Carbonate encapsulation from dissolved atmospheric CO_2 into a polyoxovanadate capsule ^{†‡}

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Section S1. FESEM-EDX analysis of compound 1 reaction mixture solution

a) FESEM-EDX analysis and elemental mapping of reaction mixture solution of compound 1 collected after one day



Element	Weight%	Atomic%
ОК	32.64	65.99
Na K	2.67	3.76
S K	0.94	0.95
VK	40.49	25.71
Bi M	23.25	3.60
Totals	100.00	



Fig. S1. FESEM-EDX analysis of reaction mixture solution of compound 1 collected after one day.





O Ka1

Fig. S2. Elemental mapping of reaction mixture solution of compound 1 collected after one day.



age



Quantitative results



Element	Weight%	Atomic%
NK	4.86	10.07
ОК	29.13	52.81
Na K	5.22	6.59
SК	2.48	2.25
VK	46.89	26.70
Bi M	11.42	1.59
Totals	100.00	

Fig. S3. FESEM-EDX analysis of reaction mixture solution of compound 1 collected after four days.



Section S2. Details of bond distances and angles for compounds 1 and 2

S Ka1

Table S1. Bond distances (Å) and angles (°) for compound 1						
V(2)-O(4)	1.6236(18)	O(3)-V(2)-V(1)	39.48(5)			
V(2)-O(5)#1	1.9067(18)	O(5)-V(2)-V(1)	110.54(6)			
V(2)-O(3)	1.9280(17)	O(8)#1-V(2)-V(1)	42.91(5)			
V(2)-O(5)	1.9287(18)	V(3)-V(2)-V(1)	74.706(15)			
V(2)-O(8)#1	1.9952(18)	V(3)#1-V(2)-V(1)	72.696(15)			
V(2)-V(3)	2.9530(6)	O(6)-V(3)-O(7)	102.61(10)			
V(2)-V(3)#1	2.9550(6)	O(6)-V(3)-O(3)	106.26(9)			
V(2)-V(1)	3.0002(4)	O(7)-V(3)-O(3)	93.21(9)			
V(3)-O(6)	1.6290(19)	O(6)-V(3)-O(8)	105.19(9)			
V(3)-O(7)	1.8318(11)	O(7)-V(3)-O(8)	92.99(10)			
V(3)-O(3)	1.9206(17)	O(3)-V(3)-O(8)	145.71(8)			
V(3)-O(8)	1.9469(19)	O(6)-V(3)-O(5)	99.96(9)			
V(3)-O(5)	1.9665(19)	O(7)-V(3)-O(5)	157.43(9)			
V(1)-O(2)	1.637(3)	O(3)-V(3)-O(5)	80.01(7)			
V(1)-O(3)#2	1.9464(17)	O(8)-V(3)-O(5)	81.44(7)			
V(1)-O(3)	1.9465(17)	O(6)-V(3)-V(2)	104.14(7)			
V(1)-O(8)#1	2.0527(18)	O(7)-V(3)-V(2)	131.05(8)			
V(1)-O(8)#3	2.0527(18)	O(3)-V(3)-V(2)	39.98(5)			
V(1)-O(1)	2.293(3)	O(8)-V(3)-V(2)	117.96(6)			
O(1)-C(1)	1.275(3)	O(5)-V(3)-V(2)	40.23(5)			
N(1)-N(1)#8	1.446(5)	O(6)-V(3)-V(2)#9	109.40(7)			
		O(7)-V(3)-V(2)#9	129.72(8)			
O(4)-V(2)-O(5)#1	109.53(9)	O(3)-V(3)-V(2)#9	112.92(6)			
O(4)-V(2)-O(3)	113.15(9)	O(8)-V(3)-V(2)#9	42.07(5)			
O(5)#1-V(2)-O(3)	137.08(8)	O(5)-V(3)-V(2)#9	39.53(5)			
O(4)-V(2)-O(5)	105.90(9)	V(2)-V(3)-V(2)#9	76.814(19)			
O(5)#1-V(2)-O(5)	92.06(11)	O(2)-V(1)-O(3)#2	103.35(8)			
O(3)-V(2)-O(5)	80.78(8)	O(2)-V(1)-O(3)	103.35(8)			
O(4)-V(2)-O(8)#1	107.87(9)	O(3)#2-V(1)-O(3)	92.18(10)			
O(5)#1-V(2)-O(8)#1	81.69(8)	O(2)-V(1)-O(8)#1	99.73(8)			
O(3)-V(2)-O(8)#1	81.29(7)	O(3)#2-V(1)-O(8)#1	156.71(8)			
O(5)-V(2)-O(8)#1	145.81(8)	O(3)-V(1)-O(8)#1	79.40(7)			
O(4)-V(2)-V(3)	112.94(7)	O(2)-V(1)-O(8)#3	99.73(8)			
O(5)#1-V(2)-V(3)	123.15(6)	O(3)#2-V(1)-O(8)#3	79.41(7)			
O(3)-V(2)-V(3)	39.80(5)	O(3)-V(1)-O(8)#3	156.71(8)			
O(5)-V(2)-V(3)	41.18(6)	O(8)#1-V(1)-O(8)#3	99.83(10)			
O(8)#1-V(2)-V(3)	117.39(5)	O(2)-V(1)-O(1)	165.98(13)			
O(4)-V(2)-V(3)#1	117.92(7)	O(3)#2-V(1)-O(1)	86.24(8)			
O(5)#1-V(2)-V(3)#1	41.03(6)	O(3)-V(1)-O(1)	86.24(8)			
O(3)-V(2)-V(3)#1	110.55(5)	O(8)#1-V(1)-O(1)	71.67(7)			
O(5)-V(2)-V(3)#1	122.91(6)	O(8)#3-V(1)-O(1)	71.67(7)			
O(8)#1-V(2)-V(3)#1	40.83(5)		. /			
V(3)-V(2)-V(3)#1	128.88(2)	V(3)#2-O(7)-V(3)	127.12(14)			
O(4)-V(2)-V(1)	124.95(7)	V(3)-O(8)-V(2)#9	97.10(8)			
O(5)#1-V(2)-V(1)	108.87(6)	V(3)-O(8)-V(1)#9	123.88(9)			

Га	ble S	<u>51.</u>	Bond	distances	(Å)) and	angl	les (°	')	for compound	11	
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V(2)#9-O(8)-V(1)#9	95.65(8)	V(2)#9-O(5)-V(3)	99.44(8)
C(1)-O(1)-V(1)	129.80(19)	V(2)-O(5)-V(3)	98.59(8)
V(3)-O(3)-V(2)	100.22(8)	O(1)#1-C(1)-O(1)	120.000(1)
V(3)-O(3)-V(1)	138.15(9)	O(1)#1-C(1)-O(1)#9	120.002(1)
V(2)-O(3)-V(1)	101.49(8)	O(1)-C(1)-O(1)#9	120.0
V(2)#9-O(5)-V(2)	146.26(11)		

 Symmetry transformations used to generate equivalent atoms:
 #1 -x+y,-x,z
 #2 x,y,-z+3/2
 #3 -x+y,-x,-z+3/2

 #4 -x+y,-x-1,z
 #5 -y-1,x-y-1,z
 #6 -x+y+1,-x,z
 #7 -y,x-y-1,z
 #8 x-y,-y,-z+1
 #9 -y,x-y,z

O(8)-V(3)	1.611(13)	Na(1)-O(9)#8	2.373(9)
V(1)-O(1)	1.605(8)	Na(1)-O(9)	2.373(9)
V(1)-O(2)#1	1.916(3)	Na(1)-O(9)#9	2.373(9)
V(1)-O(2)	1.916(3)	Na(1)-O(6)#8	2.446(9)
V(1)-O(3)	1.951(5)	Na(1)-O(6)	2.446(9)
V(1)-O(3)#2	1.951(5)	Na(1)-O(6)#9	2.446(9)
V(3)-O(3)#2	1.976(5)	O(5)-V(2)#10	1.820(5)
V(3)-O(3)#3	1.976(5)	O(6)-Na(1)#3	2.446(9)
V(3)-O(3)	1.976(5)	O(7)-Na(3)	2.34(2)
V(3)-O(3)#4	1.976(5)	Na(2)-O(10)	2.90(5)
V(2)-O(4)	1.621(8)	Na(2)-O(10)#11	2.90(5)
V(2)-O(5)	1.820(5)	Na(2)-O(2)#11	2.868(11)
V(2)-O(3)#5	1.915(5)	O(10)-Na(3)#7	3.11(3)
V(2)-O(3)	1.915(5)	Na(3)-O(7)#13	2.34(2)
V(2)-O(2)	2.009(9)	Na(3)-O(1)#14	2.291(14)
O(2)-V(1)#6	1.916(3)	Na(3)-O(1)#15	2.291(14)
O(2)-Na(2)	2.868(11)	Na(3)-O(10)#15	3.11(3)
O(1)-Na(3)#7	2.291(14)	Na(3)-O(10)#14	3.11(3)
		O(3)#2-V(3)-O(3)#4	92.8(3)
O(1)-V(1)-O(2)#1	106.4(3)	O(3)#3-V(3)-O(3)#4	79.1(3)
O(1)-V(1)-O(2)	106.4(3)	O(3)-V(3)-O(3)#4	149.3(3)
O(2)#1-V(1)-O(2)	94.3(5)	O(4)-V(2)-O(5)	104.7(4)
O(1)-V(1)-O(3)	109.0(3)	O(4)-V(2)-O(3)#5	109.47(16)
O(2)#1-V(1)-O(3)	144.0(3)	O(5)-V(2)-O(3)#5	91.16(18)
O(2)-V(1)-O(3)	82.1(3)	O(4)-V(2)-O(3)	109.47(16)
O(1)-V(1)-O(3)#2	109.0(3)	O(5)-V(2)-O(3)	91.16(18)
O(2)#1-V(1)-O(3)#2	82.1(3)	O(3)#5-V(2)-O(3)	139.0(3)
O(2)-V(1)-O(3)#2	144.0(3)	O(4)-V(2)-O(2)	99.6(4)
O(3)-V(1)-O(3)#2	80.3(3)	O(5)-V(2)-O(2)	155.7(4)
O(8)-V(3)-O(3)#2	105.34(17)	O(3)#5-V(2)-O(2)	80.67(18)
O(8)-V(3)-O(3)#3	105.34(17)	O(3)-V(2)-O(2)	80.67(18)
O(3)#2-V(3)-O(3)#3	149.3(3)	V(2)-O(3)-V(1)	99.6(2)
O(8)-V(3)-O(3)	105.34(17)	V(2)-O(3)-V(3)	137.7(3)
O(3)#2-V(3)-O(3)	79.1(3)		
O(3)#3-V(3)-O(3)	92.8(3)	V(1)-O(3)-V(3)	99.9(2)
O(8)-V(3)-O(3)#4	105.34(17)	V(1)#6-O(2)-V(1)	141.1(5)

Table S2. Bond lengths (Å) and angles (°) for compound 2

V(1)#6-O(2)-V(2)	97.6(3)	O(10)-Na(2)-O(10)#11	97.1(12)
V(1)-O(2)-V(2)	97.6(3)	O(10)-Na(2)-O(2)#11	107.1(2)
V(1)#6-O(2)-Na(2)	105.0(2)	O(10)#11-Na(2)-O(2)#11	107.1(2)
V(1)-O(2)-Na(2)	105.0(2)	O(10)-Na(2)-O(2)	107.1(2)
V(2)-O(2)-Na(2)	105.6(5)	O(10)#11-Na(2)-O(2)	107.1(2)
V(1)-O(1)-Na(3)#7	168.8(6)	O(2)#11-Na(2)-O(2)	127.4(7)
O(9)#8-Na(1)-O(9)	94.6(4)	Na(2)-O(10)-Na(3)#7	86.7(12)
O(9)#8-Na(1)-O(9)#9	94.6(4)	O(7)-Na(3)-O(7)#13	103.1(14)
O(9)-Na(1)-O(9)#9	94.6(4)	O(7)-Na(3)-O(1)#14	170.9(9)
O(9)#8-Na(1)-O(6)#8	90.5(3)	O(7)#13-Na(3)-O(1)#14	86.0(6)
O(9)-Na(1)-O(6)#8	171.4(4)	O(7)-Na(3)-O(1)#15	86.0(6)
O(9)#9-Na(1)-O(6)#8	91.8(3)	O(7)#13-Na(3)-O(1)#15	170.9(9)
O(9)#8-Na(1)-O(6)	91.8(3)	O(1)#14-Na(3)-O(1)#15	84.9(7)
O(9)-Na(1)-O(6)	90.5(3)	O(7)-Na(3)-O(10)#15	95.0(5)
O(9)#9-Na(1)-O(6)	171.4(4)	O(7)#13-Na(3)-O(10)#15	95.0(5)
O(6)#8-Na(1)-O(6)	82.5(3)	O(1)#14-Na(3)-O(10)#15	84.0(6)
O(9)#8-Na(1)-O(6)#9	171.4(4)	O(1)#15-Na(3)-O(10)#15	84.0(6)
O(9)-Na(1)-O(6)#9	91.8(3)	O(7)-Na(3)-O(10)#14	95.0(5)
O(9)#9-Na(1)-O(6)#9	90.5(3)	O(7)#13-Na(3)-O(10)#14	95.0(5)
O(6)#8-Na(1)-O(6)#9	82.5(3)	O(1)#14-Na(3)-O(10)#14	84.0(6)
O(6)-Na(1)-O(6)#9	82.5(3)	O(1)#15-Na(3)-O(10)#14	84.0(6)
V(2)-O(5)-V(2)#10	132.4(6)	O(10)#15-Na(3)-O(10)#14	163.8(17)
Na(1)#3-O(6)-Na(1)	80.8(4)		

Symmetry transformations used to generate equivalent atoms: #1 -x+y+1,-x+2,z; #2 x-y+1,-y+2,z; #3 x,y,-z+1; #4 x-y+1,-y+2,-z+1; #5 y,x,z; #6 -y+2,x-y+1,z; #7 x,y+1,z; #8 -y+1,x-y+1,z; #9 -x+y,-x+1,z; #10 y,x,-z+1; #11 y,x,-z+2; #12 -y+1,x-y,z; #13 x-y,-y,-z+2; #14 x-y+1,-y+1,-z+2; #15 x,y-1,z; #16 -x+y+1,-x+1,z.



Section S3. Coupled TGA-IR Spectra of compound 1

Fig. S5. Coupled TGA-IR spectra of compound 1 collected in the temperature range 320 – 450 °C.



Fig. S6. TGA-DTA profile of coupled TGA-IR analysis of compound 1.

Section S4. Coupled TGA-Mass Spectra of compound 1

The carrier gas (He) was flushed through the sample port for 5 min prior to the measurement to eliminate the interference of atmospheric CO₂. The TGA profile was recorded in the temperature range 50 °C to 950 °C (10 °C/min, Fig. S4) and mass spectra were harvested between 3 to 42 min. A high intense mass peak at m/z = 44 corresponding to CO₂ observed in mass spectra of TGA effluent after 39th min in GC which corresponds to ~400 °C temperature in TGA.



Fig. S7. Mass spectrum of Coupled TGA-Mass analysis of compound 1.

Section S5. Carbonate ion confirmation test for compound 1



Fig. S8. Before (left), and after (right) addition of calcium hydroxide solution to the compound 1.





Fig. S9. Powder X-ray diffraction patterns of compounds 1 and 2, and amorphous compound.

Section S7. HRMS analysis reports of CO₂ conversion product in a Grignard reaction



Fig. S10. HR-MS analysis report of triphenyl methanol in 1:1mmol (compound 1: PhMgBr reagent) ratio reaction.



Fig. S11. HR-MS analysis report of benzoic acid in 1:1 mmol (compound 1: PhMgBr reagent) ratio reaction.

Section S8. Bond valance sum (BVS) calculations of compounds 1 and 2

Compound 1

Bond valence calculation. Numbers in brackets after atom symbols are at.no., r and c - see O"Keeffe and Brese, J.A.C.S. 1991, 113, 3226.

V1

(23, 1.21, 1.45) V Rij Dij Vij (8, .63, 3.15) 1.77 1.50 -0 1.62 -0 .63, 3.15) 1.77 (8, 1.93 .65 -0 (8, .63, 3.15) 1.77 1.93 .65 -0 (8, .63, 3.15) 1.77 1.91 .70 -0 (8, .63, 3.15) 1.77 2.00 .55

Bond valence sum for V 4.05

V2

V (23, 1.21, 1.45)Rij Dij Vij -0 (8, .63, 3.15) 1.77 .59 1.97 -0 (8, .63, 3.15) 1.77 1.95 .63 -0 (8, .63, 3.15) 1.77 1.63 1.48 -0 (8, .63, 3.15) 1.77 1.83 .85 -0 (8, .63, 3.15) 1.77 1.92 .67

Bond valence sum for V 4.22

V3

V (23, 1.21, 1.45)Rij Dij Vij (8, .63, 3.15) 1.77 -0 2.29 .25 -0 (8, .63, 3.15) 1.77 1.64 1.42 (8, .63, 3.15) 1.77 -0 1.94 .63 (8, .63, 3.15) 1.77 1.94 .63 -0 .63, 3.15) 1.77 2.05 -0 (8, .47

Bond valence sum for V 3.40

Compound 2

V1

(23, 1.21, 1.45)V Rij Dij Vij -0 (8, .63, 3.15) 1.77 1.61 1.58 (8, -0 .63, 3.15) 1.77 1.92 .68 -0 (8, .63, 3.15) 1.77 1.95 .62

-0	(8	β, .	63, 3	.15) 1	.77	1.92 .	68
-0	(8	³ , .	63, 3	.15) 1	.77	1.95 .	62

Bond valence sum for V 4.18

V2

V	(23,	1.21,	1.45)						Rij	Dij	Vij
				-0	(8,	.63,	3.15)	1.77	1.62	1.51
				-0	(8,	.63,	3.15)	1.77	2.01	.53
				-0	(8,	.63,	3.15)	1.77	1.82	.88
				-0	(8,	.63,	3.15)	1.77	1.91	.68
				-0	(8,	.63,	3.15)	1.77	1.91	.68

Bond valence sum for V 4.28

V3

V	(23,	1.21,	1.45)						Rij	Dij	Vij
				-0	(8,	.63,	3.15)	1.77	1.61	1.55
				-0	(8,	.63,	3.15)	1.77	1.98	.58
				-0	(8,	.63,	3.15)	1.77	1.98	.58
				-0	(8,	.63,	3.15)	1.77	1.98	.58
				-0	(8,	.63,	3.15)	1.77	1.98	.58

Bond valence sum for V 3.87





Fig. S12. Core level and areal X-ray photoelectron spectroscopy of compounds 1 and 2.

Section S10. Calculations for Manganometric determination of Vanadium oxidation states in compounds 1 and 2

Compound 1

S No.	0.05N vanadium(IV) in 0.2N H ₂ SO ₄	Taken amount of 0.05N KMnO ₄ (in Burette)				
	solution (mL)	Initial (mL)	End point (mL)			
1	10	0	10.3			
2	10	0	10.4			
3	10	0	10.4			
	Average titre value		10.36			

Compound 2

S No.	0.05N vanadium(IV) in 0.2N H ₂ SO ₄ solution (mL)	Taken amount of 0.05N KMnO ₄ (in Burette)	
		Initial (mL)	End point (mL)
1	10	0	10.5
2	10	0	10.5
3	10	0	10.5
Average titre value			10.5

Formula for calculation:

 $\{V^{IV}_{15}\} + 3 \text{ KMnO}_4 + 24 \text{ H}^+ \rightarrow \{V^{V}_{15}\} + 3 \text{ Mn}^{2+} + 12\text{H}_2\text{O}$

10 mL of 0.05 N of KMnO₄ = 15 no. of V^{IV} centres (in 10 mL of 0.05 N of V15 cluster) x mL of 0.05 N of KMnO₄ = No. of V^{IV} centres (in 10 mL of 0.05 N of V15 cluster)

No. of V(IV) centers present(in 10 mL of 0.05 N of V15 cluster) = $\frac{15 \times x \, mL \, of \, KMn04 \, (0.05 \, N)}{10 \, mL \, of \, KMn04 \, (0.05 \, N)}$

For Compound 1,

No. of V(IV) centers present (in 10 mL of 0.05 N of V15 cluster) = $\frac{15 \times 10.36 \text{ mL of KMn04} (0.05 \text{ N})}{10 \text{ mL of KMn04} (0.05 \text{ N})}$

= 15.54

For Compound 2,

No. of V(IV) centers present(in 10 mL of 0.05 N of V15 cluster) = $\frac{15 \times 10.5 \, mL \, of \, KMn04 \, (0.05 \, N)}{10 \, mL \, of \, KMn04 \, (0.05 \, N)}$

=15.75

Section S11. ICP-OES and CHN analysis reports of compounds 1 and 2

PBMDJFKBOCKEIH KAOFMBJF PMJARAEKBFCJGD II.BAJOBF PKCAPAJGBHHGPC FGKJPHBF Issued to: Prof. Samar K. Das C/o Prof. Samar K.Das Lab -School of chemistry University of Hyderabad - 019	Report No. :LLPL/16-17/008631 Issue Date :21/12/2016 Customer Ref.:TRF
Kind Attn.:M.sateesh	Ref.Date :17/12/2016
Sample Particulars : SKD13	
Qty. Received : 260mg, Packed in Sealed bottle	
Test Parameters : Vanadium as V,Sodium as Na	
Date of Receipt of Sample : 19/12/2016	Date of Starting of Analysis : 21/12/2016
Date of completion of analysis : 21/12/2016	SAMPLE TESTED AS RECEIVED

TEST RESULTS

S.No.	. Parameters	UOM	Results
1	Vanadium as V	% by mass	34.96
2	Sodium as Na	% by mass	6.58

Instrument Used: ICP-OES Varian 720-ES

NOTE : This report and results relate only to the sample / items tested.

R.V. Rama Rao Authorized Signatory

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Fig. S13. ICP-OES analysis report of compound 1.

PENDJFKEOCKEIH KAPIKAJF WAIGENHAFCJGD PUNEBDF VEGEDIGNUGDD				
JJBHEF				
Prof. Samar K. Das	Report No.	:LLPL/17-18/008151		
C/o Prof .Samar K.Das Lab -School of chemistry University of Hyderabad - 019	Issue Date Customer R	: 10/02/2018 ef.:TRF		
Kind Attn.:M.sateesh, +91 9908652965	Ref.Date	Ref.Date :07/12/2017		
Sample Particulars: V15(CO3)				
Qty. Received : 150mg, Polythene Cover				
Test Parameters : Bismuth as Bi				
Date of Receipt of Sample : 06/02/2018 Date of completion of analysis : 10/02/2018	Date of Starting of Ana	lysis : 10/02/2018 SAMPLE TESTED AS RECEIVED		
TES	ST RESULTS	SILLE LESTED AS RECEIVED		
S.No. Parameters	UOM	Results		
1 Bismuth as Bi	nom	<10.0		
Instrument Used: ICP-OES Verter 710 ES	ppm	N10.0		
		P.V. Dorre D.c.		
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Fig. S14. ICP-OES analysis report of compound 1.

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	,					
	7					
PBMDJF	BOEKCMG					
NFJFJGJ PNCJCCC	IF CAEDCJOE					
INDDAIL)F					
KJNIHH	BF					
Issued	to:		Peport No	11 PL/D/16 17/002209		
Prof. a	samar N. Das	al of chamistry	Issue Date	21/12/2016		
Univer	sity of Hyderabad - 019	of of chemistry	Customer			
Univer	Sity Of Hyderabad - 015					
Kind A	Kind Attn.:Mr.M.Sateesh		Ref.Date :30/11/2016			
Sampl	e Particulars : [V15(CI)]					
Qty. R	eceived : 0.45gm ,Packed in	n Sealed bottle				
Test P	arameters : Vanadium as V,	Sodium as Na,Chloride as Cl				
		20/11/2016				
Date o	of Receipt of Sample :	30/11/2016	Date of Starting of Ar	nalysis : 05/12/2016		
Date o	or completion of analysis :	21/12/2010		SAMPLE TESTED AS RECEIVED		
		TEST	RESULTS			
S.No	Parameters		UOM	Results		
	Vanadium as V		% by mass	35.53		
	Sodium as Na		% by mass	10.58		
2	Chlorido os Cl		% by mass	1.69		
				B V. Pama Pao		
	Page No. 1/1			K.V. Kama Kao		
				Authorized Signatory		
	Fage No. 1/1					
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					12.1	
					1.12.8	
			Star Walks and Star		1204	
		and the second states and		AT ALCONTON DURING AL	1.11	

Fig. S15. CHN analysis report of compound 1.

Fig. S16. ICP-OES report analysis of compound 2.