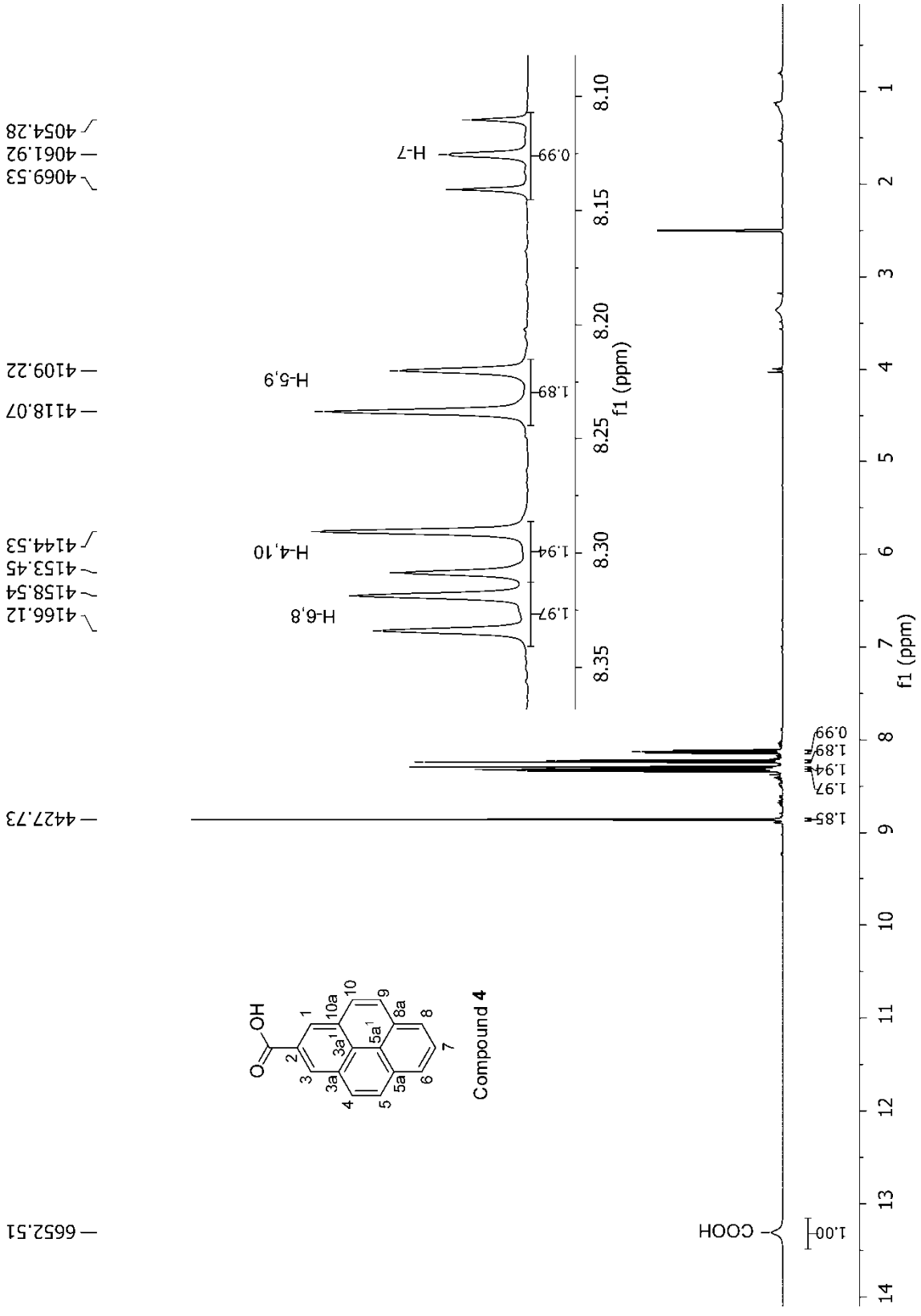

Figure S5. ^{1}H NMR spectrum of pyrene-2-carboxylic acid **4** after recrystallization from nitrobenzene. Solvent = DMSO-d^{6}.

^{13}C-NMR spectra for compounds 2-4

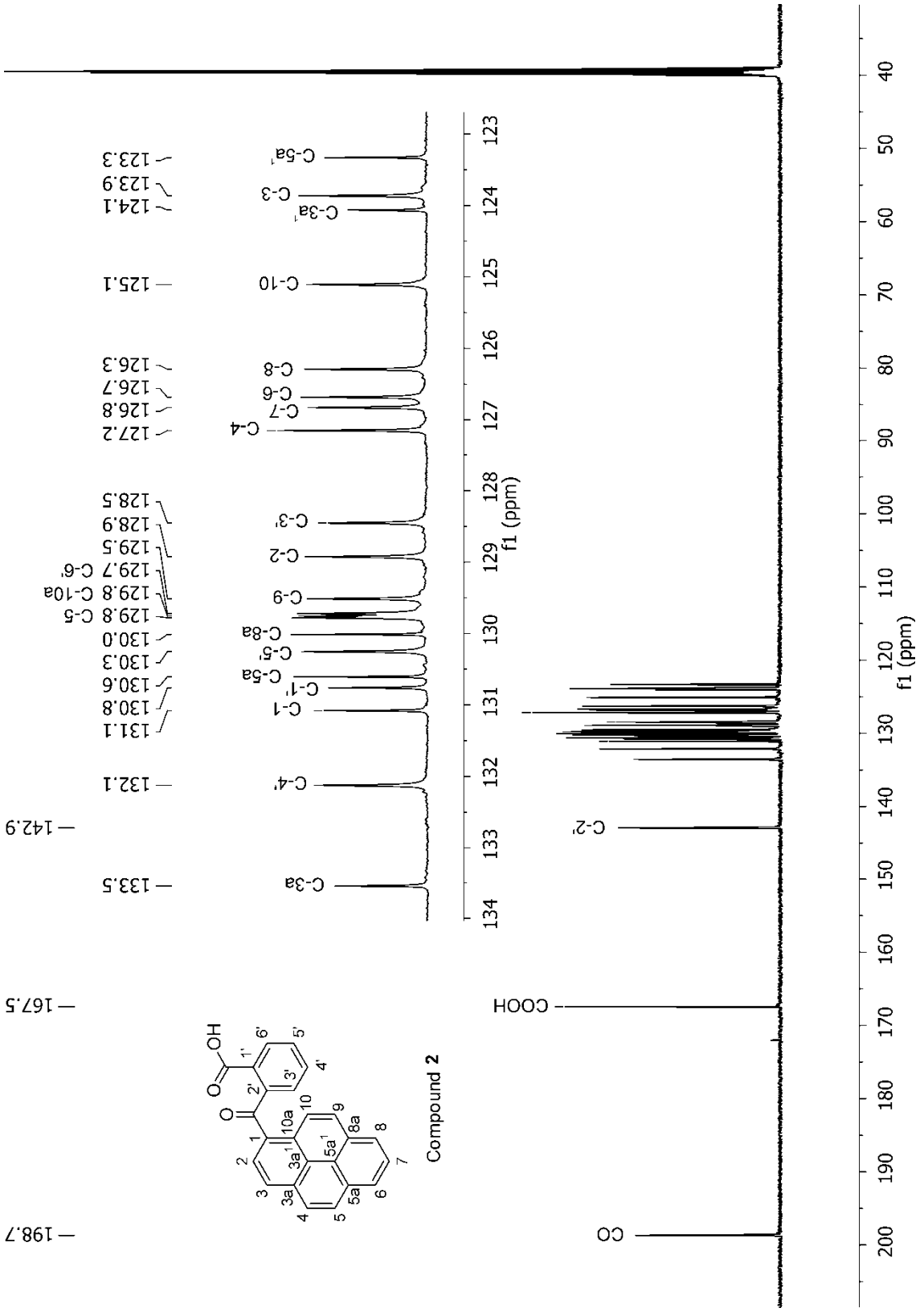

Figure S6. ^{13}C NMR spectrum of 1-(*o*-carboxybenzoyl)pyrene **2** in DMSO-d^6.

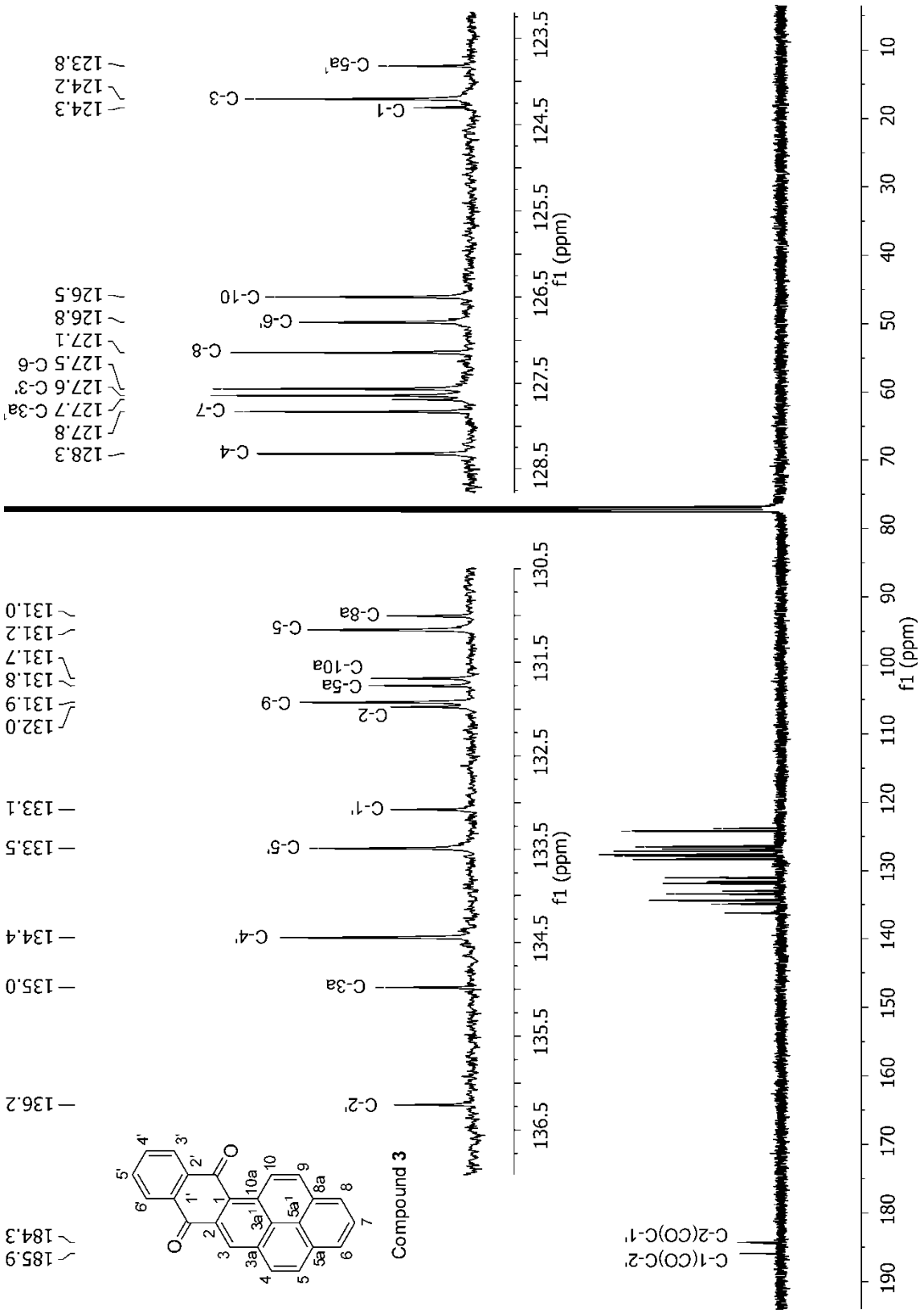

Figure S7. ^{13}C NMR spectrum of 1,2-phthaloylpyrene **3** in $CDCl_3$.

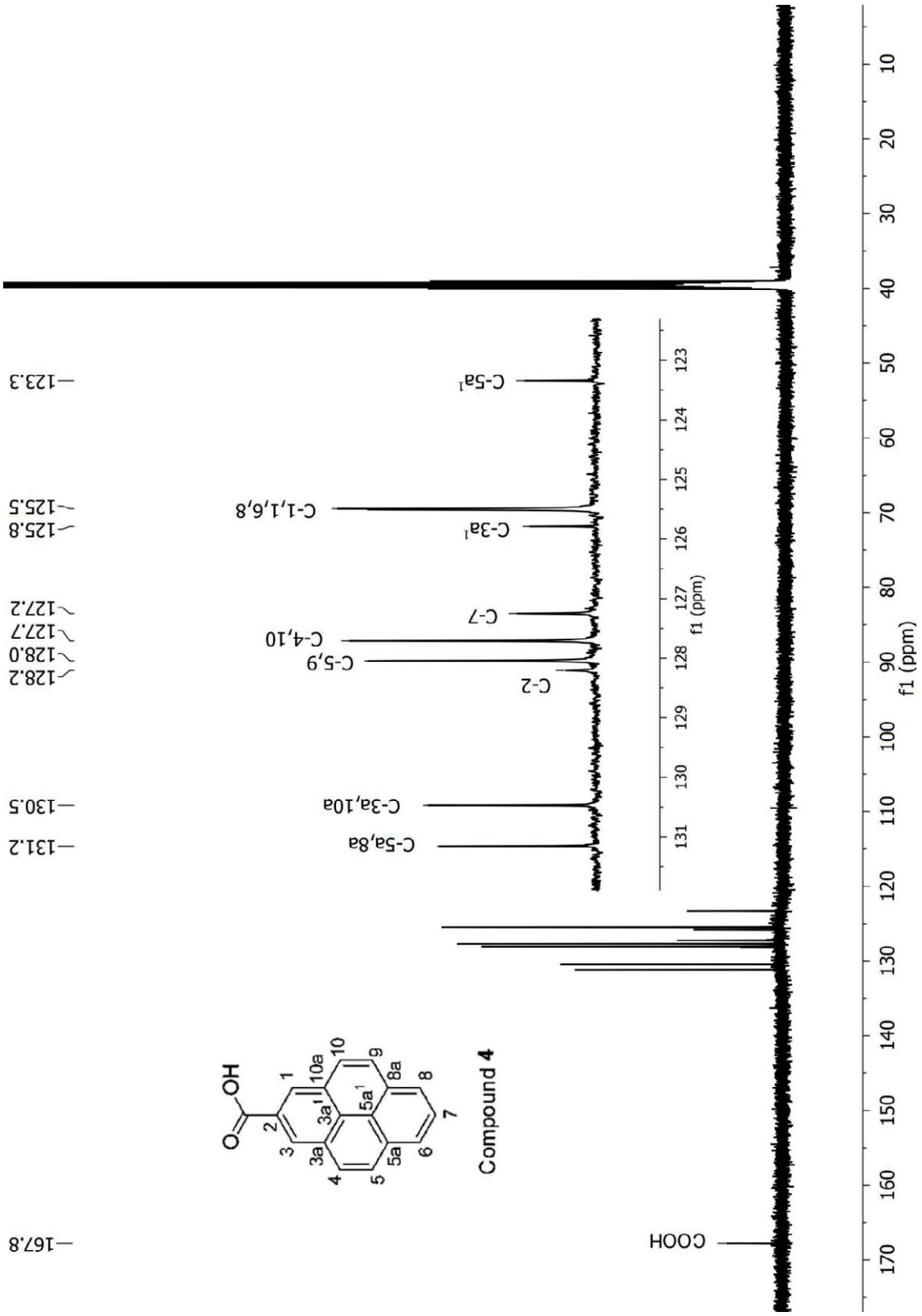

Figure S8. ^{13}C NMR spectrum of pyrene-2-carboxylic acid **4** after short column chromatography (see paper ref 28). Solvent = DMSO-d^6.

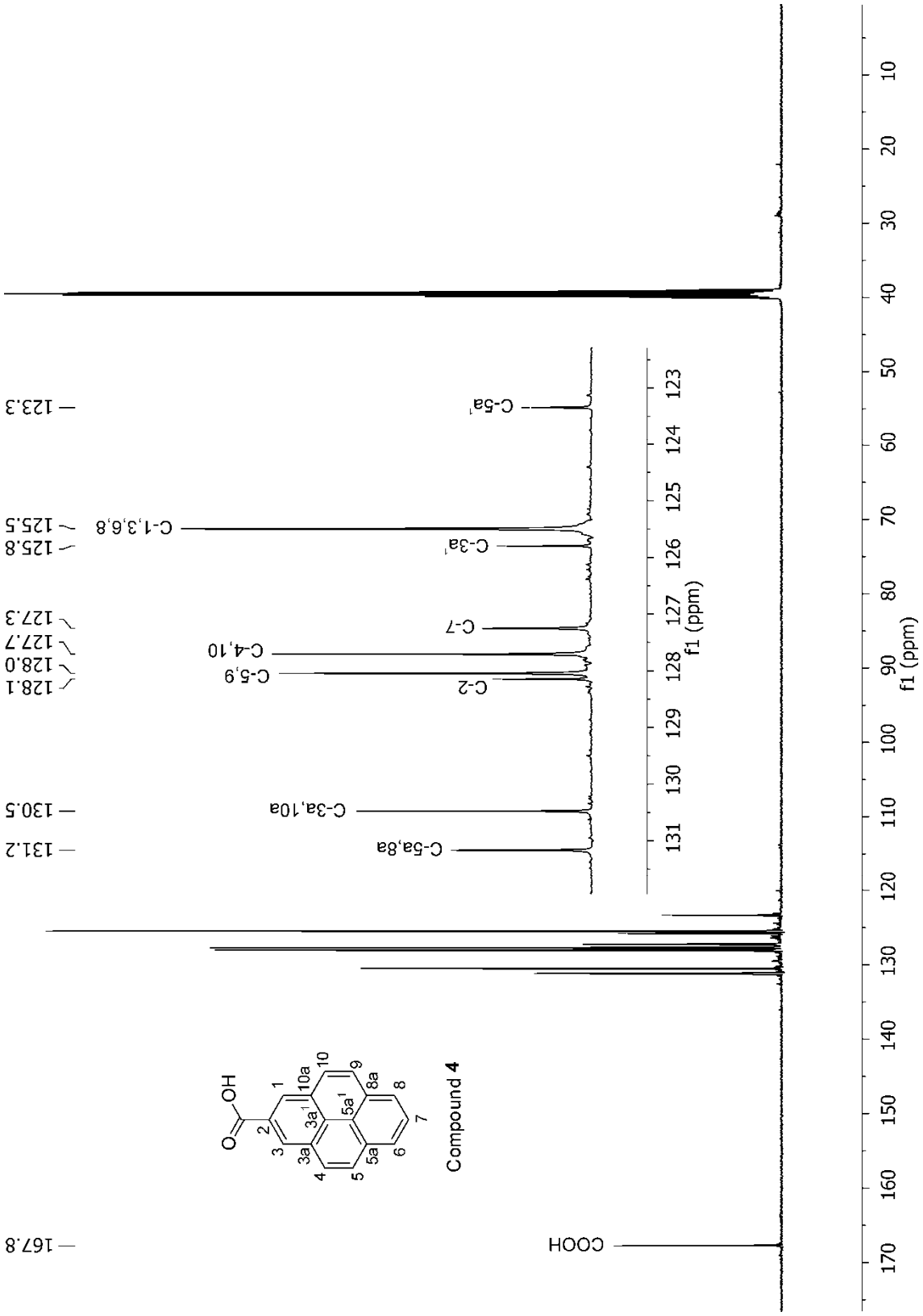

Figure S9. ^{13}C NMR spectrum of pyrene-2-carboxylic acid **4** after recrystallization from nitrobenzene. Solvent = DMSO-d^6.

2D NMR spectra of compounds 2 and 3

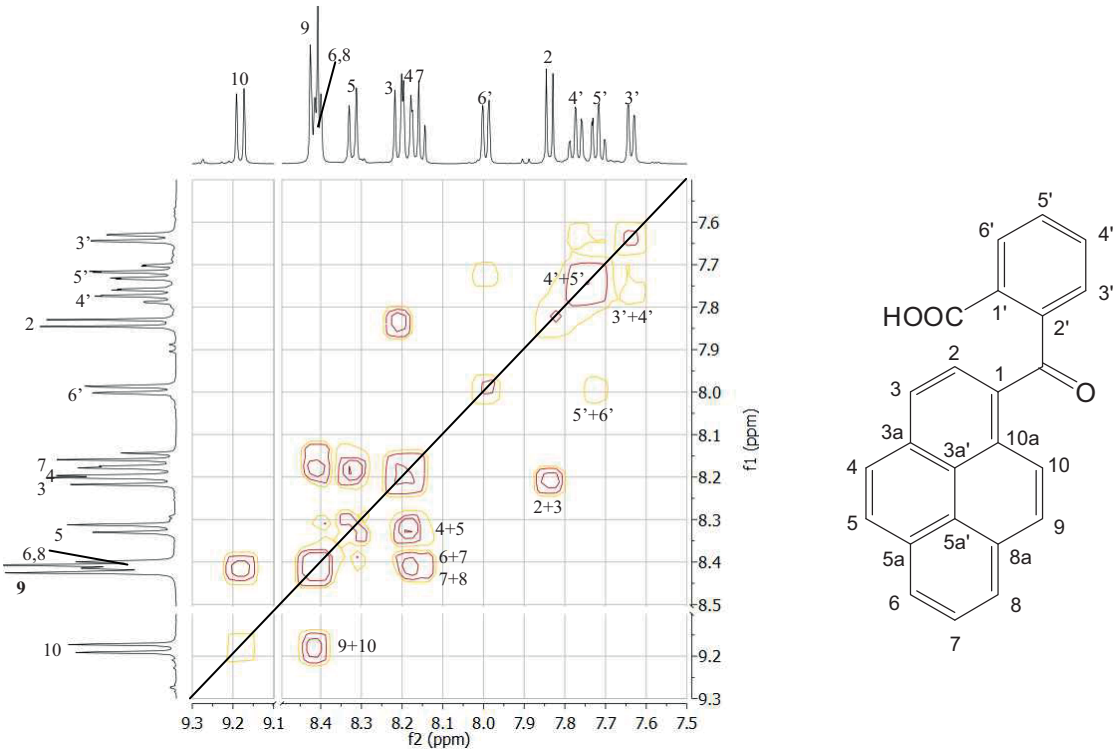

Figure S10. {^{1}H–^{1}H} COSY of compound **2** in DMSO-d^6, with assignments.

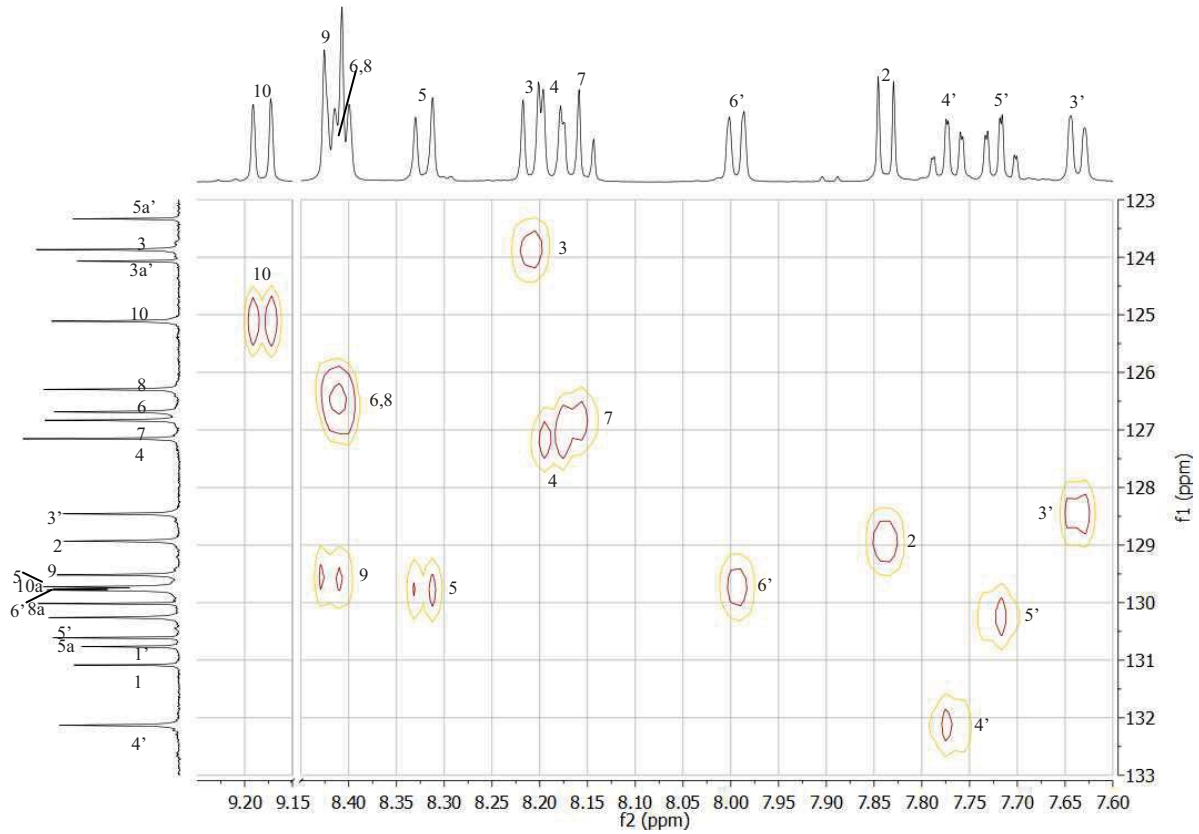

Figure S11. {^{1}H–^{13}C} HMQC of compound **2** in DMSO-d^6.

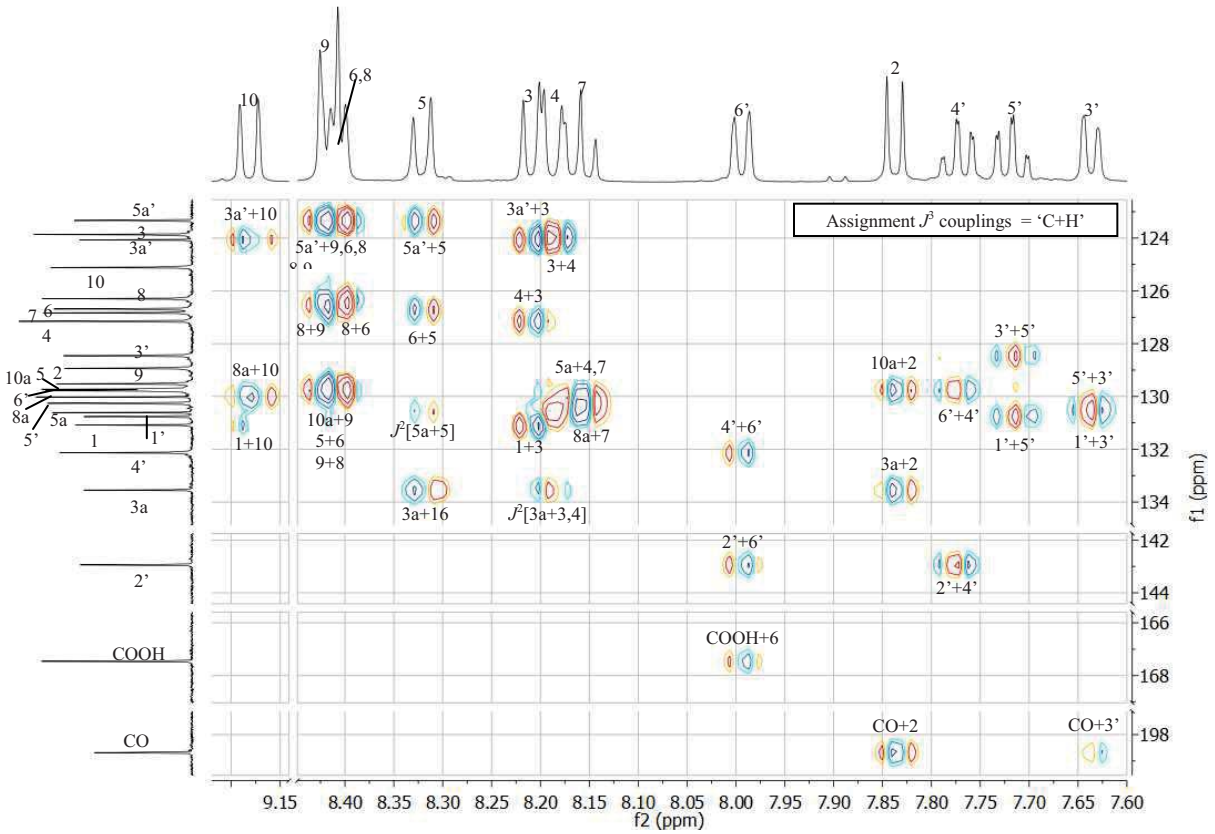

Figure S12. {^{1}H–^{13}C} HMBC of compound **2** in DMSO-d^6.

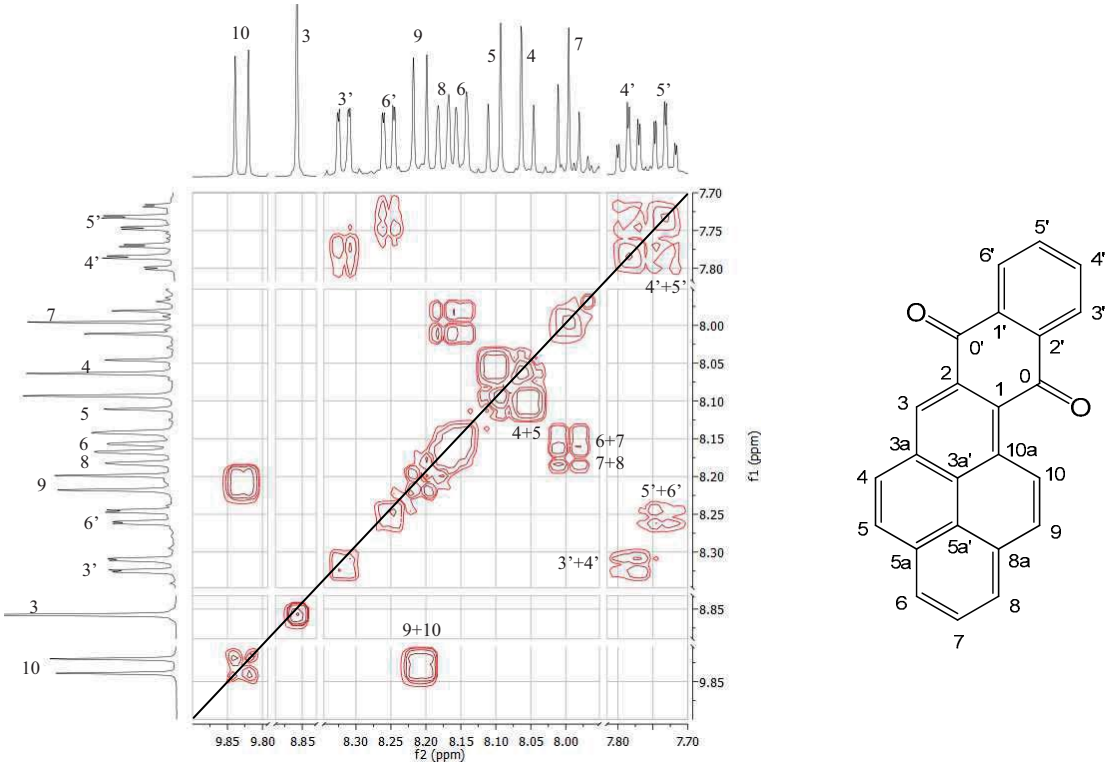

Figure S13. {^{1}H–^{1}H} COSY of compound **3** in $CDCl_3$.

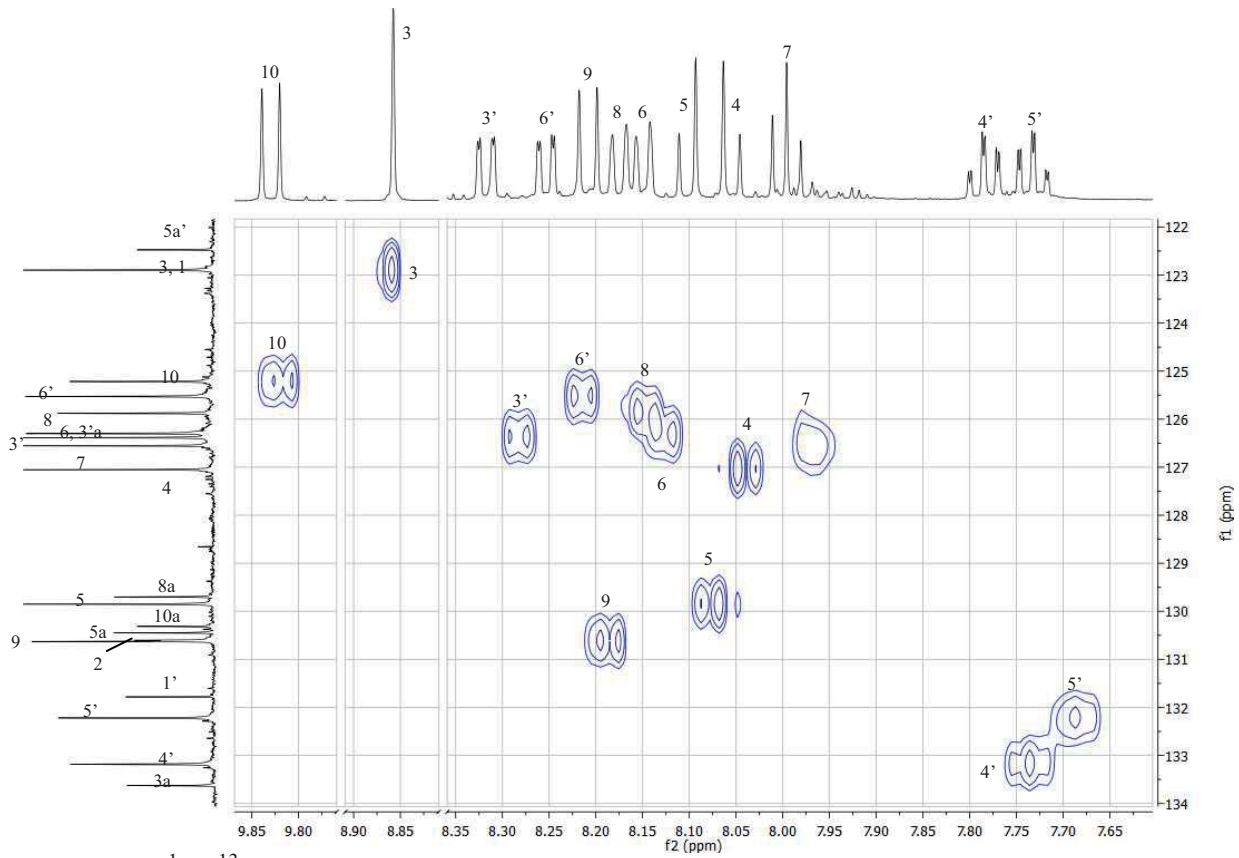

Figure S14. {^{1}H–^{13}C} HMQC of compound **3** in $CDCl_3$.

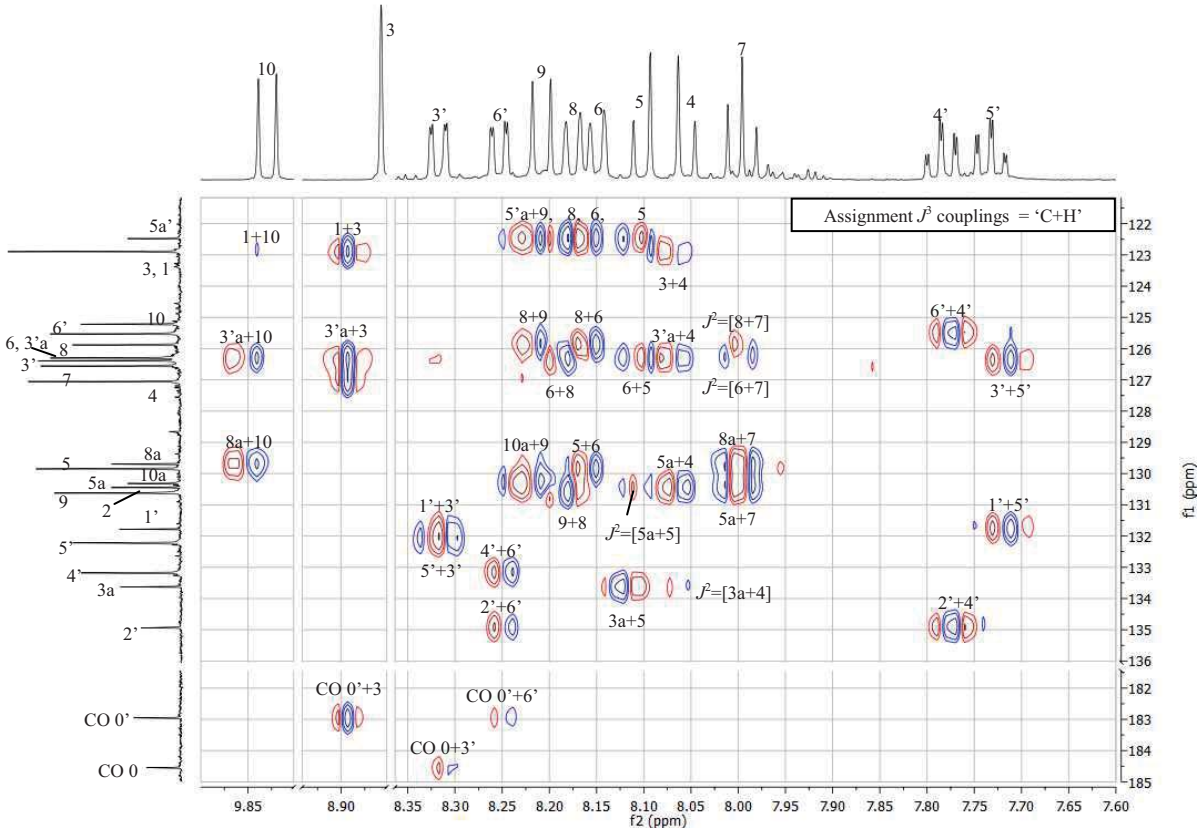

Figure S15. $\{^{1}H–^{13}C\}$ HMBC of compound **3** in $CDCl_3$.