Supporting Information

Microwave Assisted Construction of a $SnO_x/ZnSn(OH)_6$ Heterojunction for Photocatalytic CO_2 Cycloaddition

*Soumita Sarkar, Soumalya Banerjee, Sk Afsar Ali, Sunny Sarkar, and Astam K. Patra**

Department of Chemistry, University of Kalyani, Kalyani 741235, West Bengal, India.

**Corresponding author email E-mail: astamchem18@klyuniv.ac.in, Orcid.org/0000-0001-6071-8653.*

EXPERIMENTAL DETAILS

Materials

Zinc nitrate hexahydrate (96.0%, Merck), Tin (IV) chloride pentahydrate (98.0%, Sigma Aldrich), Sodium hydroxide (97%, SRL), Sodium Salicylate (Merck, 99.5%), Tetramethyl ammonium hydroxide (Sigma Aldrich 25% in water) were used for materials synthesis. All the reactions were carried out in deionised water. Styrene oxide (97.0%, Merck), Benzyl glycidyl ether (99.0%, Merck), Phenyl glycidyl ether (99.0%, TCI), Allyl glycidyl ether (99.0%, TCI), Epichlorohydrin (99.0%, Avra), Butylene oxide (99.0%, TCI), Propylene oxide (99.0%, TCI), *tetra*-butylammonium bromide (TBAB, 98.0%, Himedia), Acetonitrile (99.9%, Merck), DMF (99.9%, Merck), Ethylene glycol (Merck), Poly ethylene glycol 600 (PEG 600, Merck), Ethanol (99.9%, CSS reagent), Ethyl acetate (98%, Finar), Potassium Iodide (99.5%, Merck), Potassium Bromide (99.0%, Merck), Silver nitrate (99.0%, Sigma Aldrich), Sodium oxalate (99.8%, Merck), and 5,5-dimethyl-1-pyrroline N-oxide (DMPO, 97%, BLD pharma) were used in catalytic activity test. Nafion (5 wt% in water ethanol, Sigma Aldrich) was used in electrochemical analysis. All the chemicals were used as purchased; no further purification was done.

Synthesis of Zinc Oxide

ZnO nanomaterial was synthesized through the following previous procedure.[1] Briefly, 1.6 g sodium salicylate and 5 mL tetramethyl ammonium hydroxide was dissolved in 20 g water. The solution was stirred for 15 min. Then 4.4 g $Zn(NO_3)_2$ $6H_2O$ was dissolved in 5.0 g distilled water and this solution was slowly added to the above solution. The pH of the solution was adjusted to ca. pH = 9 by addition of tetramethylammonium hydroxide solution. Then the mixture was hydrothermally treated at 348 K for 24 h. The white solid was collected by filtration, dried at room temperature. Further, the as-synthesized material was heated at 423 K for 1 h for complete conversion to zinc oxide from zinc hydroxide. The ZnO material was characterized by powder XRD analysis (Figure S29a).

Synthesis of Tin Oxide

SnO_2 nanomaterial was prepared using the hydrothermal method following previous procedure.[2] Briefly $SnCl_4.5H_2O$ was introduced into a mixed solution of 2-propanol and distilled water with a ratio of 4:1. The solutions were adjusted to pH 12 by the addition of 2 (M) NaOH. Then transferred to a stainless autoclave, and heated at 150 °C for 24 h. Then as-synthesized SnO_2 was thermally treated at 300 °C for 1 h under. The SnO_2 material was characterized by powder XRD analysis (Figure S29b).

Light-on/Light-off Experiment

The experiments were conducted following the standard procedure, utilizing 3 mmol of styrene oxide, 0.3 mmol TBAB, 50 mg of the SnOx/$ZnSn(OH)_6$ catalyst, 2 mL of PEG-600, CO_2 balloon and a 8 Watt UV light (254 nm) at room temperature. The light was on for 6 h and then off for 6 h after that light was on again for 6 h.

Quenching Experiment

Here $AgNO_3$, $Na_2C_2O_4$, and 5,5-dimethyl-1-pyrroline N-oxide (DMPO) were used as scavengers to trap e^-, h^+, and radicals, respectively. The molar ratio between styrene oxide and trapping agents was 5:1. The catalytic experiments were carried out with standard reaction condition to study the formation rates of styrene carbonate. For the catalytic reaction, 3 mmol of epoxide, 2 mL of PEG-600, 0.3 mmol TBAB and 0.6 mmol scavengers were taken in a round bottom flask purged with CO_2 balloon. Then 50 mg of SnOx/$ZnSn(OH)_6$ catalyst were added to the solution. The solution was stirred under a light illumination of UV Light (8 Watt, 254nm) at room temperature. The progress of the reaction was monitor by TLC.

Recyclability Test

In order to assess the stability and efficiency of our SnOx/$ZnSn(OH)_6$ catalyst for repeated catalytic studies, we implemented a method of collecting the catalyst after each experiment. The reactions were performed under standard conditions. We then thoroughly washed the material with ethanol and water to remove any adsorbed substances, and subsequently dried it in a vacuum to ensure complete removal of any residual moisture. This process allowed us to accurately evaluate the performance of our catalyst over multiple catalytic synthesis cycles.

Photoelectrochemical Characterization

Electrode Preparation: The working electrode used in this experiment consisted of SnOx/$ZnSn(OH)_6$ materials on fluorine-doped tin oxide (FTO), with a 0.5 M Na_2SO_4 electrolyte solution. The working electrode was prepared using standard techniques. FTO glasses were meticulously cleaned with acetone and ethanol using a sonication bath. To disperse 2 mg of powdered SnOx/$ZnSn(OH)_6$, a mixture of 80 μL dry ethanol and 20 μL 5%

Nafion was employed, followed by 30 minutes of sonication. Subsequently, a 5 μL suspension was delicately drop-cast onto an FTO-coated plate, resulting in a circular film. The film was then dried under ambient humidity conditions to create a functional electrode.

Photoelectrochemical Measurements: The photoelectrochemical experiments were conducted using a computer-controlled electrochemical workstation (BioLogic 150e) in a standard three-electrode system. A Pt wire and a saturated Ag/AgCl electrode (in 3 M NaCl) were used as the counter and reference electrodes, respectively. White light was provided by a 300 W Xe lamp with a light intensity of 100 mW/cm^2 and a photon flux of 4.153×10^{17} cm^{-2} s^{-1}. Mott−Schottky plots were generated at frequencies of 1, 2, and 3 kHz under dark conditions in 0.5 M Na_2SO_4 solution to determine the flat-band potential, with a perturbation signal set at 10 mV. The chronoamperometry experiment was carried out in 0.5 M Na_2SO_4 solution at a bias of 1.2 V vs Ag/AgCl (NaCl Sat'd) using the chopping illumination method, where the beam intensity was alternately turned on and off. Electrochemical impedance spectroscopy (EIS) measurements were conducted in both light and dark environments. An AC voltage with amplitude of 10 mV within a frequency range of 100 kHz−100 mHz was applied to a 0.5 M Na_2SO_4 solution at a bias potential of 1.5 V vs Ag/AgCl (NaCl Sat'd). An equivalent circuit model was utilized to determine the solution resistance (R1) and charge transfer resistance (R2). The applied potentials vs Ag/AgCl (NaCl Sat'd) were converted to RHE (Reversible Hydrogen Electrode) potentials using the following equations:

$E_{RHE} = E_{Ag/AgCl} + 0.0591pH + E^{\theta}_{Ag/AgCl}$ ($E^{\theta}_{Ag/AgCl}$= 0.194 V vs NHE at 25 °C)

ESR experiment

The quenching experiment was further analyzed using Electron Spin Resonance (ESR) measurements. Both the alkyl radical (Styrene epoxide) and the $CO_2^{\bullet-}$ radical generated during the reaction were captured using 5,5-Dimethyl-1-pyrroline N-oxide (DMPO) as a radical quencher. Initially, a mixture consisting of 3 mmol of epoxide substrate (ES), 10 mg of catalyst, and 50 μL of DMPO was prepared under an argon atmosphere. This reaction mixture was then transferred into a capillary tube and positioned within an ESR tube for analysis. The ESR measurements, conducted under light-irradiated conditions (using 254 nm UV light), revealed characteristic signals corresponding to the alkyl radical adduct generated from styrene oxide. A similar experimental procedure was employed to investigate the quenching of the $CO_2^{\bullet-}$ radical. In this case, a mixture of 10 mg of catalyst and 50 μL of DMPO was purged with CO_2 gas for several minutes to ensure complete saturation. ESR measurements were subsequently performed after light irradiation. All measurements were conducted at room temperature, ensuring consistent conditions for accurate analysis.

Characterization Techniques

The synthesized materials were analyzed using various characterization techniques. Morphology analysis was conducted using a JEOL JEM 6700 Field Emission Scanning Electron Microscope (FE SEM). The powder X-ray diffraction patterns of the samples were obtained using a Bruker D-8 Advance diffractometer, operating at 40 kV voltage and 40 mA current, and utilizing Cu Ka radiation with a wavelength of 0.15406 nm. The Powder X-ray diffraction patterns were analyzed using MATCH software, Version 3.x, by CRYSTAL IMPACT located at Kreuzherrenstr, 102, 53227 Bonn, Germany to determine the phase of the synthesized materials. Fourier transform infrared (FT−IR) spectra were recorded on a Perkin Elmer–L120−000A spectrophotometer. TEM images were captured using a JEOL 2010 TEM operating at 200 kV. X-ray photoelectron spectroscopy (XPS) was performed on a Thermo Scientific (Model No ESCALAB 250Xi). X-ray Photoelectron Spectrometer operated at 15 kV and 20 mA with a monochromatic Al Kα X-ray source. UV–visible diffuse reflectance spectra were recorded on a Shimadzu UV2401PC with an integrating sphere attachment. BaSO4 was used as background standard. The electrochemical experiments were conducted using a computer-controlled electrochemical workstation (BioLogic 150e) within a standard three-electrode system. ^{1}H and ^{13}C NMR experiments were carried out on a Bruker Ultrashield 400 MHz NMR spectrometer. The ESR measurements were carried out using continuous wave spectrometers operating at X−band frequency (9−10 GHz) (BRUKER BIOSPIN, Germany, Model−EMXmicro A200−9.5/12/S/W).

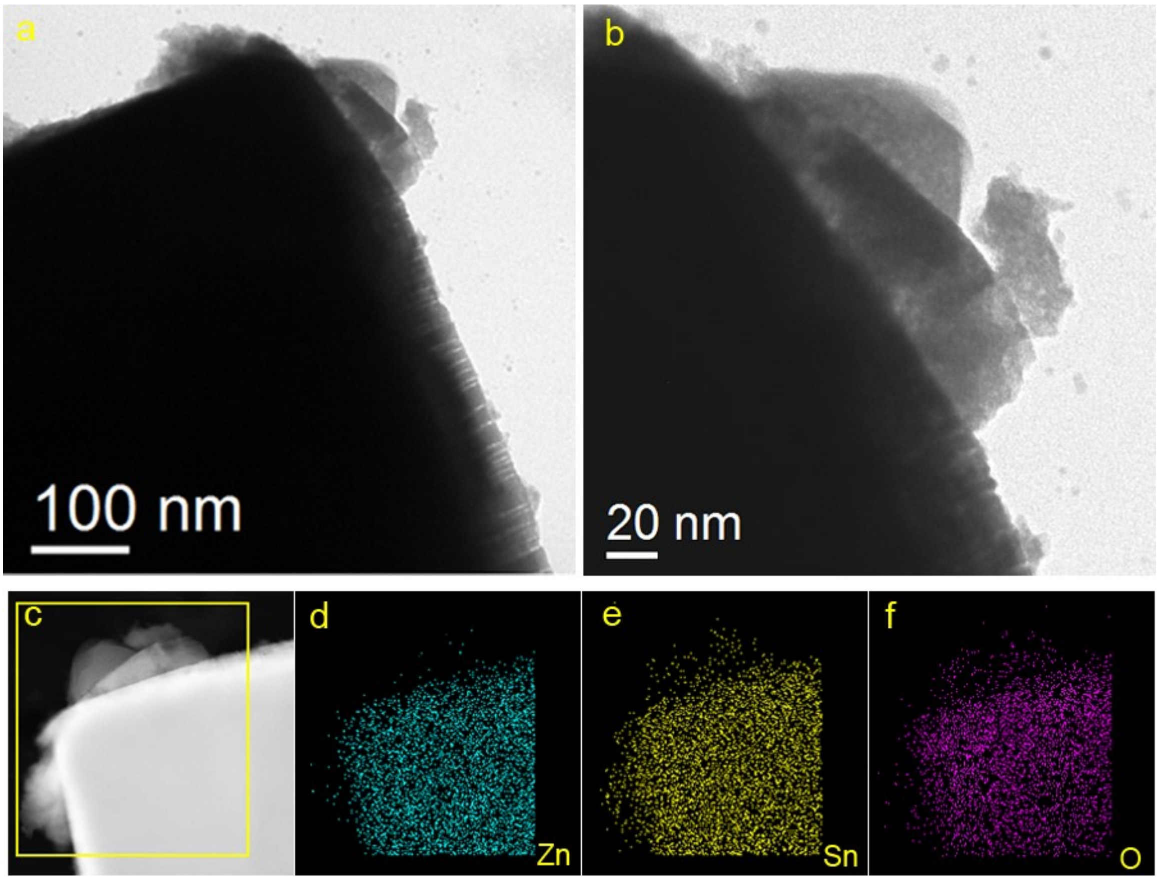


Fig. S1. a-b) TEM images of SnOx/ZnSn$(OH)_6$ materials and elemental mapping of the area in c) showing the elements d) Zn, e) Sn and f) O.

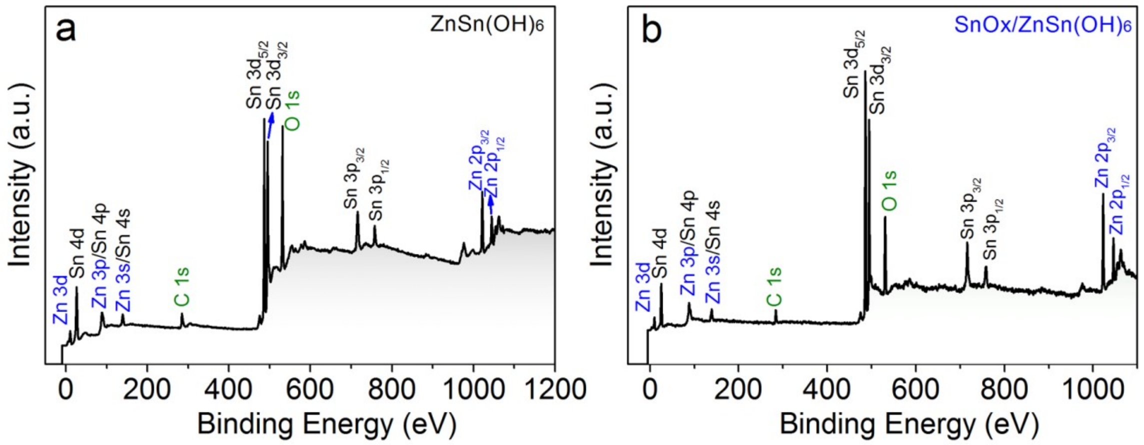
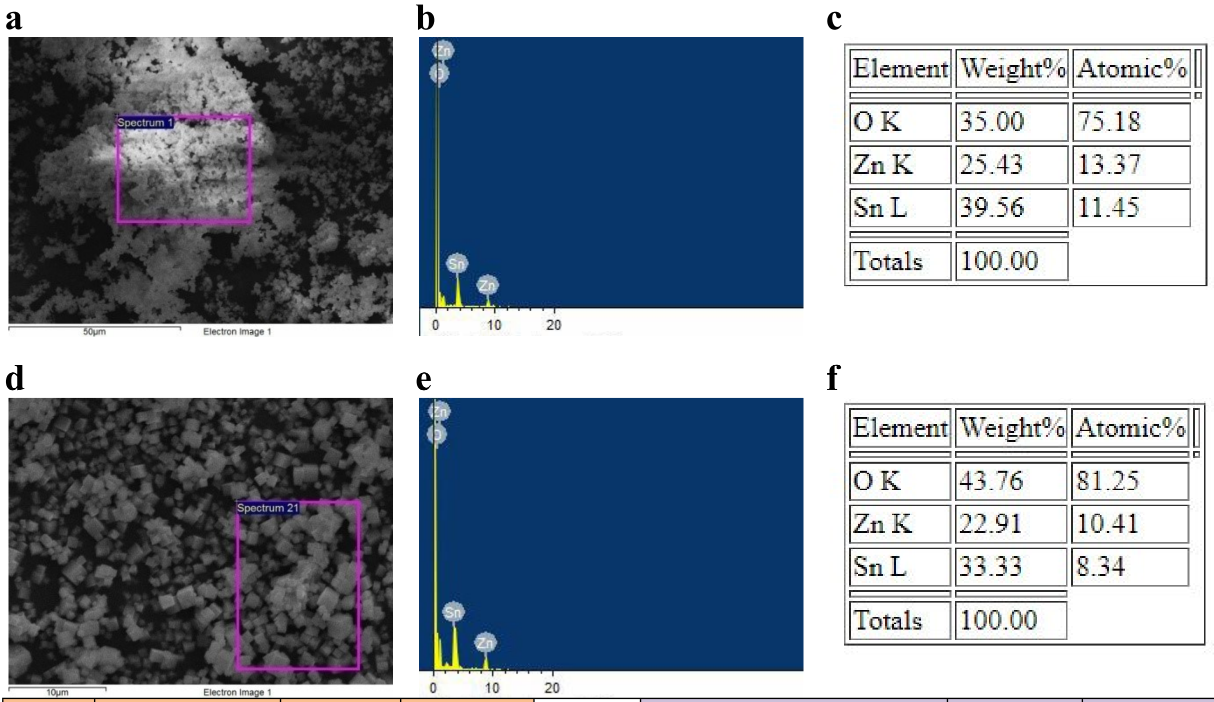


Fig. S2: XPS survey profile of a) $ZnSn(OH)_6$ and b) $SnOx/ZnSn(OH)_6$ microcubes.



Element	Weight%	Atomic%
O K	35.00	75.18
Zn K	25.43	13.37
Sn L	39.56	11.45
Totals	100.00	

Element	Weight%	Atomic%
O K	43.76	81.25
Zn K	22.91	10.41
Sn L	33.33	8.34
Totals	100.00	

	$ZnSn(OH)_6$ (Atomic %)	**Zn/O**	**Zn/Sn**
O	**75.18**	**0.1778**	**1.1676**
Zn	**13.37**		
Sn	**11.45**		

$SnOx/ZnSn(OH)_6$ (Atomic %)	**Zn/O**	**Zn/Sn**
81.25	**0.1281**	**1.2482**
10.41		
8.34		

Fig. S3: a-c) EDX analysis of $ZnSn(OH)_6$ materials and d-f) EDX analysis of $SnOx/ZnSn(OH)_6$ materials. The table shows the Zn/O and Zn/Sn ratios.

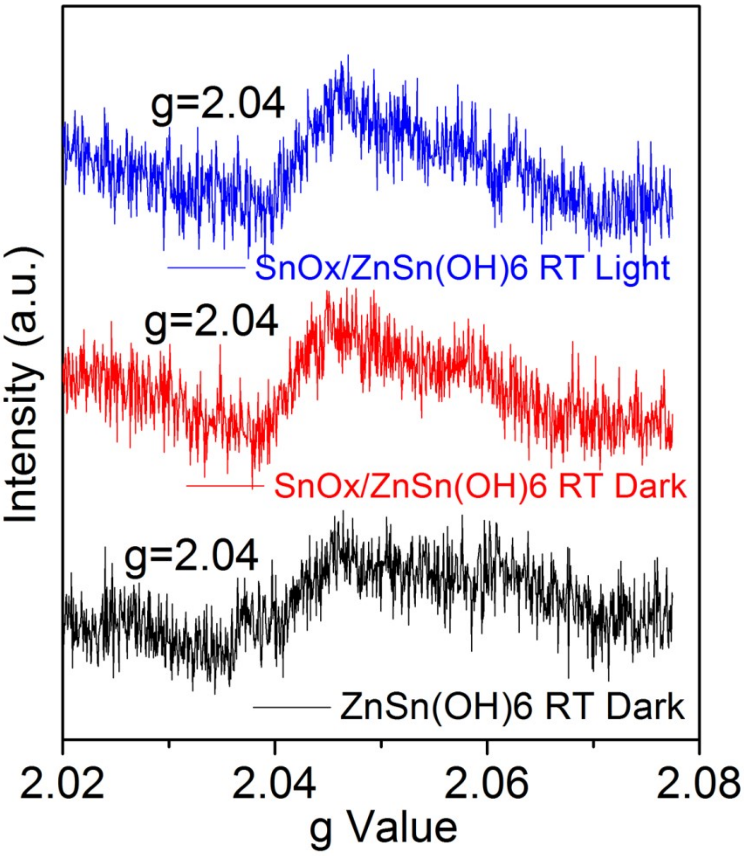


Fig. S4: ESR spectra of $ZnSn(OH)_6$ and $SnOx/ZnSn(OH)_6$ under dark and light.

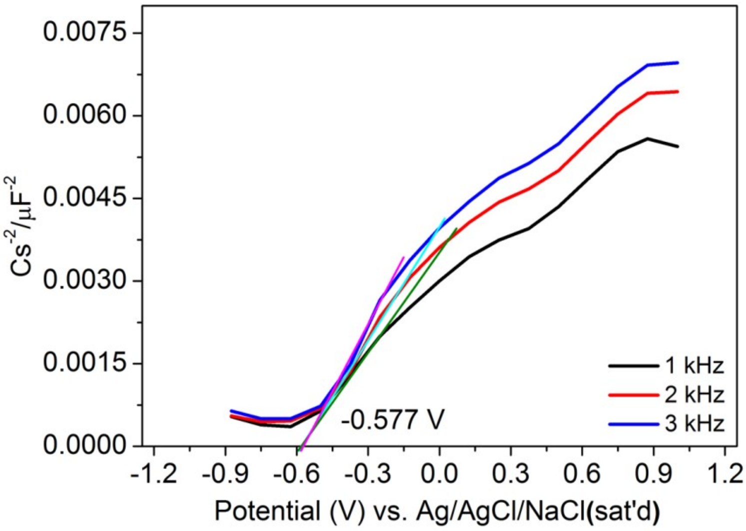


Fig. S5: Mott−Schottky plots of $ZnSn(OH)_6$ in different frequencies.

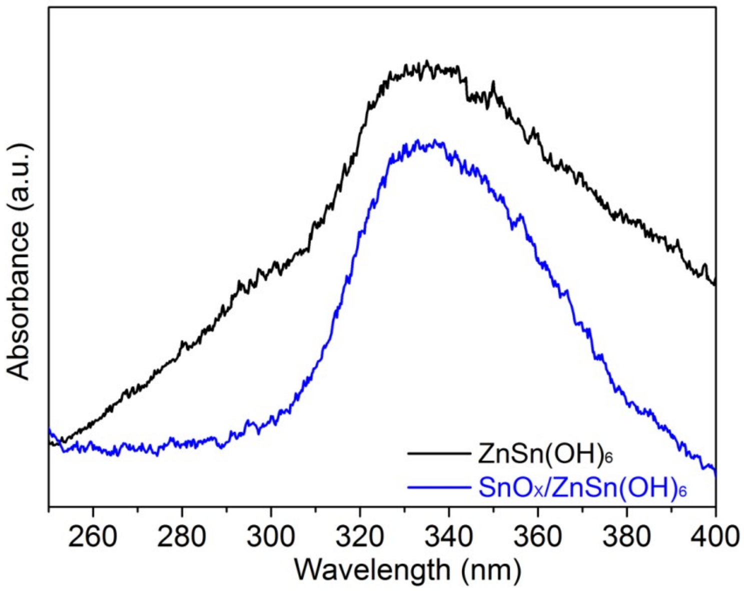


Fig. S6: The steady-state photoluminescence (PL) spectra of $ZnSn(OH)_6$ and $SnOx/ZnSn(OH)_6$.

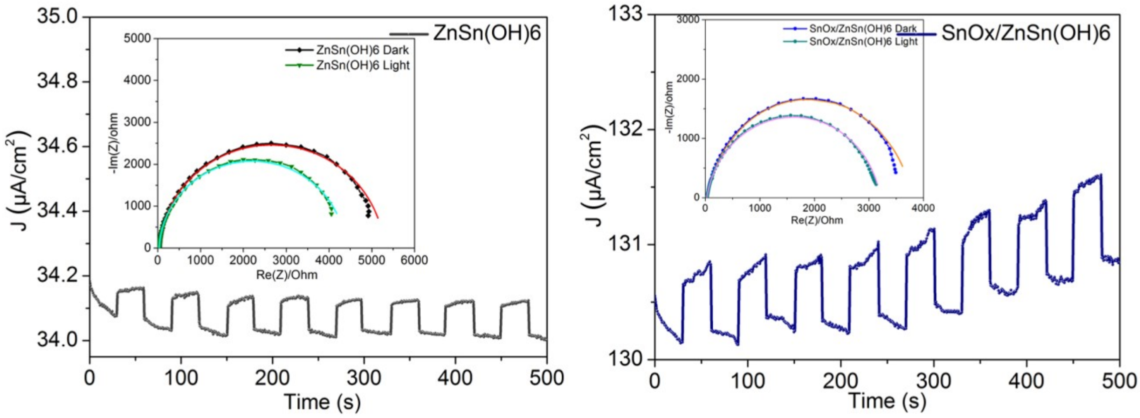
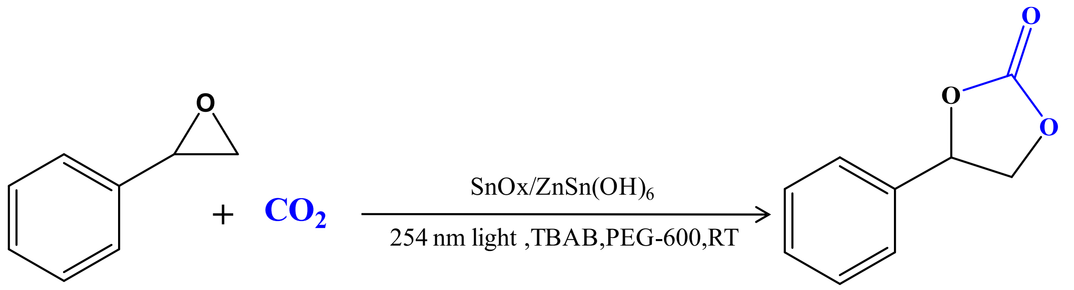


Fig. S7: Photocurrent responses and EIS Nyquist plots (inset) of $ZnSn(OH)_6$ and $SnOx/ZnSn(OH)_6$. microcubes in presence and absence of light (254 nm light).

Table S1: Summary of electrochemical properties of $ZnSn(OH)_6$ and $SnOx/ZnSn(OH)_6$ materials using different light sources.

	Sample	**R1 (ohm)**	**CPE (F s^(a-1))**	**R2 (Ohm)**
300 W Xe Lamp	$ZnSn(OH)_6$ dark	20.51	22.3e-6	1155.0
	$ZnSn(OH)_6$ light	18.89	44.65e-6	644.3
	$SnOx/ZnSn(OH)_6$ dark	14.11	27.42e-6	746.0
	$SnOx/ZnSn(OH)_6$ light	12.93	31.06e-6	464.1
254 nm UV light (8 watt)	$ZnSn(OH)_6$ dark	31.77	8.725e-6	4957
	$ZnSn(OH)_6$ light	33.06	8.725e-6	4048
	$SnOx/ZnSn(OH)_6$ dark	28.29	18.7e-6	3608
	$SnOx/ZnSn(OH)_6$ light	28.35	19.67e-6	3124

Green Chemistry Metrics



MW	120.15	44	164.16
	3 mmol	3 mmol	2.79 mmol
	0.360 g	0.132 g	0.4579 g

$$\text{Atom Economy (\%)} = \frac{\text{Molecular weight of desired products}}{\text{Molecular weight of all reactants}} \times 100$$

$$= 164.16/(120.15+44)\times 100$$

$$= 100$$

$$\text{Reaction Mass Efficiency (\%)} = \frac{\text{Mass of desired products}}{\text{Mass of all reactants}} \times 100$$

$$= 0.4579/(0.3604+0.1320)\times 100$$

$$= 92.98$$

$$\text{Process Mass Intensity} = \frac{\text{Total mass in a process or process step (g)}}{\text{Mass of product (g)}}$$

$$= (0.3604+0.1320+2.252)/0.4579$$

$$= 5.99$$

$$\text{E factor} = \frac{\text{Total mass of waste (g)}}{\text{Mass of product (g)}}$$

$$= \frac{\text{Total mass in a process or process step (g)} - \text{mass of product (g)}}{\text{Mass of product (g)}}$$

$$= (0.3604+0.1320+2.252-0.4579)/0.4579$$

$$= 4.99$$

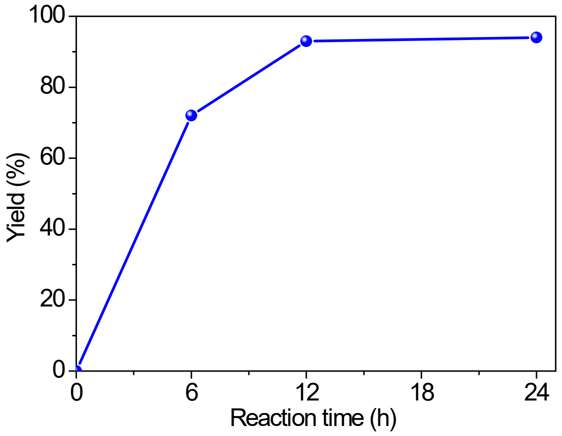
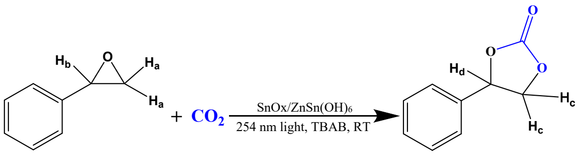


Fig. S8: Reaction kinetics of CO_2 cycloaddition reaction with styrene oxide over SnOx/ZnSn(OH)$_6$ catalyst.

Product Quantification from NMR



$$\text{Conversion}\,(\%) = \frac{I_{Hd}}{I_{Hd} + I_{Hb}} \times 100$$

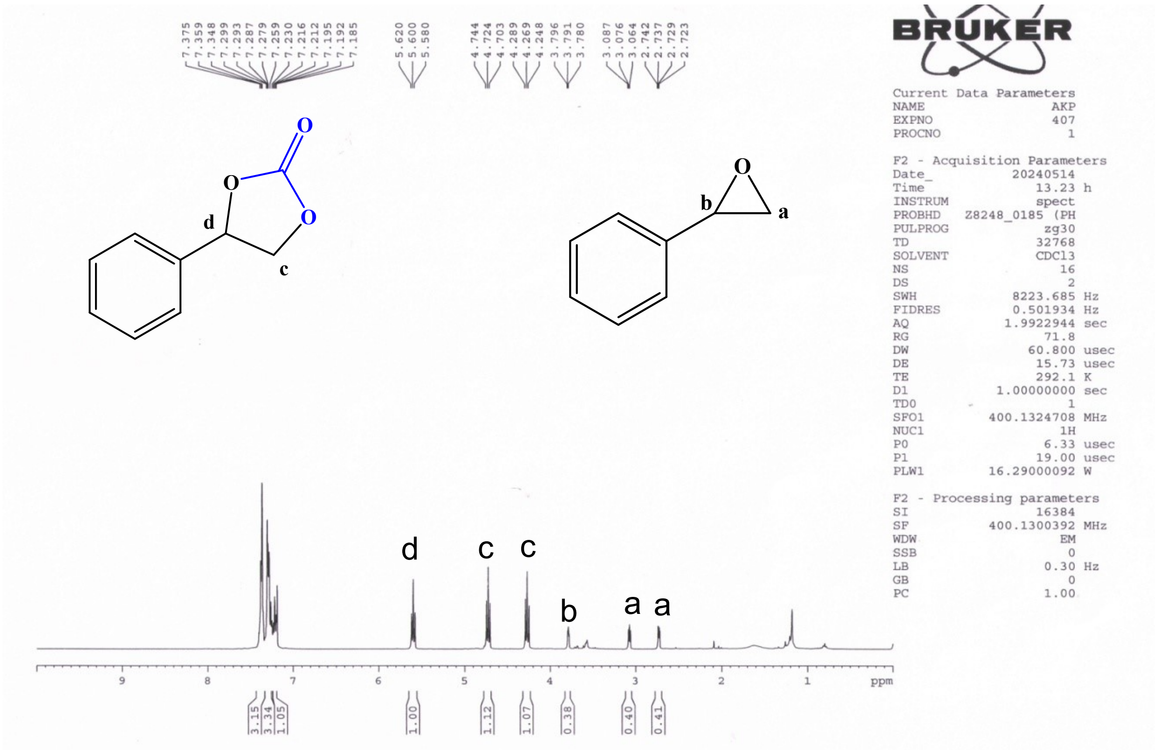


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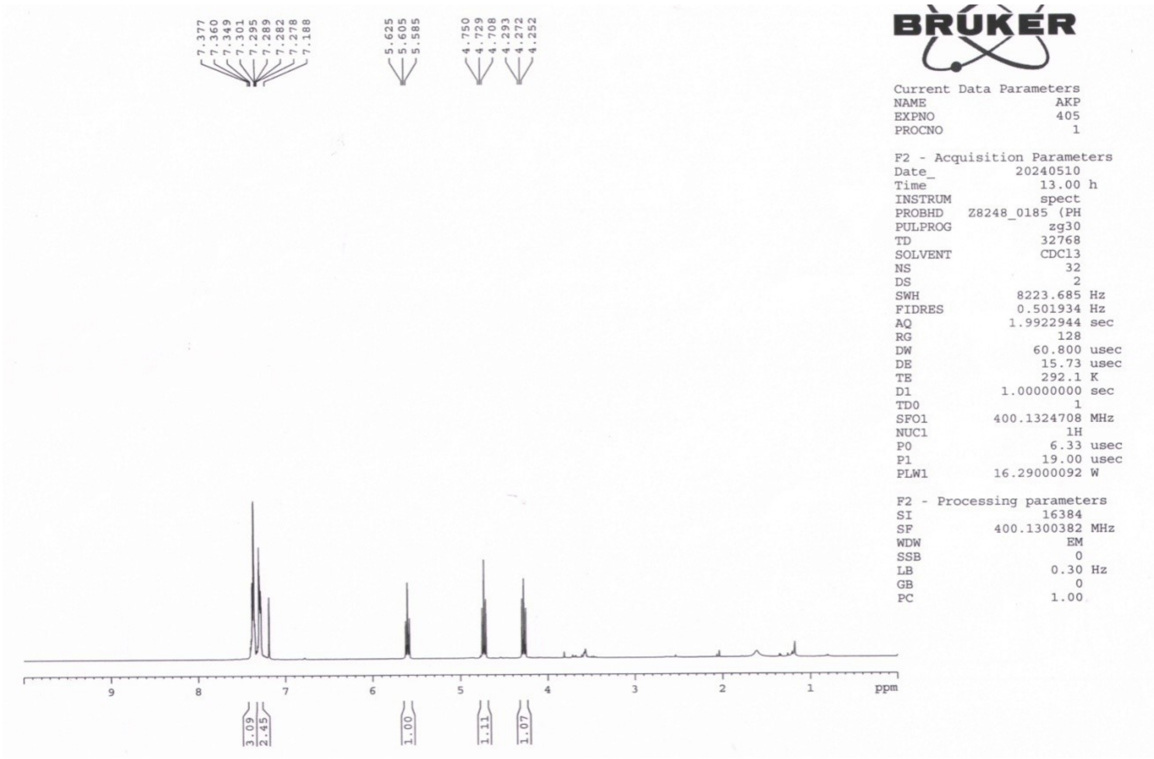
Fig. S9: ^{1}H NMR (400 MHz, $CDCl_3$) spectra of crude product of CO_2 cycloaddition reaction with styrene oxide after 6h reaction over SnOx/ZnSn(OH)$_6$ catalyst.



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TE                  292.1 K
D1             1.00000000 sec
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NUC1                   1H
P0                   6.33 usec
P1                  19.00 usec
PLW1          16.29000092 W

F2 - Processing parameters
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LB                   0.30 Hz
GB                      0
PC                   1.00
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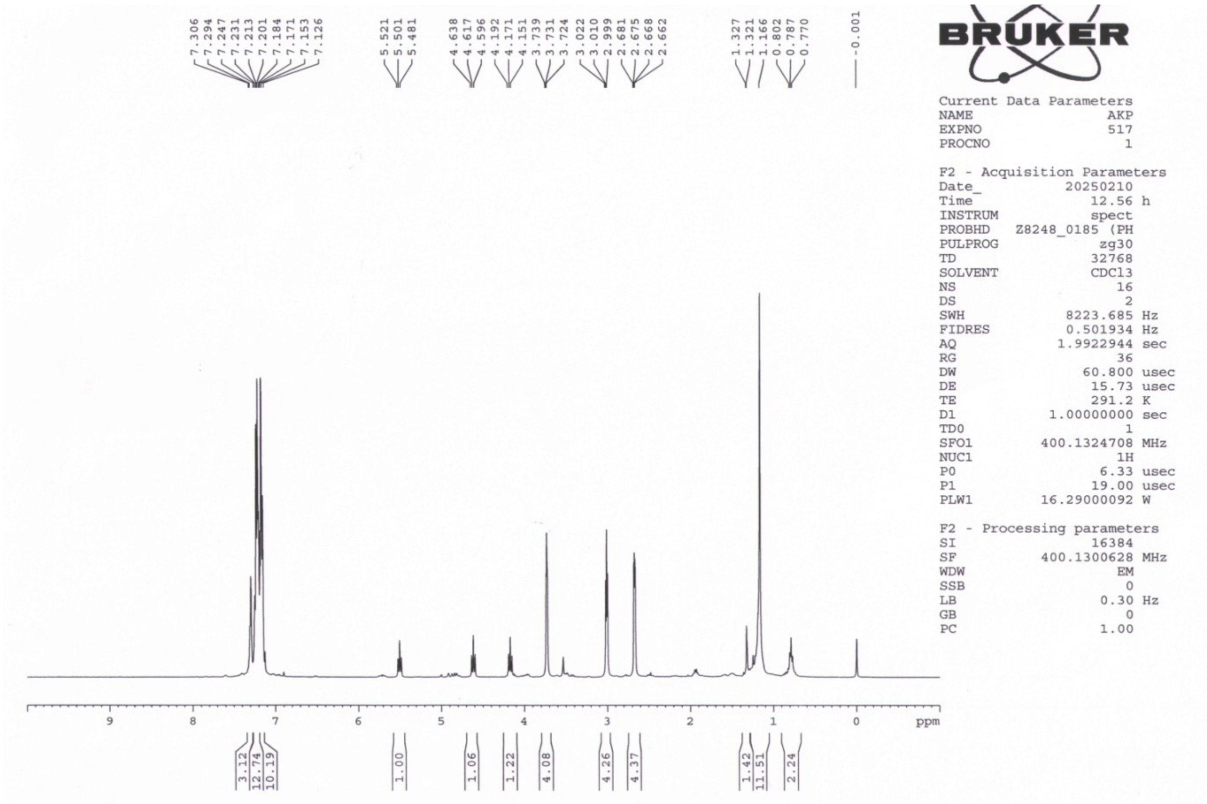
Fig. S10: ^{1}H NMR (400 MHz, $CDCl_3$) spectra of crude product of CO_2 cycloaddition reaction with styrene oxide after 12h reaction over SnOx/ZnSn(OH)$_6$ catalyst.



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F2 - Processing parameters
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Fig. S11: ^{1}H NMR (400 MHz, $CDCl_3$) spectra of crude after 12h reaction without SnOx/ZnSn(OH)$_6$ photocatalyst.

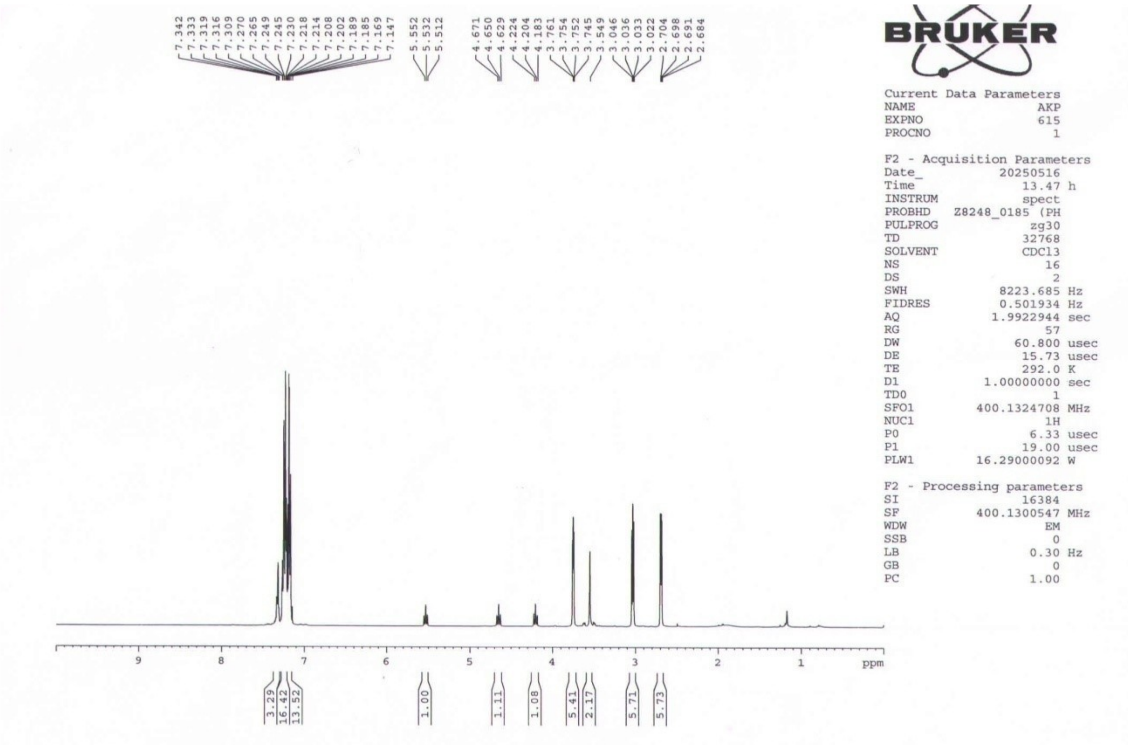


Fig. S12: ^{1}H NMR (400 MHz, $CDCl_3$) spectra of crude after 12h reaction without light.

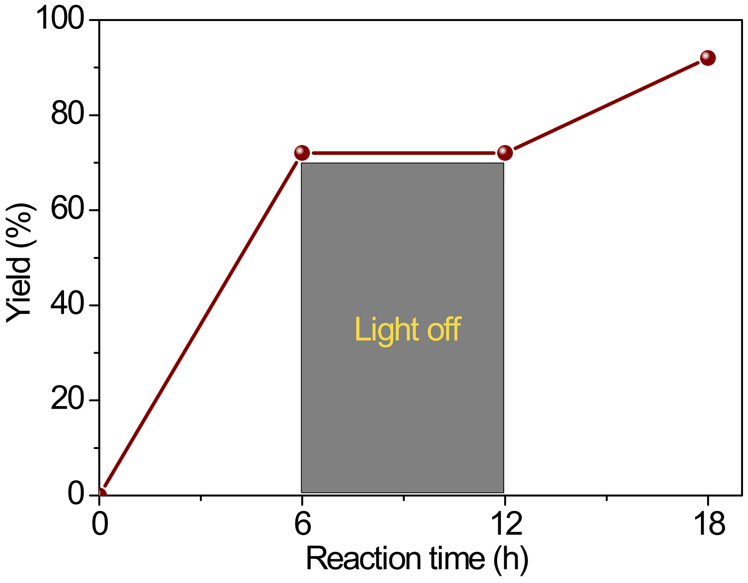


Fig. S13: light-on/light-off experiment for CO_2 cycloaddition reaction.

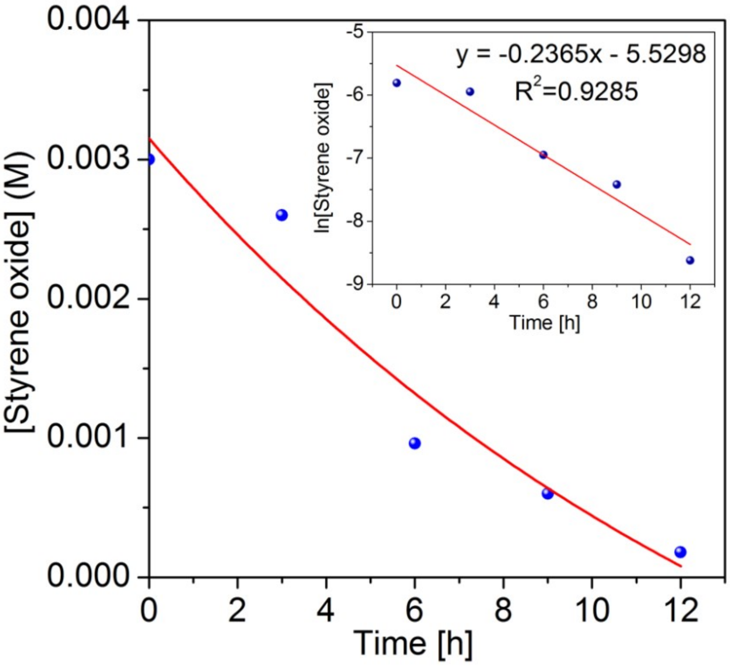


Fig. S14: First order kinetic results with respect to the concentration of epoxide.

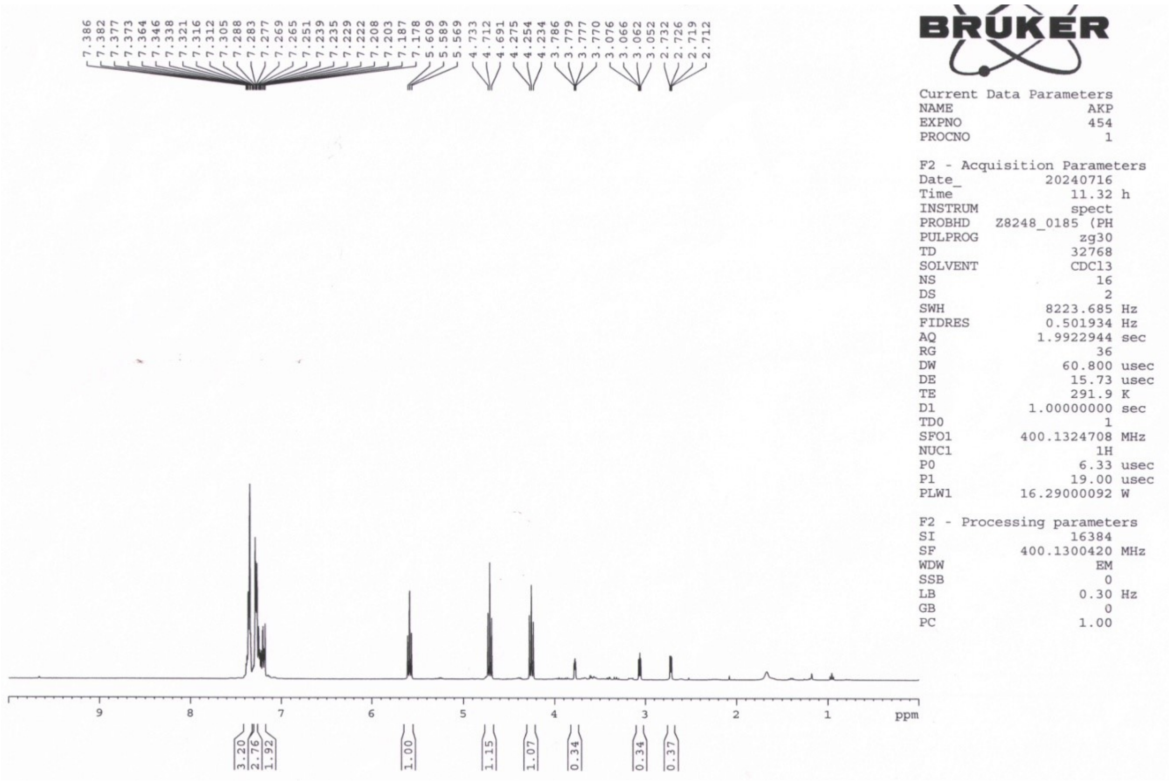


Fig. S15: ^{1}H NMR (400 MHz, $CDCl_3$) spectra of crude product of CO_2 cycloaddition reaction with styrene oxide after 12h reaction over $ZnSn(OH)_6$ catalyst.

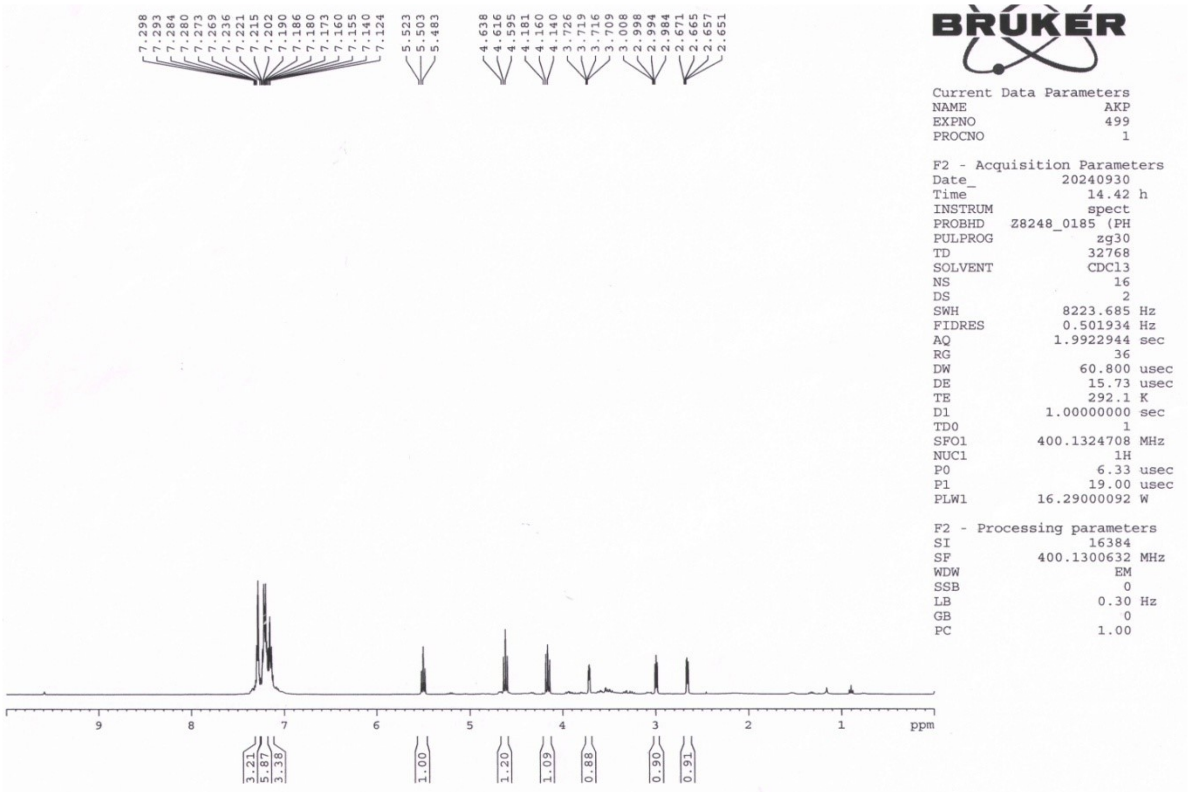
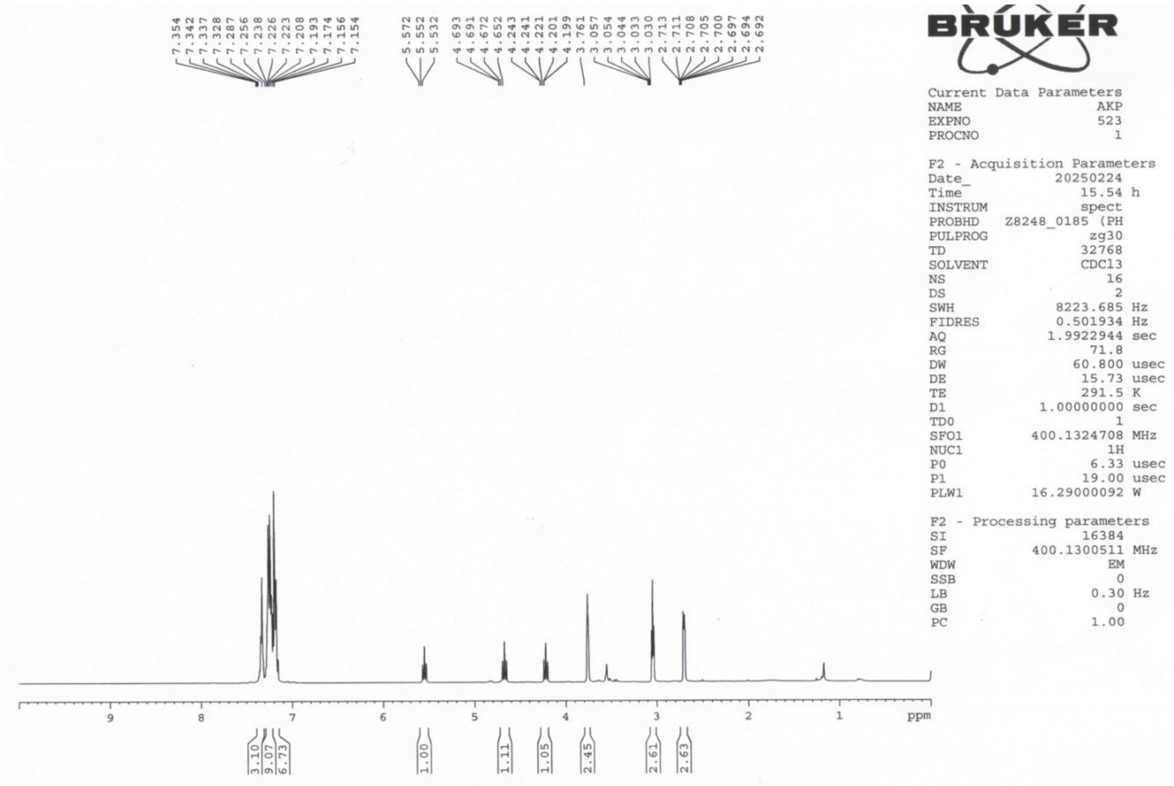


Fig. S16: ^{1}H NMR (400 MHz, $CDCl_3$) spectra of crude product of CO_2 cycloaddition reaction with styrene oxide after 12h reaction over ZnO catalyst.



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RG                 71.8
DW               60.800 usec
DE                15.73 usec
TE                291.5 K
D1           1.00000000 sec
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SFO1        400.1324708 MHz
NUC1                 1H
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P1                19.00 usec
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F2 - Processing parameters
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Fig. S17: ^1H NMR (400 MHz, $CDCl_3$) spectra of crude product of CO_2 cycloaddition reaction with styrene oxide after 12h reaction over SnO_2 catalyst.

Table S2: Different photocatalysts performance comparison for cycloaddition of CO_2 and epoxide

No	Catalyst name	Reaction Condition	Catalytic Process	Yield (%)	Activity (µmol/h/g)	Reference
1	SnOx/ZnSn(OH)$_6$	Epoxide (3 mmol) + catalyst (50 mg) + 1 MPa of CO_2, TBAB, PEG 600, RT, 12h.	Photocatalytic	87-98	4.7 mmol/g/h	**This work**
2	TiO_2/ MWCNT grafted by Pt or Pd nanoparticles	Epoxide (1 mmol) + photocatalyst (1 or 5 wt%) + triethylamine (TEA) (20 mL, v/v: 10:1), acetonitrile, Visible light, 25°C, CO_2 Pressure 1 atm, 1 h.	Photocatalytic	86-98		New J. Chem., 2021,45, 17301-17312
3	Bromide-Functionalized Polyoxometalate ImBr−PS−POM	Styrene oxide (10 mmol) + catalyst (20 mg) + CO_2 atmosphere, visible light ($\lambda > 420$ nm), 10 h.	Photocatalytic	99		ACS Sustain. Chem. Eng., 2024, 12, 16396−16408
4	2D/2D ZnIn2S4/Zn-NC S-Scheme Heterojunction	10 mmol 3-bromopropylene oxide + 30 mg Cat + 0.1 mmol TBAB + 3 mL DMF, 300 W xenon lamp and 1 bar CO_2,.4 h.	Photocatalytic	98.8		ACS Sustain. Chem. Eng., 2024, 12, 6584−659
5	$ZnCl_2$/Py-POP	Styrene oxide (4 mmol) + Catalyst (10 mg) + TBAB (0.5 mmol) + anhydrous $ZnCl_2$ (10 wt %); CO_2 1 atm, 250 W Xe lamp, room temperature, and 10 h.	Photocatalytic	94		ACS Sustain. Chem. Eng., 2023, 11, 2066−2078
6	Al–N–C catalyst	1-bromo-2,3-epoxypropane (0.67 mmol) + Catalyst 20 mg + TBAB 21 mg + 2 mL of N,N-dimethylformamide (DMF), 1 bar CO2, 400 mW cm^{-2}	Photocatalytic		3.52 mmol/g/h	Adv. Mater., 2021, 33, 2103186
7	Ti- CNO nanosheets	5 mmol styrene oxide + 20 mg catalyst + 161.2 mg (0.5 mmol) TBAB, CO_2 + 300 W Xe lamp, 6 h.	Photothermal	98.3		Angew. Chem. Int. Ed., 2024, e202404911
8	$BiNbO_4$/5% r-GO	Styrene oxide (100 µL) + photocatalyst (50 mg) + n Bu_4NBr (9 mg) CO_2 (1.48 MPa) 353 K 24 h + halogen lamp (300 W)	Photothermal	65		ACS Sustain. Chem. Eng., 2020, 8, 12072−12079
9	Cellulose based Schiff-base heterogeneous catalyst (Cell-H_2L)	Epoxide (142.9 mmol) + catalyst (0.57 mmol) + TBAB (0.28 mmol), 100 °C, CO_2 pressure 3 MPa	Thermal	98.7		Ind. Eng. Chem. Res., 2019, 58, 17255−17265
10	Copper polyhedron-based MOFs (Cu-PMOFs)	Epoxide (20 mmol) + activated Cu-PMOFs (0.25 mol % open Cu site) + TBAB (1 mmol), 25 °C, $CO_2$1 bar, 48 h.	Thermal	96		Inorg. Chem. Front., 2013, 3, 1-3
11	Bimetal mixed MOFs	Epichlorohydrine (20 mmol) + 2.0 mol% catalyst + 2.5 mol% co-catalyst NBu_4NBr 70 °C, CO_2 1.0 MPa, 12 h.	Thermal	99		Dalton Trans., 2020, 49, 312
12	Melamine functionalized Fe3O4@SiO2	Styrene oxide (17.5 mmol) + catalyst (30 mg) + co-catalyst (Bu_4NX, X = Cl, Br, I): (0.2 mol%), 70 °C, CO_2 1 atm, 24 h.	Thermal	96		J. CO_2 Util., 2021,49, 101575
13	Au/α-Fe_2O_3	Epichlorohydrin 23 mmol + 50 mg catalyst + TBAI 50 mg (1.09 mol%) 105 °C, CO_2 filled balloon, 20h.	Thermal	91		New J. Chem., 2020,44, 11887-11894

14	Graphene oxides	Styrene oxide (5 mmol) + catalysts (2.5 mg) + DMF (4 mL), 100°C, 1.0 atm of CO_2 6h.	Thermal	99		ACS Sustain. Chem. Eng. 2018, 6, 4204-4211
15	Zn-TPy+PBr−/SBA-15	Epoxide (6.63 mmol) + catalyst (0.05 mol % based on metal content) + 1.5 MPa of CO_2, 120 °C, 3.5 h.	Thermal	99		ACS Sustain. Chem. Eng., 2018, 6, 9237-9245
16	Zinc bromide and ionic liquid $ZnBr_2$ and [C_4-mim]Cl	Styrene oxide (35 mmol) + $ZnBr_2$ 0.4 mmol; [C_4-mim]Cl, 0.8 mmol; 14 MPa CO_2 pressure; 80 °C, 1 h.	Thermal	93		Green Chem., 2004, 6 , 613-616 613
17	Bi_4NbO_8Cl (BNOC-OVs)	Epoxide (0.25 mmol) + catalyst (50 mg) + 5 mL of DMF + TBAB (0.3 mmol) + 1 bar of CO_2 + full spectrum irradiation + controlled reaction temperature of 60 °C, 12 h.	Photothermal	14-90.2	9.2 mmol/g/h ,	ACS Appl. Mater. Interfaces 2025, 17, 33846-33856
18	Mg-NC	Epoxide (0.58 mmol) + catalyst (5 mg) + 2 mL DMF + TBAB (0.06 mmol) + 1 bar CO_2 + 300-W Xe lamp (200 mW•cm^{-2}) or heated at 54 °C	photo or thermal catalytic	>99	9.67	CCS Chem. 2024, 6, 3077-3088
19	Co-s ZIS)	20 mg of photocatalyst, 3 mmol of epoxide substrates, 0.1 mmol TBAB, CO2 atmosphere, visible light (λ > 420 nm), 24 h.	Photocatalytic		7.21 mmolg^{-1} h^{-1}	ACS Catal. 2023, 13, 8317−8329

Table S3: Amount of SnOx/ZnSn(OH)$_6$ variation for CO$_2$ cycloaddition reaction.

Entry	Variation from the standard conditions	Time (h)	Yield (%)	Rate (μmol/h)	Activity (μmol/h/g)	AQY *10^5
1	10 mg SnO$_x$/ZnSn(OH)$_6$	12	39.2	98	9800.0	304.70
2	30 mg SnO$_x$/ZnSn(OH)$_6$	12	49.2	123.2	4106.6	383.05
3	50 mg SnO$_x$/ZnSn(OH)$_6$	12	93.0	232.5	4650.0	722.89
4	70 mg SnO$_x$/ZnSn(OH)$_6$	12	94.6	236.6	3380.0	735.63
5	90 mg SnO$_x$/ZnSn(OH)$_6$	12	98.0	245	2722.2	761.75
[a]Standard conditions**:** SnOx/ZnSn(OH)$_6$ Catalyst (50 mg), styrene oxide (3 mmol), PEG 600 (2 mL), 1 atm CO$_2$ balloon, TBAB (0.3 mmol), 8 Watt UV light (254 nm), Room temperature, 12 h. (PEG 600- Polyethylene Glycol 600)						

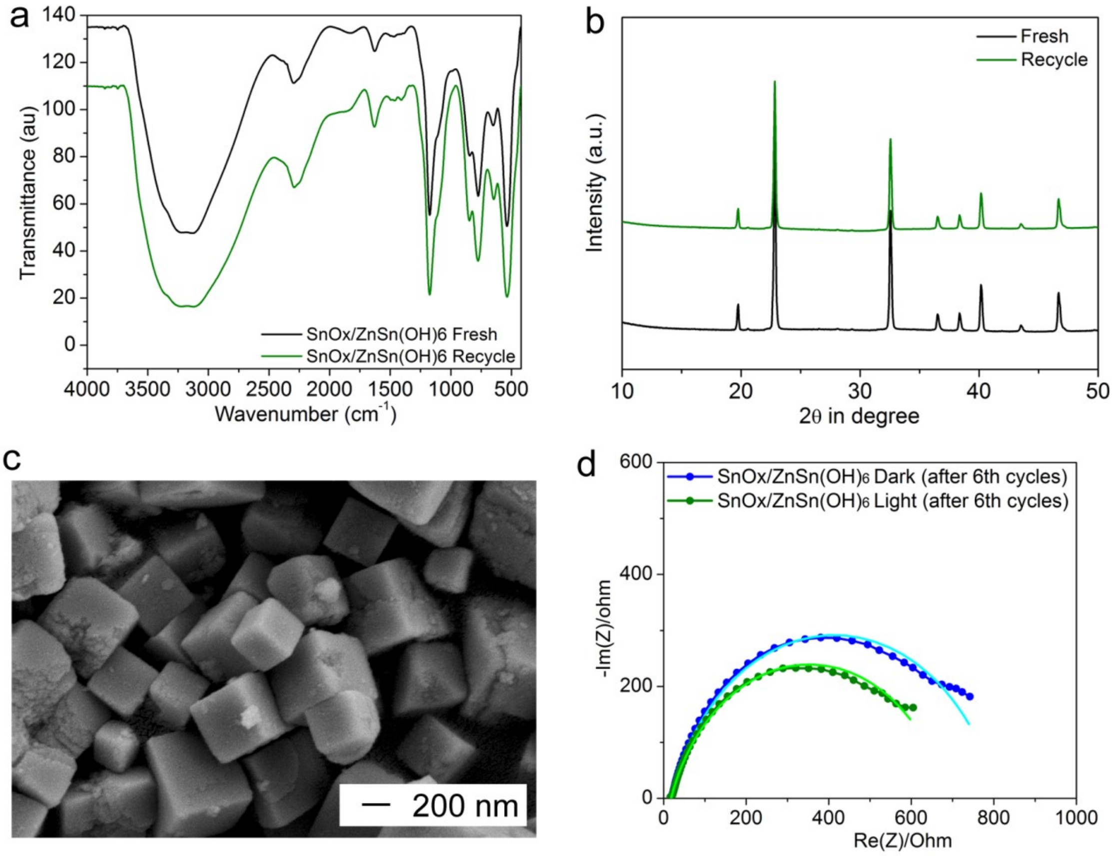


Fig. S18: a) FT IR, b) Powder XRD pattern, c) FE SEM and d) Nyquest plot of SnOx/ZnSn$(OH)_6$ fresh and recycle materials (after 6th cycle)s.

Table S4: Summary of electrochemical properties of SnOx/ZnSn$(OH)_6$ materials after recycle.

	Sample	**R1 (ohm)**	**CPE (F s^(a-1))**	**R2 (Ohm)**
300 W Xe Lamp	SnOx/ZnSn$(OH)_6$ dark (Fresh)	14.11	27.42e-6	746.0
	SnOx/ZnSn$(OH)_6$ light (Fresh)	12.93	31.06e-6	464.1
	SnOx/ZnSn$(OH)_6$ dark (after 6th cycles)	17.89	50.33e-6	790.7
	SnOx/ZnSn$(OH)_6$ light (after 6th cycles)	18.17	58.99e-6	669.6

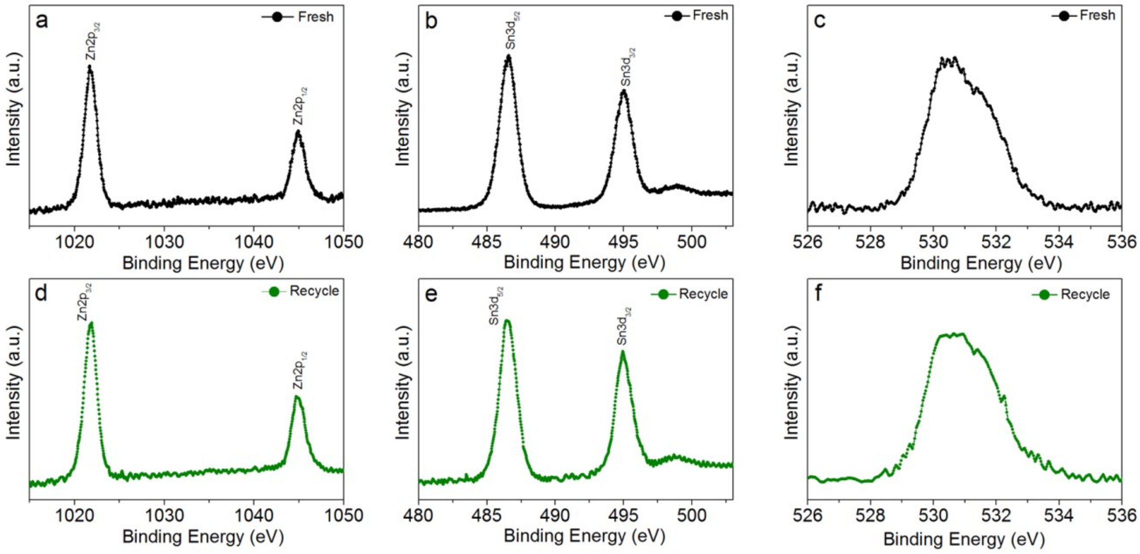


Fig. S19: High resolution XPS spectra of Zn 2p, Sn 3d and O 1s region of a-c) fresh and d-f) recycle SnOx/ZnSn$(OH)_6$ microcubes.

^{1}H and ^{13}C NMR data of Table 2

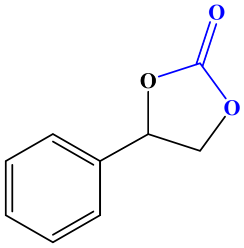
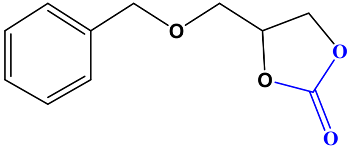
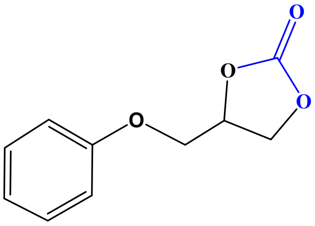
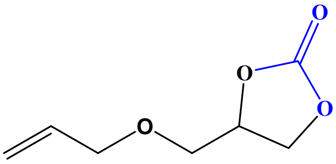
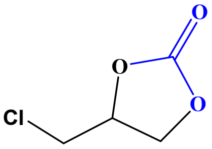
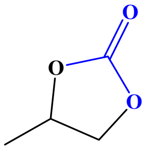
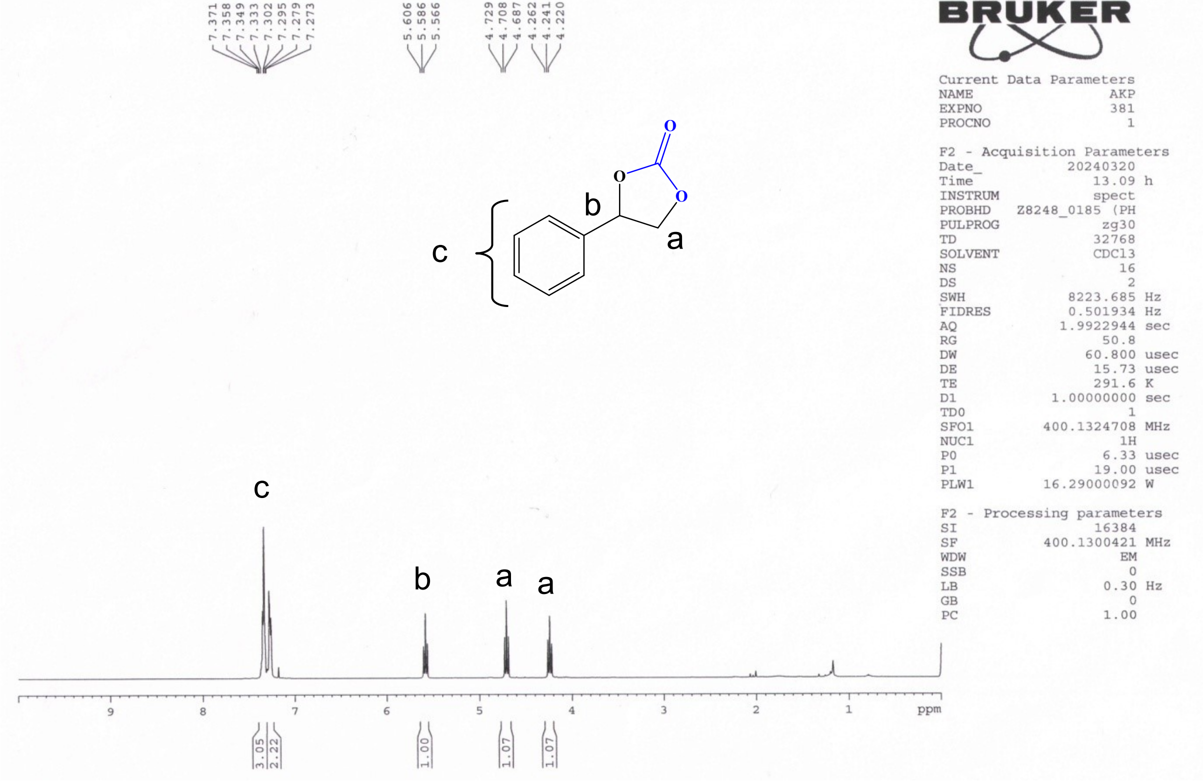
Table 2. Entry 1 Product 1.	
	4-phenyl-1, 3-dioxolan-2-one: ^{1}H NMR (400MHz, CDCl$_3$) δ 7.371 - 7.333 (3H, m, J=6.4 Hz), 7.302 – 7.273 (2H, m, J=6.4 Hz), 5.606 -5.566 (1H, t, J=8.0), 4.729-4.687 (1H, t J=8.4), 4.262-4.220 (1H, t, J=8.4); ^{13}C NMR (400 MHz, CDCl$_3$) δ 154.97, 135.84, 129.73, 129.24, 125.95, 78.06, 71.22.
Table 2. Entry 2 Product 2.	
	4-((benzyloxy)methyl)-1, 3-dioxolan-2-one: ^{1}H NMR (400MHz, CDCl$_3$) δ 7.304 - 7.182 (5H, m, J=5.6 Hz), 4.759-4.705 (1H, m), 4.556-4.470 (2H, q, J= 11.2), 4.417-4.375(1H, t, J= 8.4), 4.319-4.282 (1H, m), 3.650-3.613 (1H, dd, J=11.4, 3.8 Hz,), 3.555-3.518 (1H, dd, J = 11.2, 3.8 Hz); ^{13}C NMR (400MHz, CDCl$_3$) δ 154.98, 137.09, 128.60, 128.11, 127.78, 75.03, 73.71, 68.84, 66.31.
Table 2. Entry 3 Product 3.	
	4-((phenoxy) methyl)-1, 3-dioxolan-2-one: ^{1}H NMR (400MHz, CDCl$_3$) δ 7.263-7.223 (2H, m, J=6.4 Hz), 6.970 – 6.932 (1H, t, J=7.8 Hz), 6.854 – 6.834 (2H, d, J=8.4 Hz), 4.982-4.947 (1H, m), 4.575-4.533 (1H, t, J=8.4 Hz), 4.494-4.458 (1H, m), 4.194-4.156 (1H, dd, J=11.4, 4.4 Hz,), 4.105-4.070 (1H, dd, J = 11.2, 3.4 Hz, 1H); ^{13}C NMR (400MHz, CDCl$_3$) δ 157.65, 154.77, 129.71, 122.04, 114.61, 74.03, 66.87, 66.26.
Table 2. Entry 4 Product 4	
	4-((allyloxy)methyl)-1, 3-dioxolan-2-one: ^{1}H NMR (400MHz, CDCl$_3$) δ 5.854 - 5.757 (1H, m), 5.248 - 5.146 (2H, m), 4.787 - 4.732 (1H, m), 4.460 - 4.418 (1H, t, J=8.4 Hz), 4.354 - 4.318 (1H, m), 4.006 - 3.980 (1H, m), 3.648 - 3.611 (2H, m), 3.572 - 3.544 (2H, m); ^{13}C NMR (400MHz, CDCl$_3$) δ 154.94, 133.66, 117.97, 75.02, 72.62, 68.84, 66.29.
Table 2. Entry 5 Product 5	
	4-(chloromethyl)-1, 3-dioxolan-2-one: ^{1}H NMR (400MHz, CDCl$_3$) δ 4.969-4.912 (1H, m), 4.563 - 4.520 (1H, t, J=8.4 Hz), 4.369 - 4.333 (1H, m), 3.777-3.733 (1H, dd, J = 12.2, 5.4 Hz), 3.695 -3.655 (1H, dd, J =12.2, 3.6 Hz); ^{13}C NMR (400MHz, CDCl$_3$) δ 154.36, 74.39, 66.98, 43.89.
Table 2. Entry 6 Product 6	
	4-methyl-1,3-dioxolan-2-one: ^{1}H NMR (400MHz, CDCl$_3$) δ 4.824 - 4.774 (1H, m), 4.517 - 4.477 (1H, t, J=8.0 Hz), 3.986 - 3.947 (1H, m), 1.440-1.419 (1H, d); ^{13}C NMR (400MHz, CDCl$_3$) δ 155.03, 73.52, 70.64, 19.45.

Table 2. Entry 1 Product 1.

^{1}H NMR (400 MHz, CDCl$_3$)



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PROCNO                1

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TD                32768
SOLVENT           CDCl3
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DS                    2
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FIDRES         0.501934 Hz
AQ            1.9922944 sec
RG                 50.8
DW               60.800 usec
DE                15.73 usec
TE                291.6 K
D1           1.00000000 sec
TD0                   1
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NUC1                 1H
P0                 6.33 usec
P1                19.00 usec
PLW1        16.29000092 W

F2 - Processing parameters
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WDW                  EM
SSB                   0
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GB                    0
PC                 1.00
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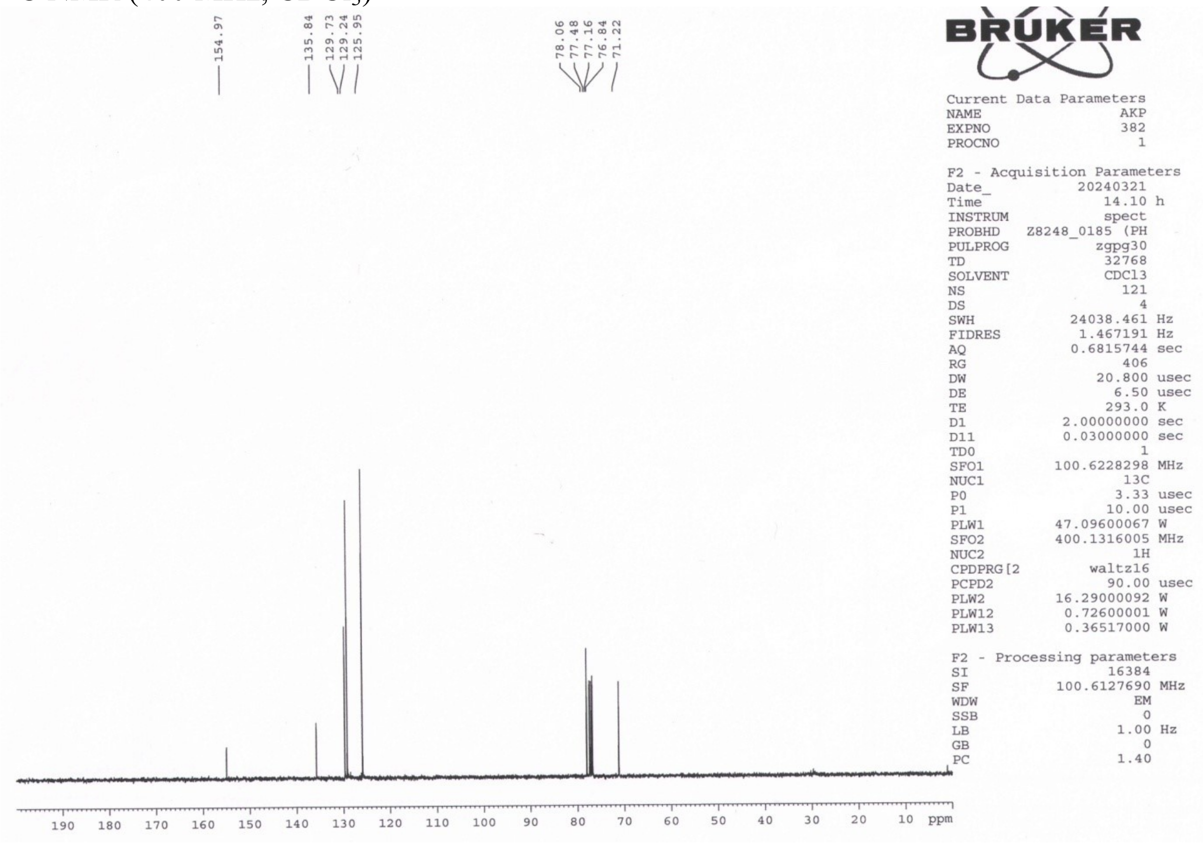
^{13}C NMR (400 MHz, CDCl$_3$)



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EXPNO               382
PROCNO                1

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PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  121
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG                  406
DW               20.800 usec
DE                 6.50 usec
TE                293.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1        47.09600067 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.29000092 W
PLW12        0.72600001 W
PLW13        0.36517000 W

F2 - Processing parameters
SI                16384
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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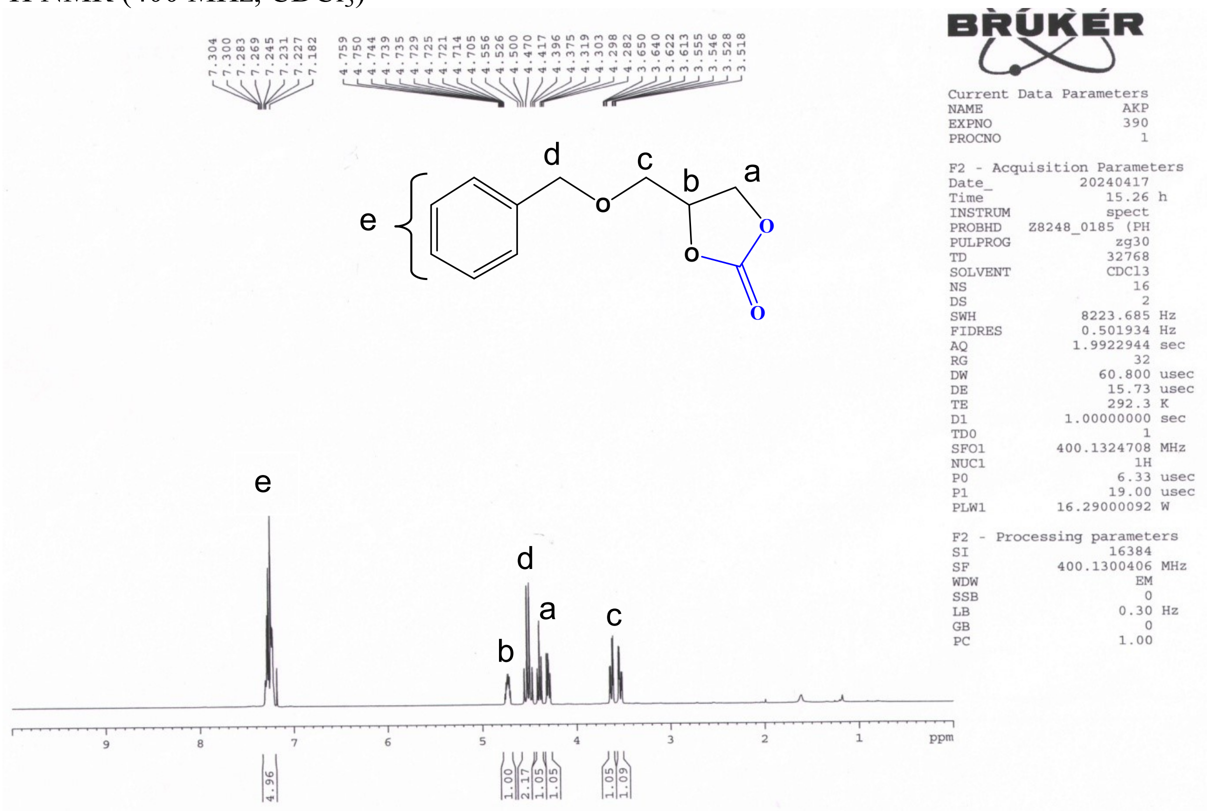
Table 2. Entry 2 Product 2.

^{1}H NMR (400 MHz, $CDCl_3$)



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EXPNO 390
PROCNO 1

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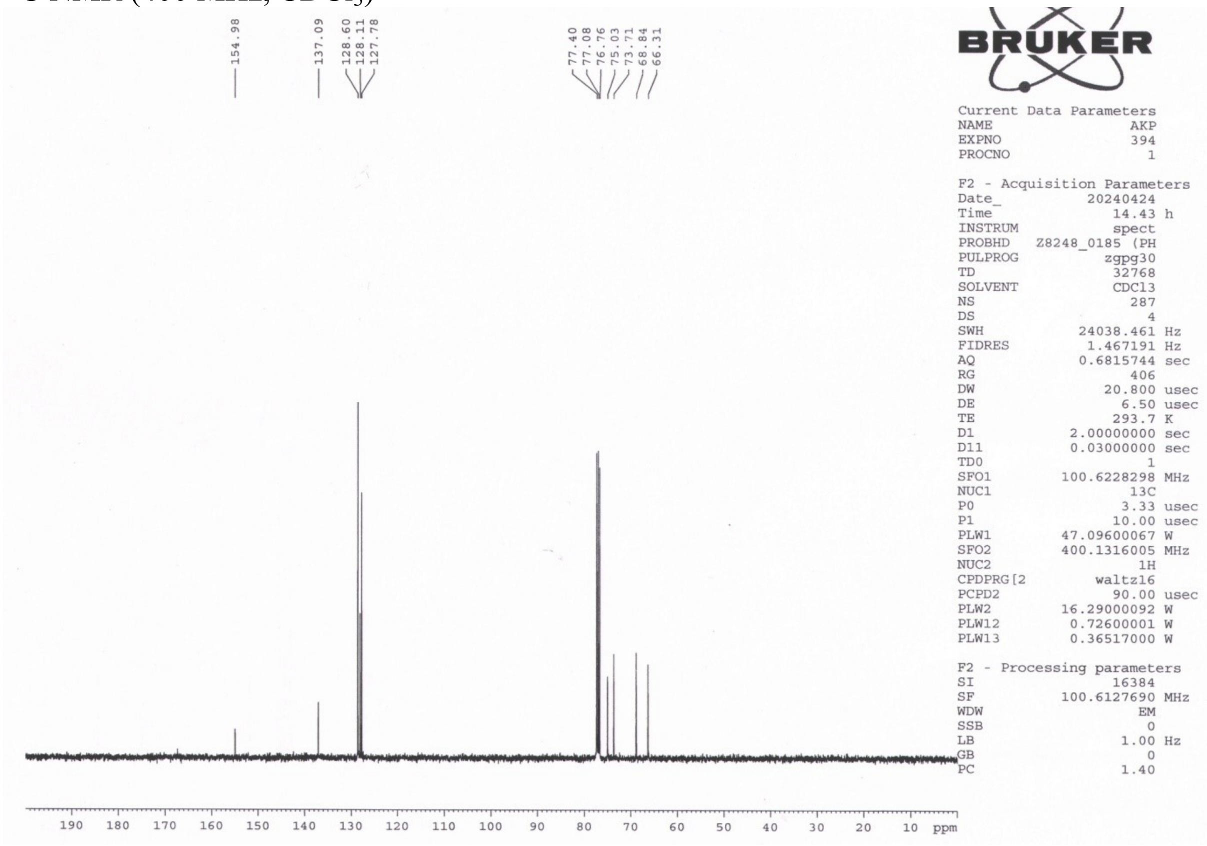
PROBHD Z8248_0185 (PH
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.501934 Hz
AQ 1.9922944 sec
RG 32
DW 60.800 usec
DE 15.73 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P0 6.33 usec
P1 19.00 usec
PLW1 16.29000092 W

F2 - Processing parameters
SI 16384
SF 400.1300406 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR (400 MHz, $CDCl_3$)



Current Data Parameters
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EXPNO 394
PROCNO 1

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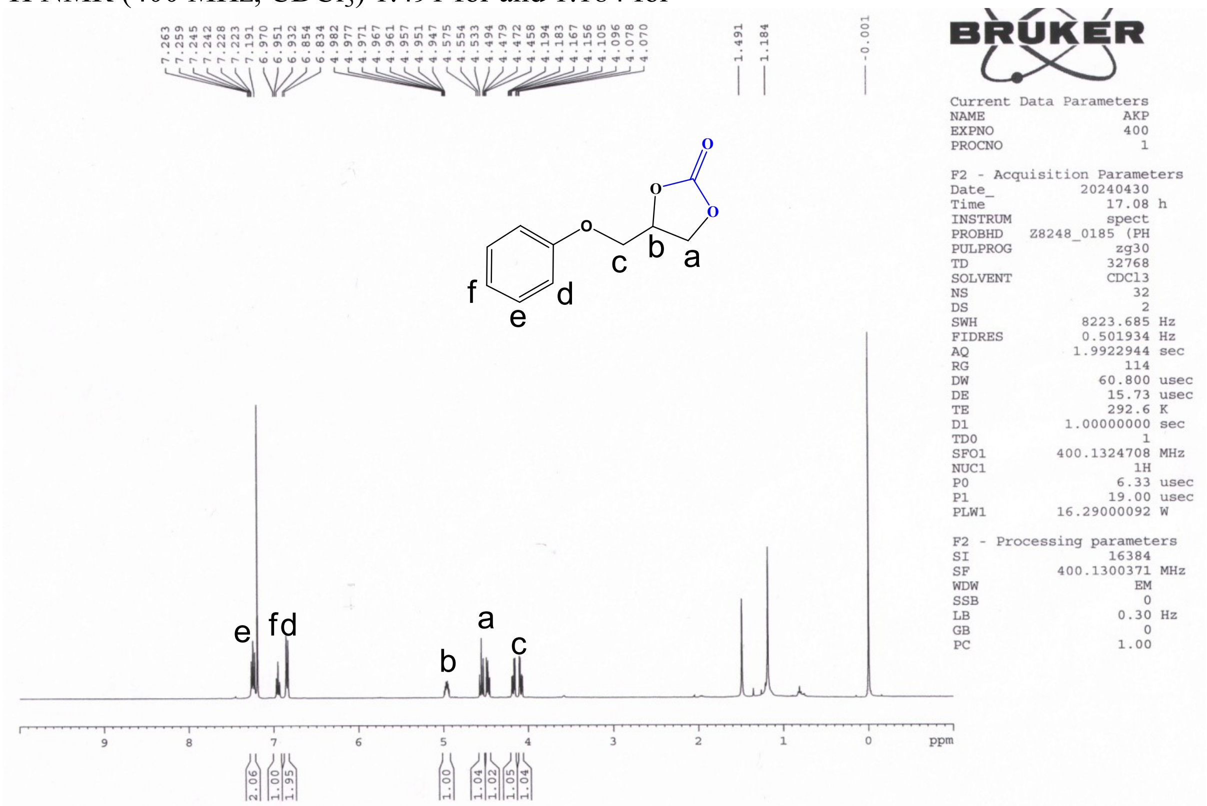
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 287
DS 4
SWH 24038.461 Hz
FIDRES 1.467191 Hz
AQ 0.6815744 sec
RG 406
DW 20.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 47.09600067 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.29000092 W
PLW12 0.72600001 W
PLW13 0.36517000 W

F2 - Processing parameters
SI 16384
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Table 2. Entry 3 Product 3.

^{1}H NMR (400 MHz, CDCl$_3$) 1.491 for and 1.184 for



^{13}C NMR (400 MHz, CDCl$_3$)

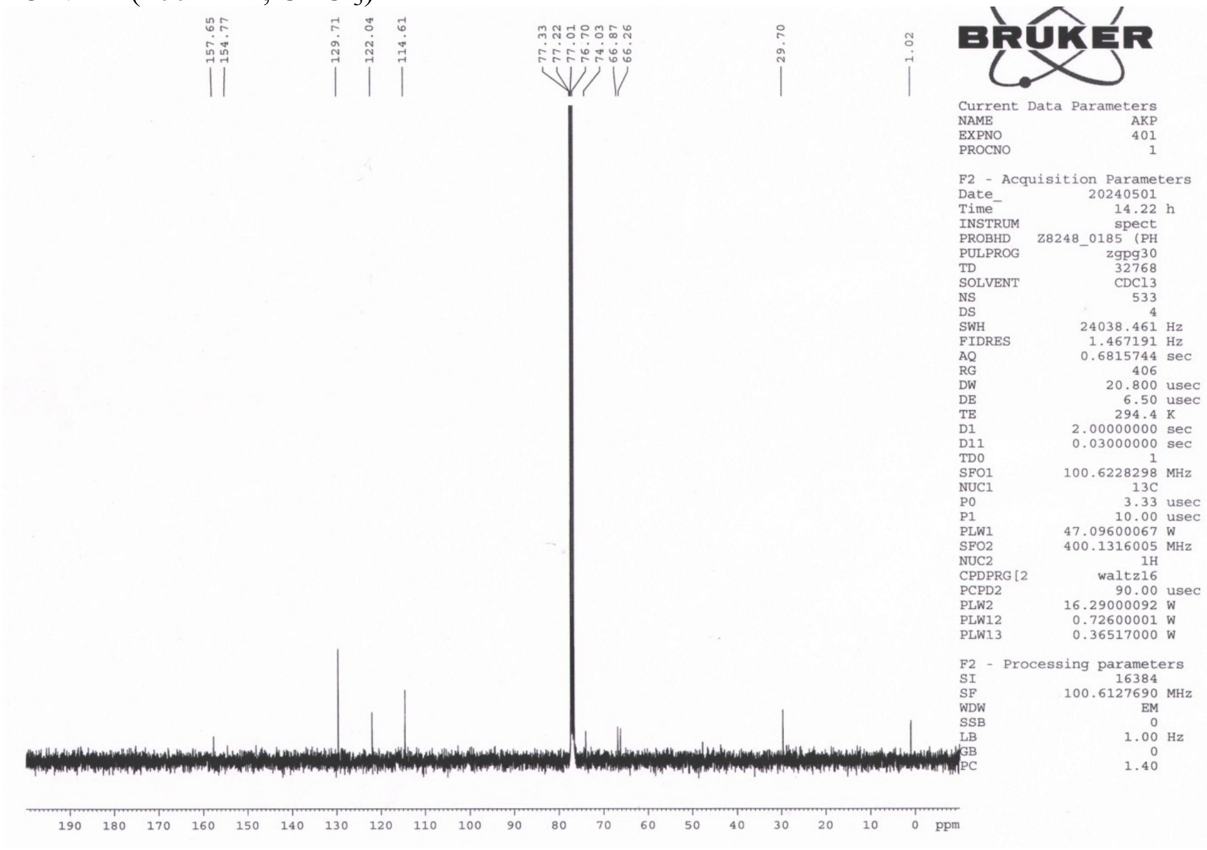
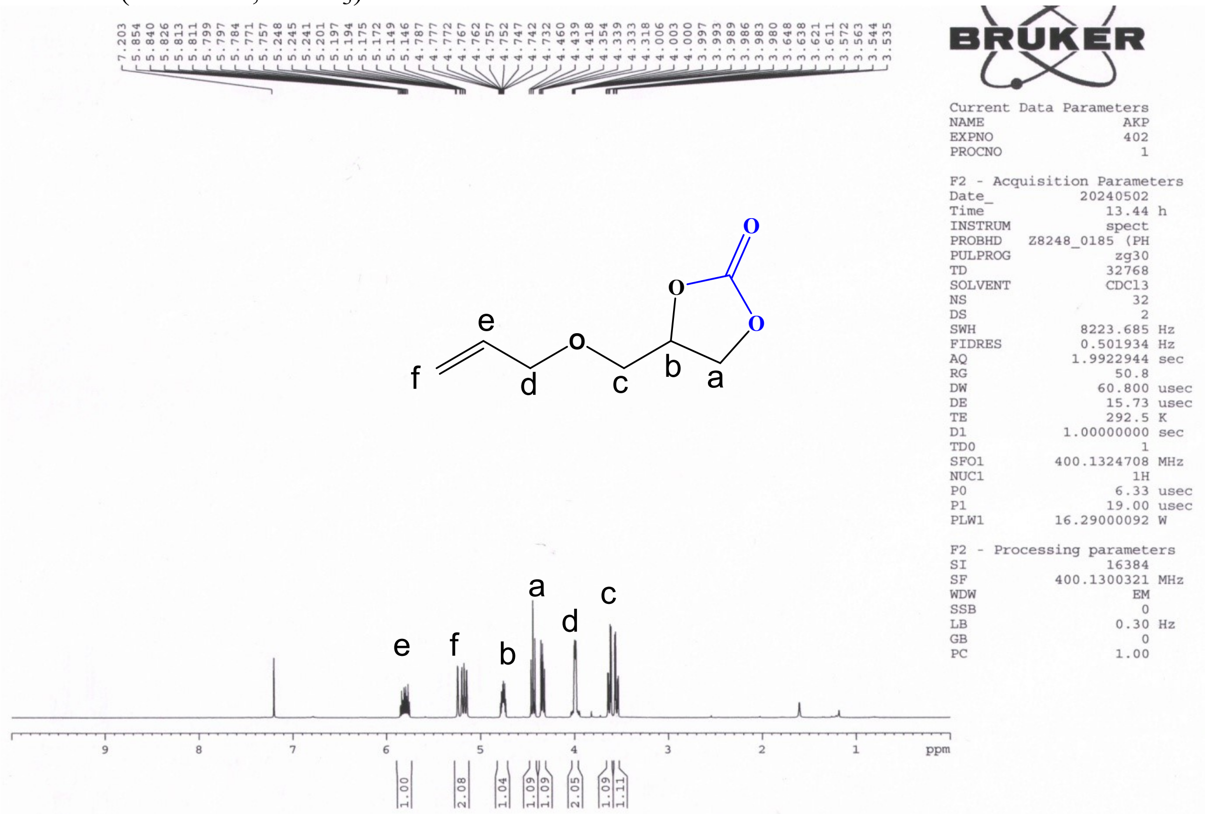


Table 2. Entry 4 Product 4.

^{1}H NMR (400 MHz, $CDCl_3$)



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EXPNO               402
PROCNO                1

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PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            8223.685 Hz
FIDRES         0.501934 Hz
AQ            1.9922944 sec
RG                 50.8
DW               60.800 usec
DE                15.73 usec
TE                292.5 K
D1           1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                 1H
P0                 6.33 usec
P1                19.00 usec
PLW1        16.29000092 W

F2 - Processing parameters
SI                16384
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WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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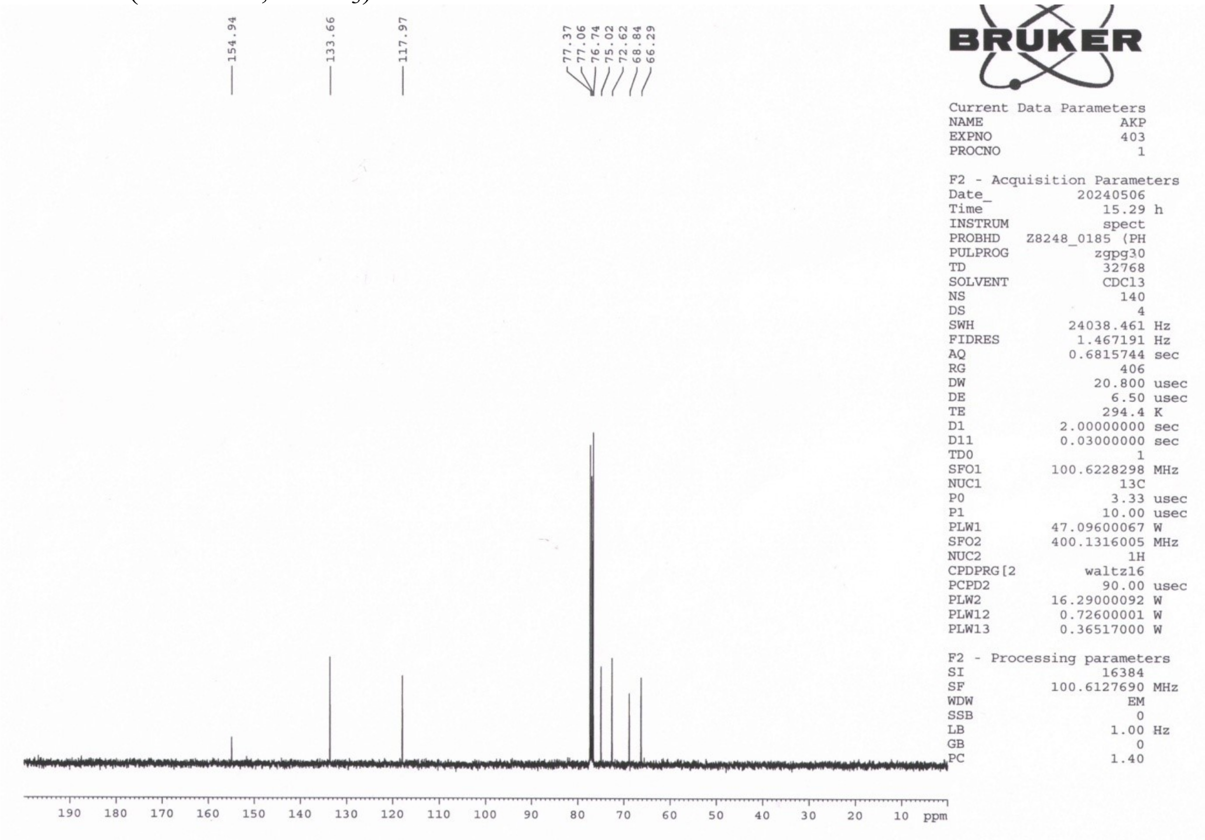
^{13}C NMR (400 MHz, $CDCl_3$)



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EXPNO               403
PROCNO                1

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PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  140
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG                  406
DW               20.800 usec
DE                 6.50 usec
TE                294.4 K
D1           2.00000000 sec
D11          0.03000000 sec
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NUC1                13C
P0                 3.33 usec
P1                10.00 usec
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SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.29000092 W
PLW12        0.72600001 W
PLW13        0.36517000 W

F2 - Processing parameters
SI                16384
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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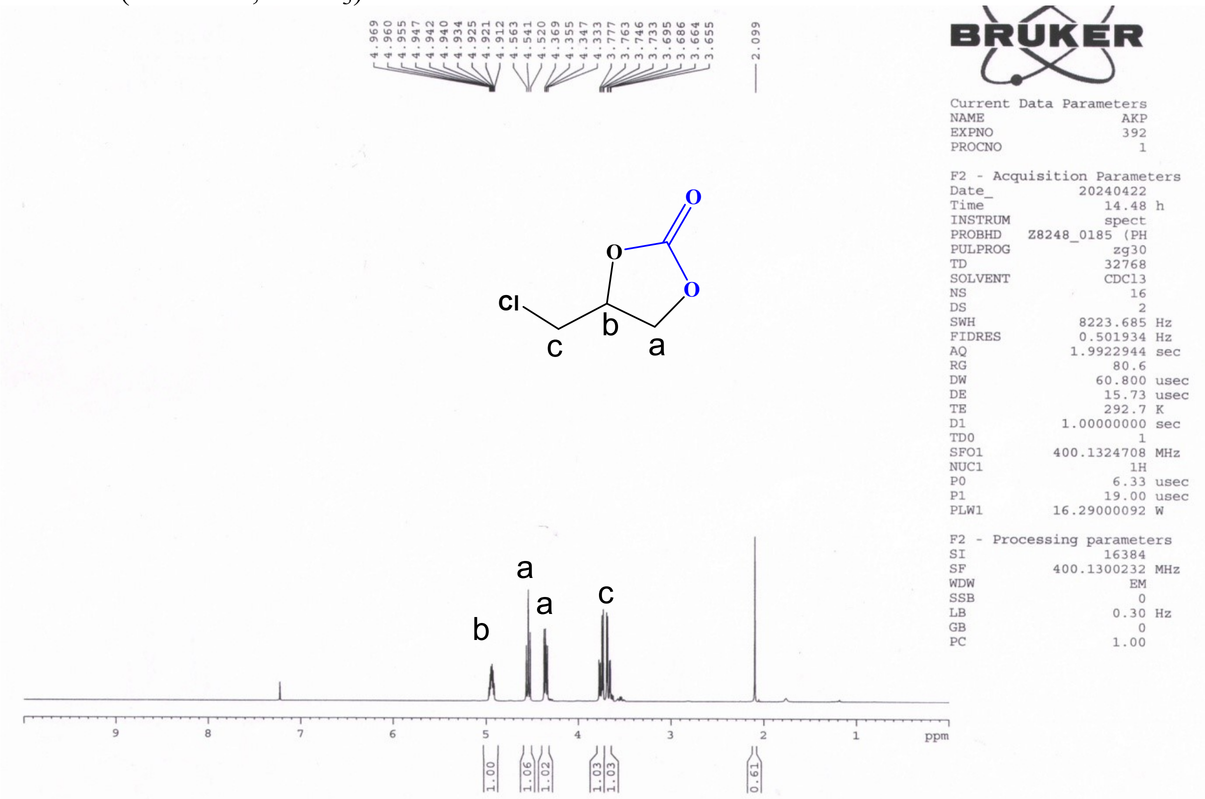
Table 2. Entry 5 Product 5.

^{1}H NMR (400 MHz, CDCl$_3$)



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Current Data Parameters
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EXPNO                 392
PROCNO                  1

F2 - Acquisition Parameters
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INSTRUM             spect
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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.501934 Hz
AQ              1.9922944 sec
RG                   80.6
DW                 60.800 usec
DE                  15.73 usec
TE                  292.7 K
D1             1.00000000 sec
TD0                     1
SFO1          400.1324708 MHz
NUC1                   1H
P0                   6.33 usec
P1                  19.00 usec
PLW1          16.29000092 W

F2 - Processing parameters
SI                  16384
SF            400.1300232 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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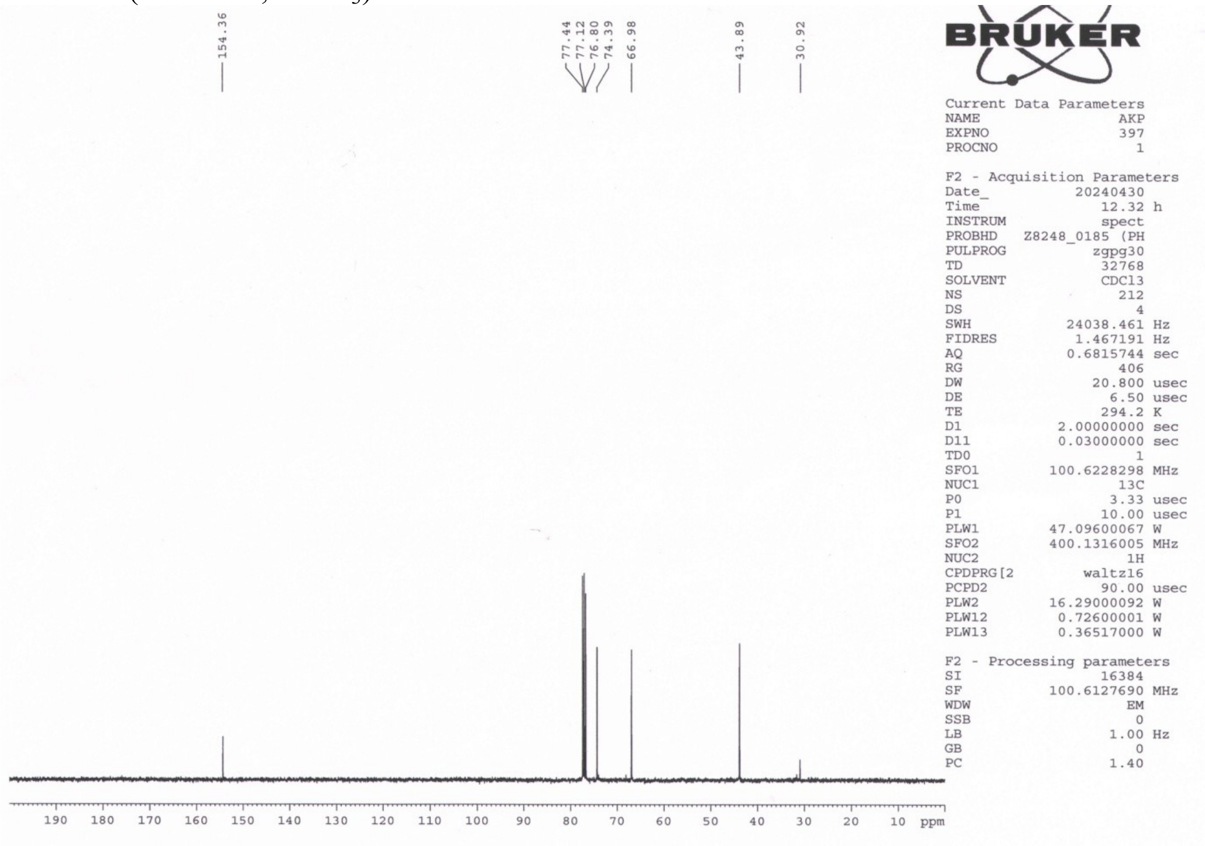
^{13}C NMR (400 MHz, CDCl$_3$)



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EXPNO                 397
PROCNO                  1

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PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    212
DS                      4
SWH             24038.461 Hz
FIDRES           1.467191 Hz
AQ              0.6815744 sec
RG                    406
DW                 20.800 usec
DE                   6.50 usec
TE                  294.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P0                   3.33 usec
P1                  10.00 usec
PLW1          47.09600067 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          16.29000092 W
PLW12          0.72600001 W
PLW13          0.36517000 W

F2 - Processing parameters
SI                  16384
SF            100.6127690 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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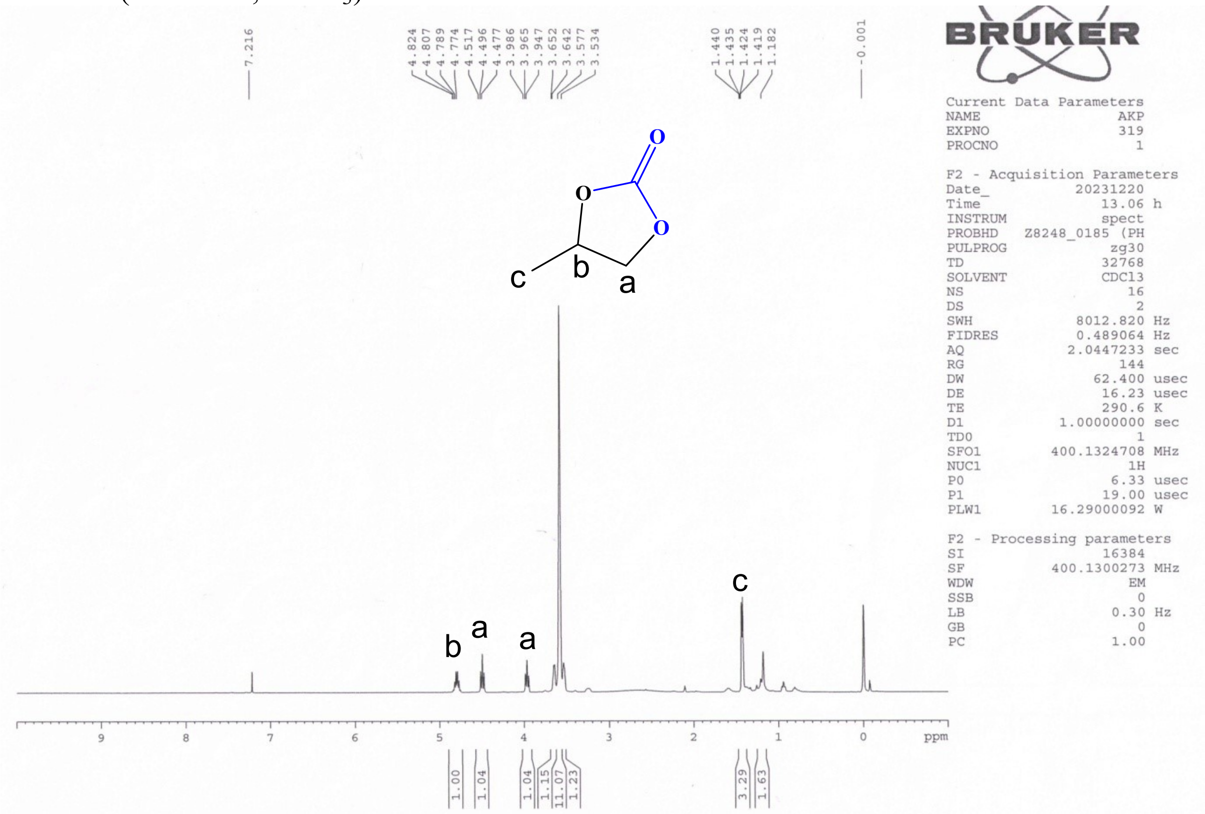
Table 2. Entry 6 Product 6.

^{1}H NMR (400 MHz, CDCl$_3$)



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PROCNO                1

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PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.489064 Hz
AQ            2.0447233 sec
RG                  144
DW               62.400 usec
DE                16.23 usec
TE                290.6 K
D1           1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                 1H
P0                 6.33 usec
P1                19.00 usec
PLW1        16.29000092 W

F2 - Processing parameters
SI                16384
SF          400.1300273 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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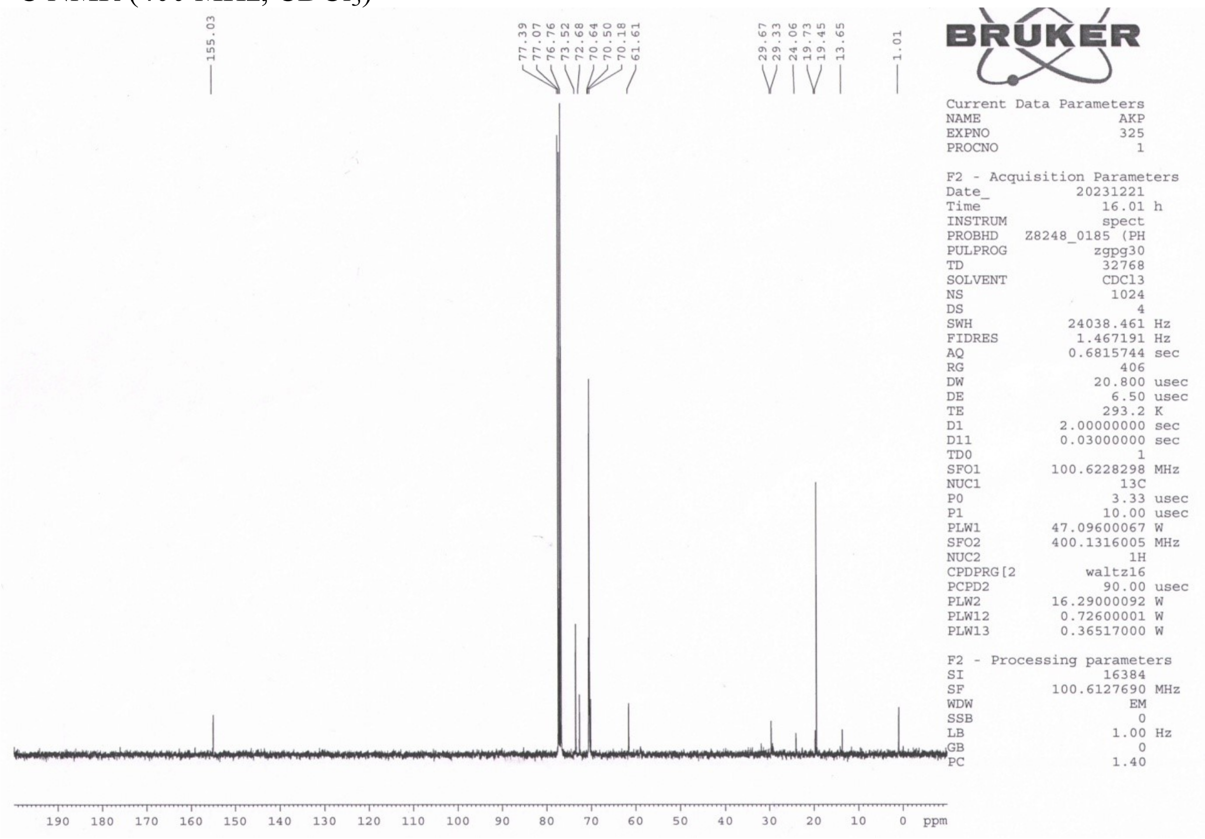
^{13}C NMR (400 MHz, CDCl$_3$)



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Current Data Parameters
NAME                AKP
EXPNO               325
PROCNO                1

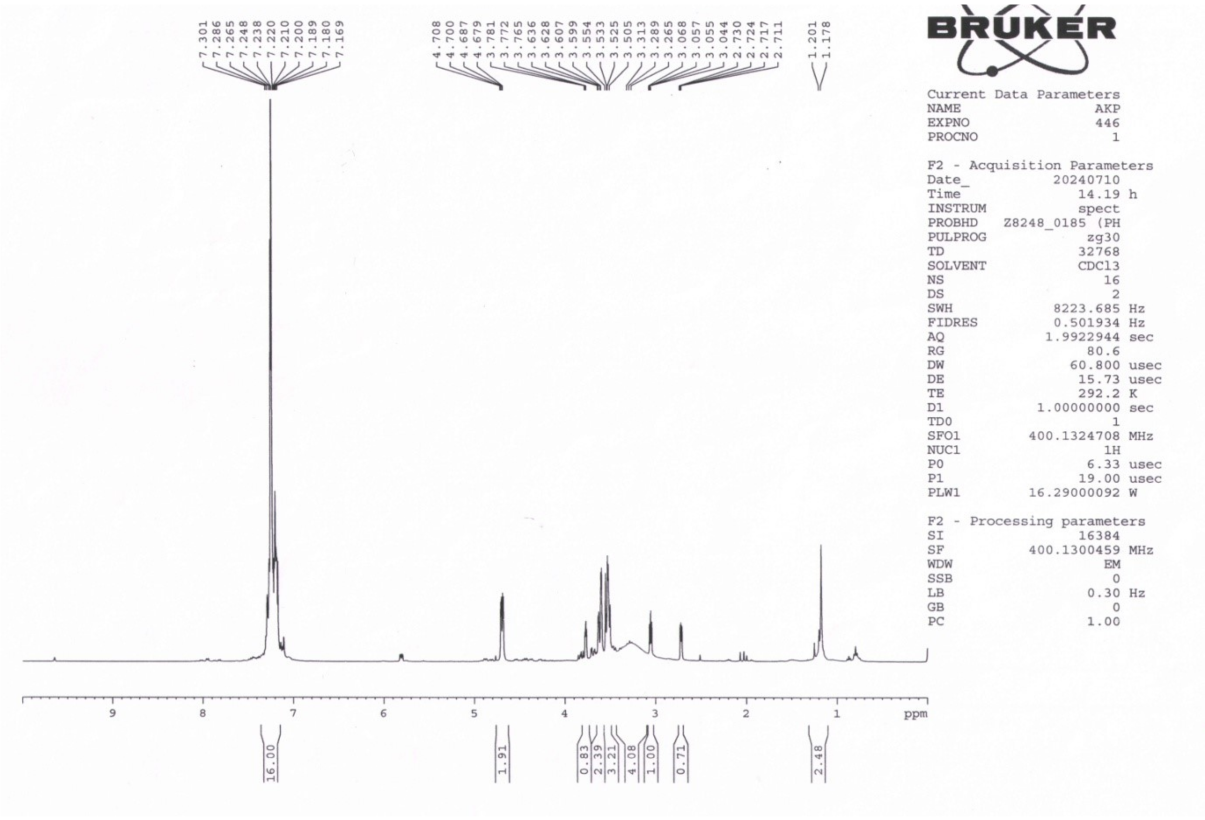
F2 - Acquisition Parameters
Date_          20231221
Time              16.01 h
INSTRUM           spect
PROBHD   Z8248_0185 (PH
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         1.467191 Hz
AQ            0.6815744 sec
RG                  406
DW               20.800 usec
DE                 6.50 usec
TE                293.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1        47.09600067 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        16.29000092 W
PLW12        0.72600001 W
PLW13        0.36517000 W

F2 - Processing parameters
SI                16384
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Table S5: Assessment of quenchers and different co-catalyst for CO_2 cycloaddition reaction.

Entry	Variation from the standard conditions	Time (h)	Yield (%)	Rate (µmol/h)	Activity (µmol/h/g)
1	$AgNO_3$ (e quencher)	12	0.0	0.0	0.0
2	KI (h quencher)	12	99.0	247.5	4950.0
3	$Na_2C_2O_4$ (h quencher)	12	Trace	0.0	0.0
4	DMPO (Radical quencher)	12	35.0	87.5	1750.0
5	Catalyst+KI (Without TBAB)	12	99.0	247.5	4950.0
6	Catalyst+KBr (Without TBAB)	12	37.0	92.5	1850.0
7	Catalyst+TBAI (Without TBAB)	12	87.0	217.5	4350.0
8	KI Only (Without Catalyst & TBAB)	12	50.0	125.0	2500.0

[a]Standard conditions: $SnO_x/ZnSn(OH)_6$ Catalyst (50 mg), styrene oxide (3 mmol), PEG 600 (2 mL), 1 atm CO_2 balloon, TBAB (0.3 mmol), 8 Watt UV light (254 nm), Room temperature, 12 h. (PEG 600- Polyethylene Glycol 600)



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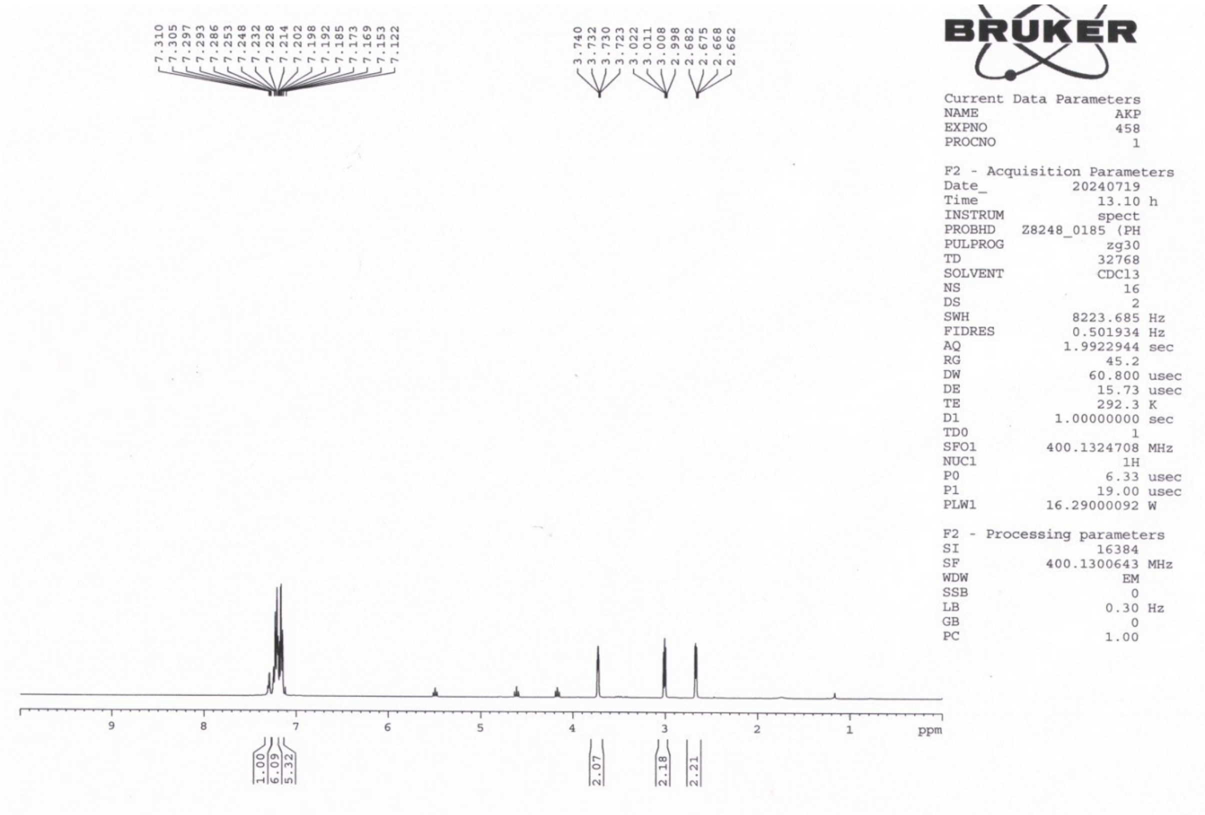
Current Data Parameters
NAME                 AKP
EXPNO                446
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240710
Time               14.19 h
INSTRUM            spect
PROBHD   Z8248_0185 (PH
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.501934 Hz
AQ             1.9922944 sec
RG                  80.6
DW                60.800 usec
DE                 15.73 usec
TE                 292.2 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                  6.33 usec
P1                 19.00 usec
PLW1         16.29000092 W

F2 - Processing parameters
SI                 16384
SF           400.1300459 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Fig. S20: Standard reaction was carried out in presence of $AgNO_3$

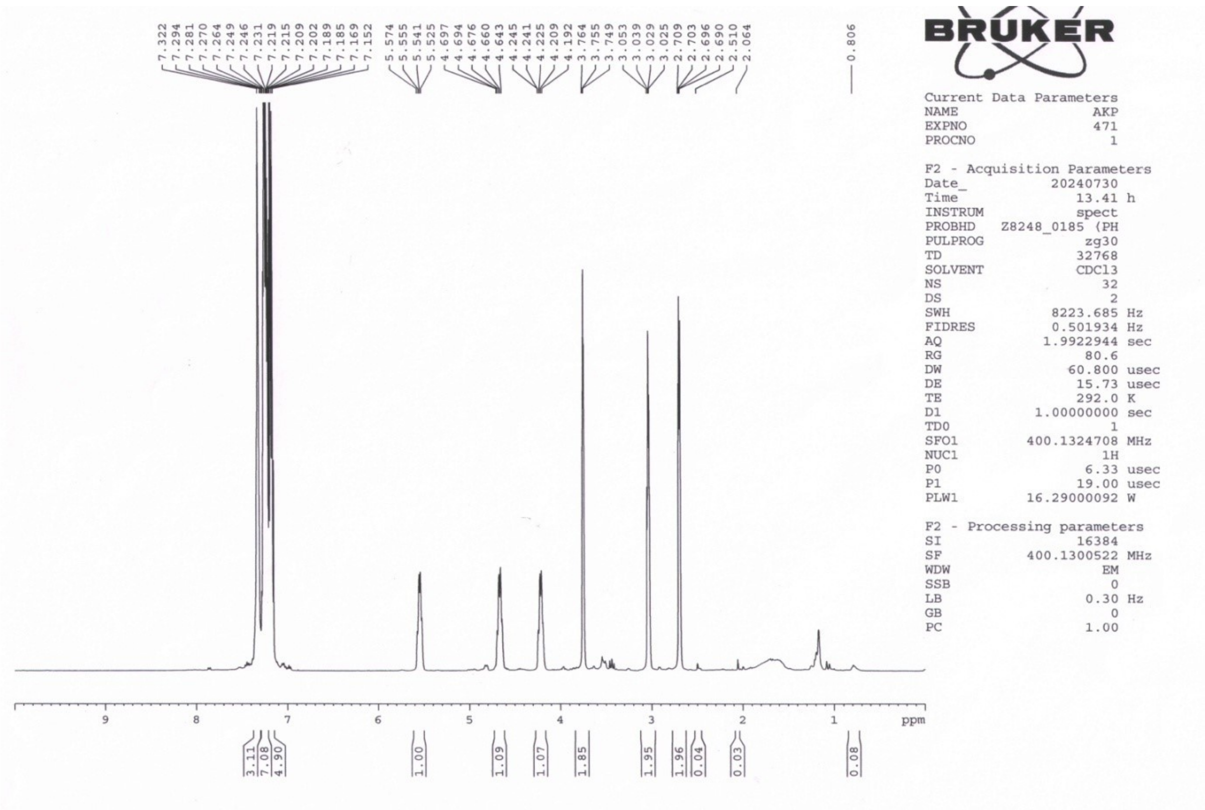


```
Current Data Parameters
NAME                 AKP
EXPNO                458
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240719
Time               13.10 h
INSTRUM            spect
PROBHD   Z8248_0185 (PH
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.501934 Hz
AQ             1.9922944 sec
RG                  45.2
DW                60.800 usec
DE                 15.73 usec
TE                 292.3 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                  6.33 usec
P1                 19.00 usec
PLW1         16.29000092 W

F2 - Processing parameters
SI                 16384
SF           400.1300643 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Fig. S21: Standard reaction was carried out in presence of $Na_2C_2O_4$.



```
Current Data Parameters
NAME                AKP
EXPNO               471
PROCNO                1

F2 - Acquisition Parameters
Date_          20240730
Time              13.41 h
INSTRUM           spect
PROBHD   Z8248_0185 (PH
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            8223.685 Hz
FIDRES         0.501934 Hz
AQ            1.9922944 sec
RG                 80.6
DW               60.800 usec
DE                15.73 usec
TE                292.0 K
D1           1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                 1H
P0                 6.33 usec
P1                19.00 usec
PLW1        16.29000092 W

F2 - Processing parameters
SI                16384
SF          400.1300522 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

Fig. S22: Standard reaction was carried out in presence of DMPO.

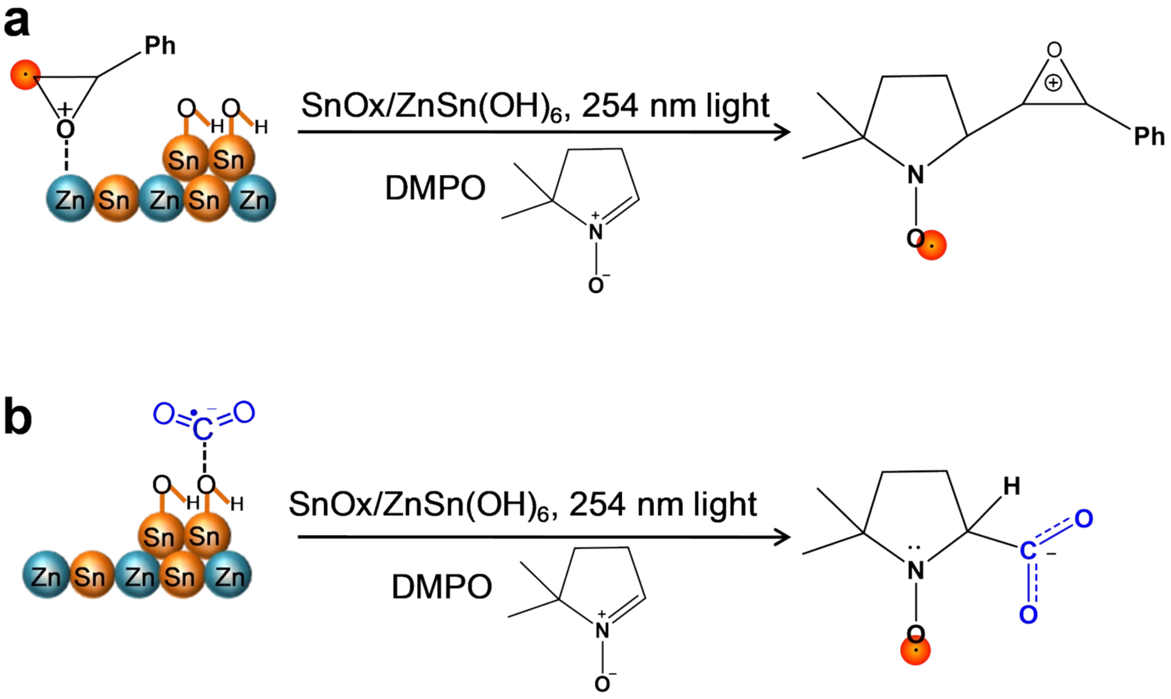
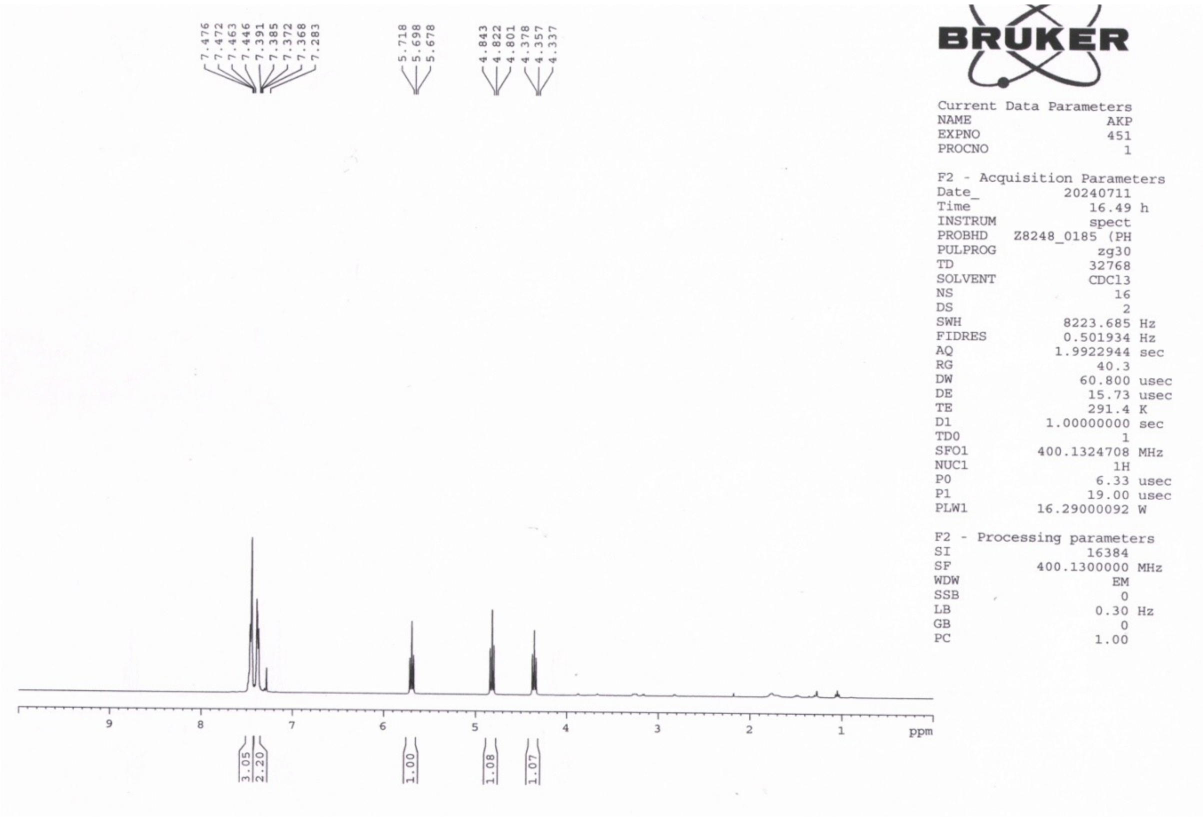


Fig. S23: Schematic diagram of a) styrene oxide radical[3] and b) CO_2 radical[4] intermediates trapped by DMPO.

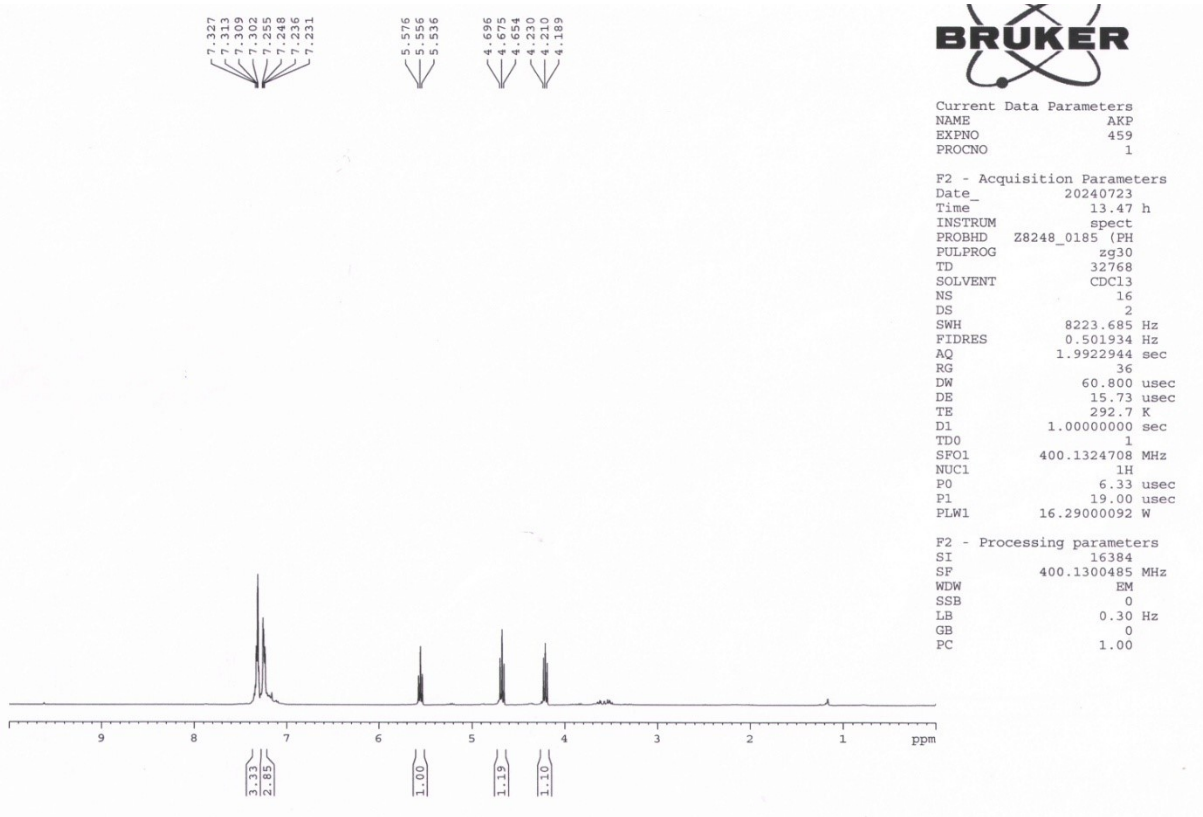


```
Current Data Parameters
NAME               AKP
EXPNO              451
PROCNO               1

F2 - Acquisition Parameters
Date_         20240711
Time             16.49 h
INSTRUM          spect
PROBHD   Z8248_0185 (PH
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8223.685 Hz
FIDRES        0.501934 Hz
AQ           1.9922944 sec
RG                40.3
DW              60.800 usec
DE               15.73 usec
TE               291.4 K
D1          1.00000000 sec
TD0                  1
SFO1       400.1324708 MHz
NUC1                1H
P0                6.33 usec
P1               19.00 usec
PLW1       16.29000092 W

F2 - Processing parameters
SI               16384
SF         400.1300000 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

Fig. S24: Standard reaction was carried out in presence of TBAB and KI.

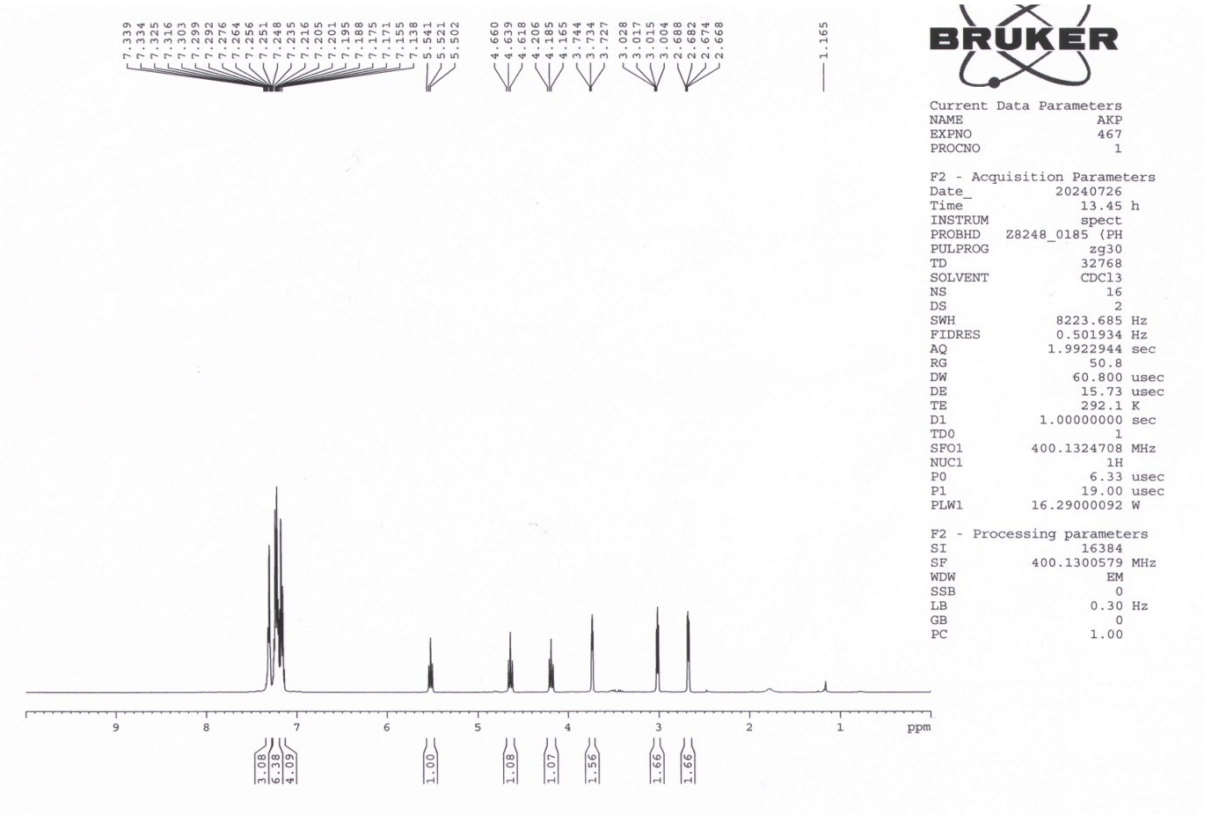


```
Current Data Parameters
NAME               AKP
EXPNO              459
PROCNO               1

F2 - Acquisition Parameters
Date_         20240723
Time             13.47 h
INSTRUM          spect
PROBHD   Z8248_0185 (PH
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8223.685 Hz
FIDRES        0.501934 Hz
AQ           1.9922944 sec
RG                  36
DW              60.800 usec
DE               15.73 usec
TE               292.7 K
D1          1.00000000 sec
TD0                  1
SFO1       400.1324708 MHz
NUC1                1H
P0                6.33 usec
P1               19.00 usec
PLW1       16.29000092 W

F2 - Processing parameters
SI               16384
SF         400.1300485 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

Fig. S25: The reaction was carried out with KI instead of TBAB.

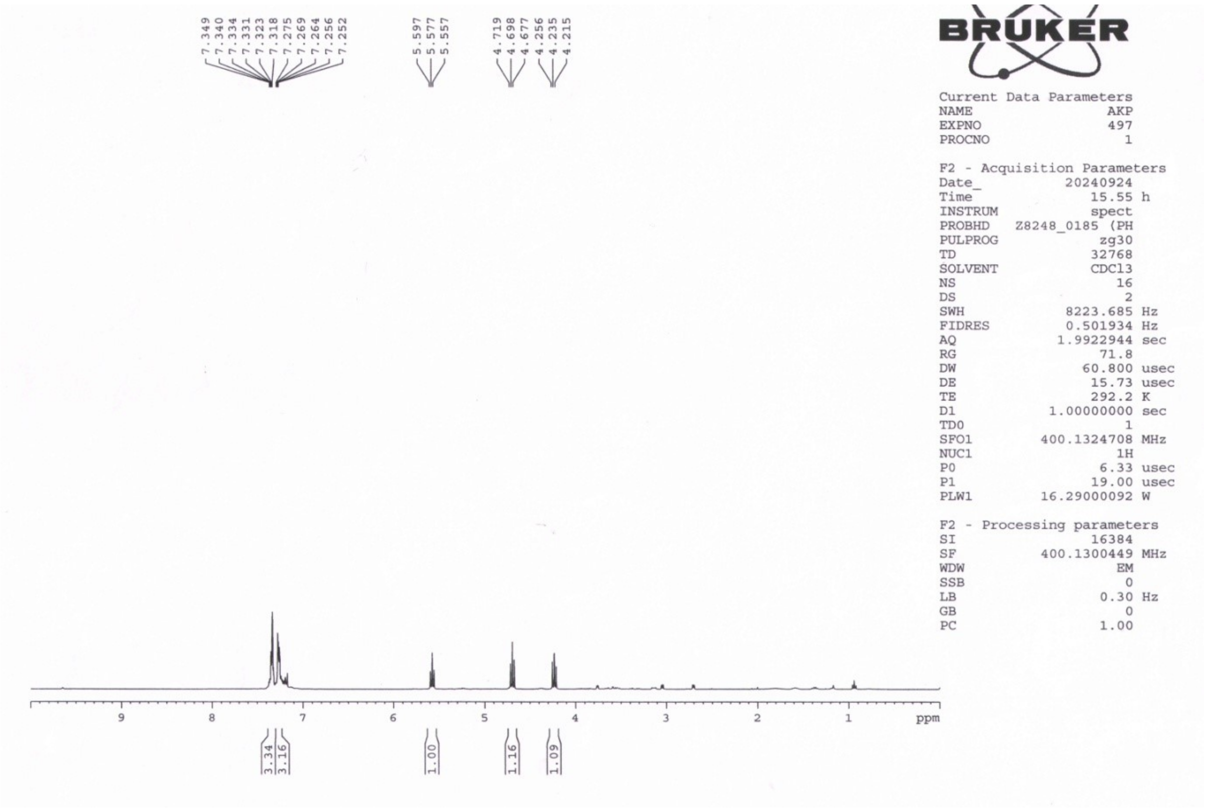


```
Current Data Parameters
NAME                  AKP
EXPNO                 467
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240726
Time                13.45 h
INSTRUM             spect
PROBHD   Z8248_0185 (PH
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.501934 Hz
AQ              1.9922944 sec
RG                   50.8
DW                 60.800 usec
DE                  15.73 usec
TE                  292.1 K
D1             1.00000000 sec
TD0                     1
SFO1          400.1324708 MHz
NUC1                   1H
P0                   6.33 usec
P1                  19.00 usec
PLW1          16.29000092 W

F2 - Processing parameters
SI                  16384
SF            400.1300579 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

Fig. S26: The reaction was carried out with KBr instead of TBAB.

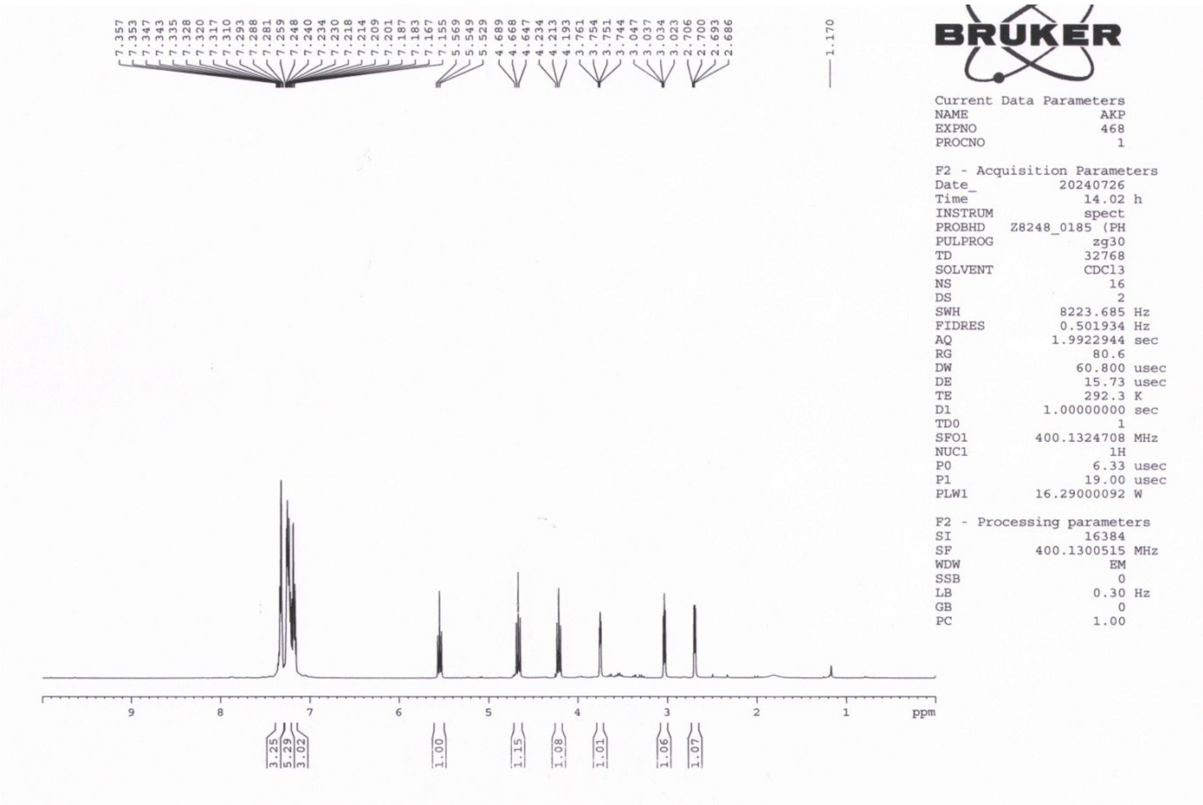


```
Current Data Parameters
NAME                  AKP
EXPNO                 497
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240924
Time                15.55 h
INSTRUM             spect
PROBHD   Z8248_0185 (PH
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.501934 Hz
AQ              1.9922944 sec
RG                   71.8
DW                 60.800 usec
DE                  15.73 usec
TE                  292.2 K
D1             1.00000000 sec
TD0                     1
SFO1          400.1324708 MHz
NUC1                   1H
P0                   6.33 usec
P1                  19.00 usec
PLW1          16.29000092 W

F2 - Processing parameters
SI                  16384
SF            400.1300449 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

Fig. S27: The reaction was carried out with TBAI instead of TBAB.



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Current Data Parameters
NAME                AKP
EXPNO               468
PROCNO                1

F2 - Acquisition Parameters
Date_          20240726
Time              14.02 h
INSTRUM           spect
PROBHD   Z8248_0185 (PH
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.501934 Hz
AQ            1.9922944 sec
RG                 80.6
DW               60.800 usec
DE                15.73 usec
TE                292.3 K
D1           1.00000000 sec
TD0                   1
SFO1        400.1324708 MHz
NUC1                 1H
P0                 6.33 usec
P1                19.00 usec
PLW1        16.29000092 W

F2 - Processing parameters
SI                16384
SF          400.1300515 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

Fig. S28: The reaction was carried out with only KI instead of $SnOx/ZnSn(OH)_6$ photocatalyst and TBAB.

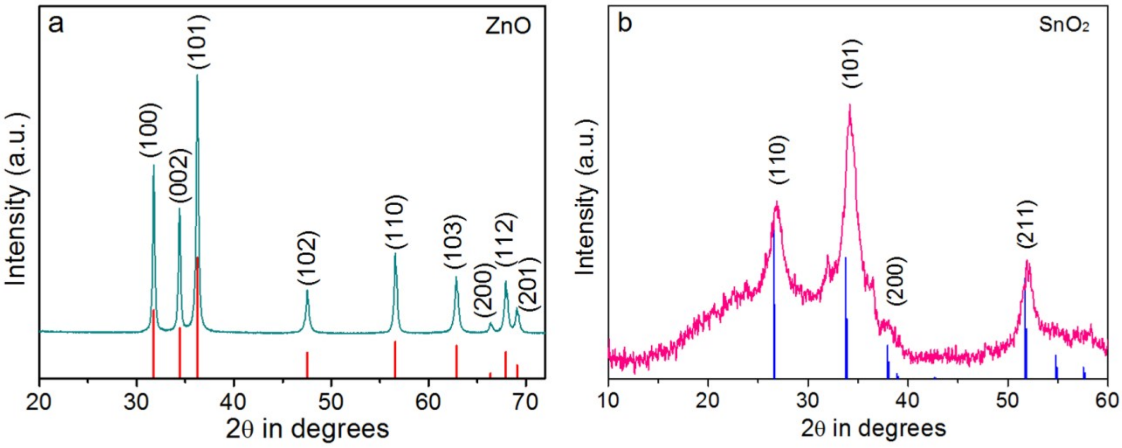

Fig. S29: Powder XRD analysis of a) ZnO and b) SnO_2.

References

1. *J. Solid State Chem*., **2014**, 215, 135-142.

2. *Mater. Chem. Phys*. **2007**, 101, 433-440.

3. *Angew. Chem. Int. Ed*. **2024**, 63, e202404911

4. *J. Phys. Chem. A* **2006**, 110, 49, 13253–13258