A Catecholamine Neurotransmitter: Epinephrine as a CO_2 Wet Scrubbing Agent

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Electronic Supplementary Information (ESI)

1. Materials and Methods

A. Chemicals

All chemicals were used without purification. Epinephrine hydrochloride (EPN•HCl), dimethylsulfoxide (DMSO-d_6), deuterium oxide (D_2O, 99 atom% D), sodium hydride (NaH, 60% in mineral oil), sodium bicarbonate ($NaH^{13}CO_3$, 99 atom% ^{13}C), monoethanolamine (MEA, 98%, d = 1.012 g.mL^{-1}), and *o*-catechol (CAT, ≥ 99%) were purchased from Sigma-Aldrich. Moreover, 1,1,3,3-tetramethylguanidine (TMG, 99%) and DMSO (HPLC/Spectro grade) were obtained from Acros and TEDIA, respectively. The metal hydroxides, NaOH and KOH were purchased from Gainland Chemical Co. and Net Tech Ltd, respectively. CO_2 (industrial grade) together with N_2 (industrial grade) were purchased from Advanced Technical Gases Co. (Amman, Jordan).

B. Instruments

Solution ^1H, ^{13}C, and ^1H-^{15}N heteronuclear single quantum coherence (HSQC), nuclear magnetic resonance (NMR) spectra were collected at room temperature using (AVANCE-III 400 MHz (^1H: 400.13 MHz, ^{13}C: 100.61 MHz, ^{15}N: 40.560 MHz) FT-NMR NanoBay spectrometer (Bruker, Switzerland). *Ex situ* ATR-FTIR spectra were recorded using a Bruker Vertex 70-FT-IR spectrometer at room temperature coupled with a Vertex Pt-ATR-FTIR accessory.

C. Experimental procedures

In a 100 mL, Schlenk flask, 45 mg (0.204 mmol) of EPN•HCl was dried *in vaccuo* for two hours using a Schlenk line. The sample was dissolved in a 1.0 mL DMSO-d_6. Upon dissolution, a NaOH pellet (*ca*. 426 mg, 10.6 mmol) or NaH (*ca.* 22.1 mg, 0.92 mol; after rinsing with hexane five to six times) were introduced under CO_2 atmosphere *via* Schlenk line and stirred for 30.0 minutes. Because of the limited solubility of KOH (0.13 g\L in in DMSO), 22 mg of EPN•HCl was used. A shorter stirring time (10 minutes) was applied when the sample preparation was conducted under N_2 atmosphere. For the model compounds, a 1.0 mL (16.57 mmol) of neat MEA was mixed with 1.0 mL DMSO-d_6 and bubbled with CO_2 for 30.0 minutes. In the case of CAT, a 168 mg (1.53 mmol) was dissolved in 1.0 mL DMSO-d_6. The solution was bubbled with CO_2 for 30.0 minutes upon activation with NaOH pellet (*ca*. 413 mg, 10.3 mmol). When TMG (*ca*. 0.38 ml, 3 mmol) was used, the bubbling was spanned for 60 minutes. It is worth to mention that a pink suspension was formed when the sample was treated with NaOH.

For the *ex situ* ATR-FTIR measurements, the same protocols were followed using normal DMSO as a solvent.

D. DFT Calculations

Calculations were performed by using Gaussian 09.* Geometry optimizations and energy calculations were carried out using the M026X functional with the 6-311G** basis set at 298 K and 1 atm. Minima were characterized by the absence of imaginary frequencies. A polarisable continuum model (PCM) was used for implicit solvent calculations. Carbon and nitrogen chemical shifts were computed using the gauge-independent atomic orbital (GIAO) method, in which tetramethylsilane and NH_3 were used as reference for the ^{13}C and ^{15}N, respectively.

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2. Tables

Table S1. ^{1}H NMR chemical shift (ppm) of EPN•HCl and its basic form upon activation with KOH, NaH and NaOH under N_2 and CO_2 atmospheres.

	EPN•HCl		EPN/KOH		EPN/NaH		EPN/NaOH	
	as received	CO_2	N_2	CO_2	N_2*	CO_2	N_2	CO_2
CH_3	2.54	2.71	2.48	2.54	n.a	2.71	2.25	2.71
CH_2	2.92	3.15	2.84	2.95	n.a	3.10	2.40	3.10

** The measurement was unsuccessful due to the formation of coagulates.*

3. Figures

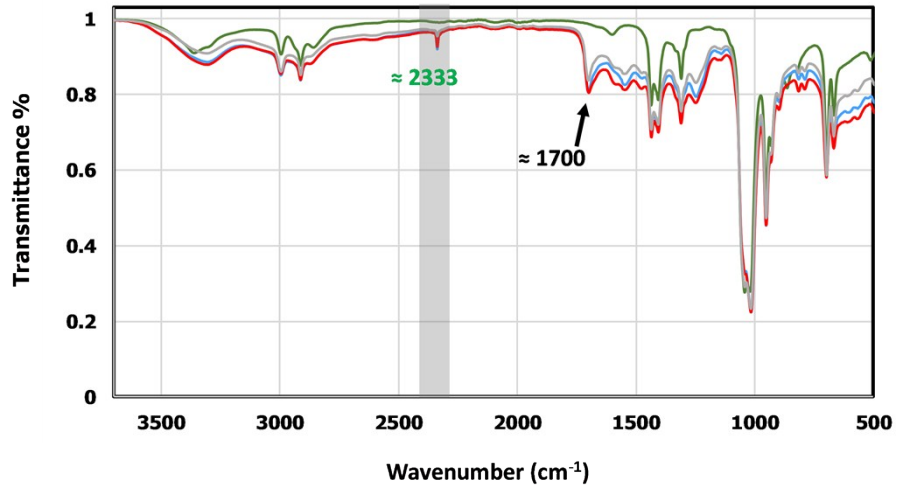

Figure S1. ATR-FTIR spectra of MEA/DMSO before (**green**) and after bubbling with CO_2 (**blue**), and upon activation with NaOH (**red**) and KOH (**grey**)

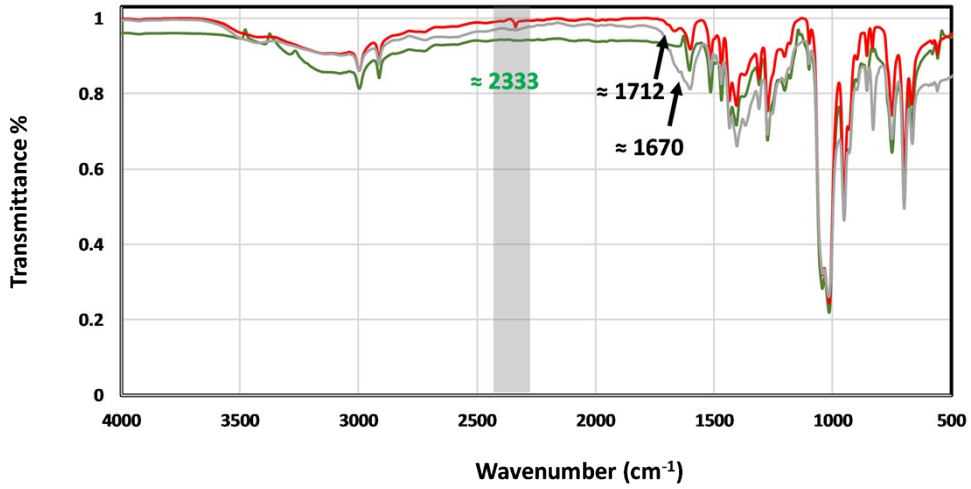

Figure S2. ATR-FTIR spectra of catechol/DMSO before (**green**) and after bubbling with CO_2 upon activation with NaOH (**red**) and KOH (**grey**)