

Supporting Information for

Direct Cyclic Carbonate Synthesis from CO₂ and Diol over Carboxylation/Hydration Cascade Catalyst of CeO₂ with 2-Cyanopyridine

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Details of the reagents used in experiments

The details of the reactants are described below: CO₂ (Shimakyu Co. Ltd., >99.5%), 2-cyanopyridine (Tokyo Chemical Industry Co. Ltd., 99.0% min.), 2-picolinamide (TCI, 98.0% min.), ethylene glycol (Wako, 99.5% min.), 1,2-propanediol (Wako, 99.0% min.), 1,2-butanediol (Wako, 98.0% min.), 3-methoxy-1,2-propanediol (TCI, 98.0% min.), 3,4-dihydroxy-1-butene (Wako, 94.0% min.), phenylethylene glycol (TCI, 98.0% min.), *cis*-1,2-cyclopentanediol (Frinton Laboratories, 97% min.), 1,4-anhydroerythritol (Aldrich, 95% min.), 1,3-propanediol (Wako, 97.0% min.), 1,3-butanediol (Wako, 98.0% min.), 2-methyl-1,3-propanediol (TCI, 98.0% min.), 2,2-dimethyl-1,3-propanediol (TCI, 98.0% min.), 2-phenyl-1,3-propanediol (TCI, 98.0% min.), 2,4-pentanediol (TCI, 98.0% min.), ethanol (Wako, 99.5% min.) and 1-hexanol (TCI, 98.0% min.).

In order to determine the retention time and molar sensitivity, commercially available reagents were used; Ethylene carbonate (TCI, 99.0% min.), propylene carbonate (TCI, 98.0% min.), 1,3-dioxan-2-one (TCI, 98.0 min.) and 1,2-butylene carbonate (TCI, 98.0% min.). In the case of carbonates derived from 3-methoxy-1,2-propanediol, phenylethylene glycol, 1,3-propanediol, 2-methyl-1,3-propanediol, 2,2-dimethyl-1,3-propanediol and 2,4-pentanediol, the retention time was determined by GC-MS, and these compounds were identified by MS pattern. The yields were calculated by using the molar sensitivity of corresponding diols and gas chromatogram area. In the case of carbonates derived from 3,4-dihydroxy-1-butene, *cis*-1,2-cyclopentanediol, 1,4-anhydroerythritol and 2-phenyl-1,3-propanediol, ¹H-NMR spectra were recorded on Bruker RTX-400 (400 MHz; CDCl₃; Me₄Si) for identification. The yields were also calculated by using the molar sensitivity of corresponding diols and gas chromatogram area.

¹H-NMR, ¹³C-NMR and MS analyses

¹H and ¹³C NMR spectra were recorded using at ambient temperature on Bruker RTX-400 operating at 400 MHz. Chemical shifts are reported in δ (ppm) referenced to the residual peaks of CDCl₃ (δ 7.26) for ¹H NMR and CDCl₃ (δ 77.00) for ¹³C NMR. Abbreviations used in the NMR experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad.

Ethylene carbonate (2a)

¹H NMR and ¹³C NMR were confirmed by the literature.¹

Propylene carbonate (2b)

¹H NMR and ¹³C NMR were confirmed by the literature.²

4-Ethyl-1,3-dioxolan-2-one (2c)

¹H NMR and ¹³C NMR were confirmed by the literature.³

4-Ethenyl-1,3-dioxolan-2-one (2e)

¹H NMR δ_H ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ 5.86 (1H, m), 5.44 (1H, dd, J=17, 6.8 Hz), 5.39 (1H, dd, J=10, 0.8 Hz), 5.08 (1H, q, J=7.5 Hz), 4.56 (1H, t, J=8.3 Hz), 4.10 (1H, t, J=8.0 Hz).

¹³C NMR δ_C ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ 155, 132, 121, 77, 69.

The mass spectrum (EI, 70 eV) is as follows: *m/z* (%): 69 (5), 55 (7), 43 (13), 42 (100), 41 (25), 40 (24) 39 (47),

38 (5), 30 (5).

4-Phenyl-1,3-dioxolan-2-one (2f)

¹H NMR and ¹³C NMR were confirmed by the literature.²

Tetrahydro-4*H*-cyclopenta-1,3-dioxol-2-one (2g)

¹H NMR δ_{H} ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ . 5.15-5.11 (2H, dd *J*=3.77, 3.51 Hz), 2.2-1.8 (6H, m).

¹³C NMR δ_{C} ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ .151, 82, 33, 22.

The mass spectrum (EI, 70 eV) is as follows: *m/z* (%): 128 (3), 99 (3), 83 (11), 69 (4), 57 (5), 56 (29), 55 (100), 53 (5), 44 (8), 43 (4), 42 (10), 41 (26), 39 (14).

Tetrahydro-furo[3,4-*d*]-1,3-dioxol-2-one (2h)

¹H NMR δ_{H} ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ . 5.26 (2H, dd *J* 1.0 Hz), 4.27 (2H, dd *J* 1.2 Hz), 3.60 (2H, dd *J* 1.2 Hz).

¹³C NMR δ_{C} ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ . 155, 80, 73.

The mass spectrum (EI, 70 eV) is as follows: *m/z* (%): 130 (18), 101 (3), 73 (5), 57 (13), 56 (17), 55 (100), 45 (3), 44 (7), 43 (10), 42 (4), 41 (3), 39 (5), 31 (23), 30 (3).

1,3-Dioxan-2-one (3a)

¹H NMR and ¹³C NMR were confirmed by the literature.⁴

4-Methyl-1,3-dioxan-2-one (3b)

¹H NMR and ¹³C NMR were confirmed by the literature.⁵

5-Methyl-1,3-Dioxan-2-one (3c)

¹H NMR and ¹³C NMR were confirmed by the literature.⁶

5-Phenyl-1,3-dioxan-2-one (3d)

¹H NMR δ_{H} ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ . 7.43-7.30 (3H, m), 7.23 (2H, m) 4.62-4.49 (4H, m) 3.50 (1H, m)

¹³C NMR δ_{C} ppm (400 MHz; CDCl₃; (CH₃)₄Si): δ . 149, 134, 130, 129, 128, 72, 38.

The mass spectrum (EI, 70 eV) is as follows: *m/z* (%): 178 (23), 105 (11), 104 (100), 103 (17), 91 (8), 79 (4), 78 (22), 77 (14), 65 (4), 63 (4), 52 (4), 51 (13), 50 (4), 39 (6).

5,5-Dimethyl-1,3-dioxan-2-one (3e)

¹H NMR and ¹³C NMR were confirmed by the literature.⁴

4,6-Dimethyl-1,3-dioxan-2-one (3f)

¹H NMR and ¹³C NMR were confirmed by the literature.¹

Table S1. Details of Table 1

Catalyst	BET surface area / m ² g ⁻¹	1,2-PrD conv. / %	PC yield / %	PC selec. / %	Produced amount of PC / mmol	Residual amount of 2-cyanopyridine / mmol	Produced amount of 2-picolinamide / mmol
CeO ₂ ^a	89	9.4	9.4	>99	0.94	99	1.0
MgO	41	59	0.9	1.4	0.09	98	0.23
ZrO ₂	116	1.2	0.5	38	0.05	99	0.49
ZnO	4	1.8	0	0	0.0	99	0.05
SnO ₂	35	0.9	0	0	0.0	98	0.37
TiO ₂	48	8.9	0	0	0.0	99	0.33
γ-Al ₂ O ₃	97	0.2	0	0	0.0	99	0.06
α-Al ₂ O ₃	11	0.2	0	0	0.0	99	0.02
La ₂ O ₃	26	0.3	0	0	0.0	99	0.11
Pr ₆ O ₁₁	30	4.1	0	0	0.0	96	0.25
Sm ₂ O ₃	41	4.1	0	0	0.0	98	0.09
Y ₂ O ₃	72	0.9	0	0	0.0	99	0.10
SiO ₂ -Al ₂ O ₃	334	16	0	0	0.0	95	2.2
SiO ₂	419	0.2	0	0	0.0	99	0.0
none	-	0.0	0	0	0.0	>99	0.05
Ce(NO ₃) ₃ ·6H ₂ O	-	0.6	0	0	0.0	99	0.09

Reaction conditions: Catalyst (metal=2 mmol), 1,2-PrD/2-cyanopyridine = 10 mmol/100 mmol, CO₂ 5 MPa, 423 K, 1 h. ^aCeO₂ 0.03 mmol.

The details of the catalysts are described below: Ce(NO₃)₃·6H₂O (Wako, 98.0%), ZrO₂ (Daiichi Kigenso Kogyo Co. Ltd., Zr(OH)₂ was calcined under air at 673 K for 3 h.), MgO (Ube Industries, Ltd., MgO 500A), ZnO (Kanto Chemical Co. Inc., 99.0% min.), SnO₂ (Kanto Chemical Co. Inc., 98.0% min.), TiO₂ (Nippon Aerosil Co. Ltd., P-25), α-Al₂O₃ (Sumitomo Chemical, KHO-24 was calcined at 1423 K under air for 3 h.), γ-Al₂O₃ (Nippon Aerosil Co. Ltd.), SiO₂ (Fuji Silysia Chemical Ltd., G-6 was calcined under air at 773 K for 1 h.). La₂O₃, Pr₆O₁₁, Sm₂O₃ and Y₂O₃ were prepared by precipitation method. La(NO₃)₃·6H₂O (Wako, 99.9% min.), Pr(NO₃)₃·nH₂O (Wako, 99.5% min.), Sm(NO₃)₃·6H₂O (Wako, 99.5% min.), Y(NO₃)₃·nH₂O (Wako, 99.9% min.) and SiO₂-Al₂O₃ (JRC-SAL-3) were used as a precursor. A precursor (25 g) was dissolved in water (100 ml) and NH₃aq (1 M) was dropped with stirring. The pH of the solution was set to 10 and resulted in a precipitate. The precipitate was filtered and washed by water, following a drying at 383 K overnight (12 h) and calcined under air at 673 K (La) and 873 K (Pr, Sm, Y) for 3 h.

Low PC selectivity in the case of MgO, TiO₂, Pr₆O₁₁ and Sm₂O₃ is mainly attributed to the formation of ether.

Table S2. Details of Table 2

Entry	Amount of 2-cyano- Pyridine / mmol	P_{CO_2} / MPa	Temp. / K	Time / h	1,2-PrD conv. / %	PC yield / %	Produced amount of PC / mmol	Residual amount of 2-cyanopyridine / mmol	Produced amount of 2-picolinamide / mmol
1	0	5	413	24	0.34	0.33	0.03	-	-
2	30	5	413	1	92	92	9.2	21	9.4
3	100	5	403	1	95	95	9.5	91	10
4	100	5	363	48	>99	>99	9.7	87	11
5*	200	0.8	413	24	97	90	8.9	183	5.2

Reaction conditions: CeO_2 2 mmol, 1,2-PrD 10 mmol, * CeO_2 5 mmol.

In entry 5, ester was detected by GC-MS. It can be formed by the reaction of 1,2-PrD with 2-picolinamide.

Table S3. Reported yields for the synthesis of cyclic carbonates from CO₂ and corresponding alcohols

Entry	Catalyst	Alcohol	P _{CO₂} / MPa	Solvent or dehydrating agent	T / K	t / h	Yield / %	Selec. / %	Ref.
Homogeneous catalyst									
1	<i>n</i> -Bu ₂ SnO	1,2-PrD	15	DMF	453	12	1.7	100	7
2	<i>n</i> -Bu ₂ Sn(OMe) ₂	1,2-PrD	15	DMF	453	12	1.9	100	7
3	<i>n</i> -Bu ₂ Sn(OMe) ₂	1,2-PrD	15	DMF + ketal	453	12	3.4	100	7
4	Ti(OPr) ₄	1,2-PrD	15	DMF	453	12	1.2	100	7
5	<i>n</i> -Bu ₂ Sn(OMe) ₂	Glycerol	5	Molecular sieves	450	14	6.9	100	8
6	Cs ₂ CO ₃ + (NH ₄) ₂ CO ₃	1,2-PrD	10	CH ₃ CN	448	15	10.5	100	9
7	Zn(OAc) ₂	EG	10	CH ₃ CN	443	12	10.8	58.4	10
8	Zn(OAc) ₂	1,2-PrD	10	CH ₃ CN	443	12	24.2	62.2	10
9	Zn(OAc) ₂	1,3-PrD	10	CH ₃ CN	443	12	13.8	60.5	10
10	Zn(OAc) ₂	1,2-BuD	10	CH ₃ CN	443	12	17.3	60.9	10
11	Zn(OAc) ₂	1,3-BuD	10	CH ₃ CN	443	12	12.1	61.7	10
12	TBD	1,2-PrD	10	CH ₃ CN	448	15	22.5	60.3	11
13	Zn(OAc) ₂	1,2-PrD	3	CH ₃ CN	433	2	12.3	64.1	12
14	Mg	EG	15	-	453	12	0.7	100	13
15	Mg	1,2-PrD	15	-	453	12	2.8	100	13
16	Mg	PhEG	15	-	453	12	1	100	13
17	<i>n</i> -Bu ₂ SnO	EG	13.8	CH ₃ OH + Zeolite	353	4	61	100	14
18	<i>n</i> -Bu ₂ SnO	1,2-PrD	13.8	CH ₃ OH + Zeolite	353	4	42	100	14
19	<i>n</i> -Bu ₂ SnO	Glycerol	13.8	CH ₃ OH + Zeolite	353	4	35	100	14
20	RhCl ₃ + PPh ₃ + KI (H ₂ 1)	Glycerol	4	CH ₃ OH	413	59	0.24	100	15
21	K ₂ CO ₃	1,2-PrD	10	C ₆ H ₅ CN	448	18	19.8	45	16
22	Cs ₂ CO ₃	1,2-PrD	10	C ₆ H ₅ CN	448	18	16.3	37	16
23	Na ₂ CO ₃	1,2-PrD	10	C ₆ H ₅ CN	448	18	7.9	36	16
24	Li ₂ CO ₃	1,2-PrD	10	C ₆ H ₅ CN	448	18	4.0	67	16
25	K ₂ CO ₃	1,2-OcD	10	C ₆ H ₅ CN	448	18	10	-	16
Heterogeneous catalyst									
26	CeO ₂ -ZrO ₂	1,2-PrD	3.5	CH ₃ CN	383	8	2.0	100	17
27	CeO ₂ -ZrO ₂	EG	7	CH ₃ CN	423	2	1.33	100	18

28	KI/ZnO	1,2-PrD	10	CH ₃ CN	433	15	26.0	61.0	19
29	La ₂ O ₃ CO ₃ -ZnO	Glycerol	4.0	CH ₃ CN	443	12	14.3	47.2	20

EG: Ethyleneglycol, 1,2-PrD: 1,2-Propanediol, 1,3-PrD: 1,3-Propanediol, 1,2-BuD: 1,2-Butanediol, 1,3-BuD: 1,3-Butanediol, PhEG: Phenylethylene glycol, 1,2-OcD: 1,2-Octanediol.

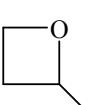
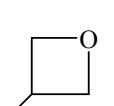
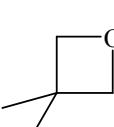
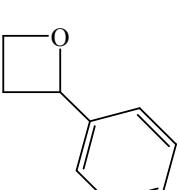
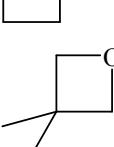
Table S4. Details of Schemes 2 and 3

Entry	Diol	Temp. / K	Time / h	Diol conv. / %	Carbonate yield / %	Produced amount of carbonate / mmol	Residual amount of 2-cyanopyridine / mmol	Produced amount of 2-picolinamide / mmol
1		403	1	>99	>99	10	90	10
2		413	1	99	99	9.9	89	10
3		413	1	>99	>99	10	88	11
4		423	2	99	97	9.7	89	11
5		433	1	72	67	6.7	90	8.3
6		453	48	96	95	9.5	88	10
7		403	1	>99	>99	10	89	11
8		443	8	>99	>99	10	89	10
9 ^{a)}		413	1	86	58	5.8	85	10
10		413	1	>99	84	8.4	87	11
11		433	1	95	73	7.3	89	10
12		443	2	67	62	6.2	93	7.1
13		423	2	96	95	9.5	89	10
14		443	8	97	97	9.7	89	10

Reaction conditions: CeO₂ 2 mmol, diol/2-cyanopyridine = 10 mmol/100 mmol, CO₂ 5 MPa, ^{a)} 2-cyanopyridine 200 mmol.

The difference of the amounts between carbonate and 2-picolinamide (entries 5, 6, 9, 13) is due to the formation of by-products such as an ester derived from 2-picolinamide with diol (entry 5), pyridine (entries 6, 13) and oligomers of diols (entry 9).

Table S5. Catalytic reactions of 6-membered ring carbonate formation from CO₂ + corresponding oxetane

Catalyst	Oxetane / mmol	CO ₂ / MPa	Solvent / ml	Temp. / K	Time / h	Conv. / %	Selec. / %	Yield / %	Ref.
Ph ₄ SbI		4.9	-	373	4			96	21
Bu ₃ SnI + HMPA*		4.9	-	373	24			100	22
(C ₆ H ₅) ₄ SbI		4.9	-	373	4			96	23
(C ₆ H ₅) ₄ SbI		4.9	-	423	4			85	23
(C ₆ H ₅) ₄ SbI		4.9	-	393	4			93	23
(C ₆ H ₅) ₄ SbI		4.9	-	423	4			19	23
(C ₆ H ₅) ₄ SbI		4.9	-	373	4			96	23
Co(acac) ₂ + n-Bu ₄ NBr		3.5	Toluene 10	333	8	95	100	95	24
(salen)CrCl + n-Bu ₄ NBr		1	Toluene 10	323	24	20.3	100	20.3	25
Al complex + TBAI**		10	-	343	4	>99	95	95	26
Al complex + TBAI**		10	-	343	66	59	54	26	26

*HMPA: Hexamethylphosphoric triamide, **TBAI: *n*-Tetrabutylammonium iodide

Table S6. Details of the catalyst recycle test

Usage times	1,2-PrD conv. / %	PC / %	Produced amount of PC / mmol	Residual amount of 2-cyanopyridine / mmol	Produced amount of 2-picolinamide / mmol	BET surface area / m ² g ⁻¹	Particle size / nm
1 (Fresh)	99	99	9.9	89	10	89	9.5
2	>99	99	9.9	89	11	82	10
3	>99	99	9.9	89	11	78	9.5
4	>99	99	9.9	88	11	77	9.5

Reaction conditions: CeO₂ 2 mmol, 1,2-PrD/2-cyanopyridine =10 mmol/100 mmol, CO₂ 5 MPa, 413 K, 1 h.

^{a)} Without calcination.

The reusability test of CeO₂ was conducted as follows: The used catalysts were collected by the filtration. The collected catalysts were washed by ethanol (30 ml) for 3 times and dried at 383 K for 2 h, followed by calcination in air at 873 K for 3 h. After this treatment, the recovered catalyst is applied to the successive reaction. Multiple runs were conducted at the same time in the same conditions to collect enough amount of used catalyst, and the loss of the catalyst was compensated by reducing the number of runs. The surface area of CeO₂ was measured with BET method (N₂ adsorption) using Gemini (Micromeritics). X-ray diffraction (XRD) patterns were recorded by Rigaku Ultima IV with CuK_a (40 kV, 40 mA) radiation and particle size was estimated by Sherrer equation. From these results, it is concluded that the structure of the catalyst was stable during the reaction and the regeneration procedure, and the catalyst is easily reusable.

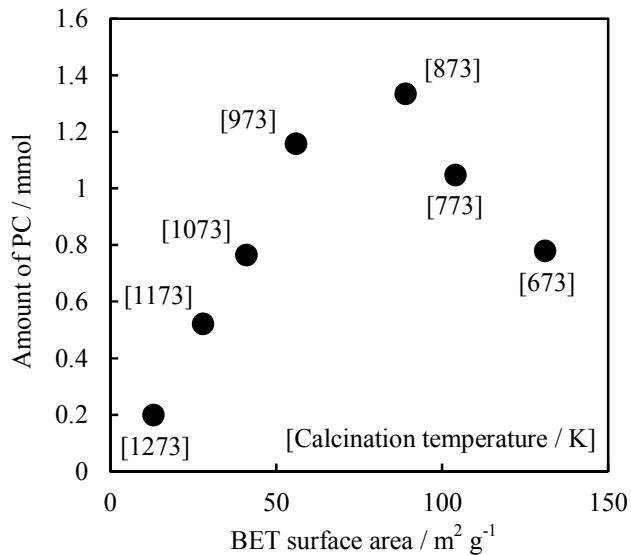


Figure S1. Effect of calcination temperature of CeO_2 in the reaction of 1,2-PrD + CO_2 with 2-cyanopyridine. Reaction conditions: CeO_2 0.06 mmol, 1,2-PrD/2-cyanopyridine = 10 mmol/100 mmol, CO_2 5 MPa, 413 K, 1 h. The acidities of CeO_2 calcined at 673, 873 and 1073 K are 0.14, 0.056 and 0.007 mmol g^{-1} , respectively²⁷.

Preparation of CeO_2 catalyst was carried out by calcinating commercially available cerium oxide HS (Daiichi Kigenso Kagaku Kogyo Co. Ltd., Japan) for 3 hours, under air atmosphere at each temperature. The preparation conditions were optimized on the basis of the results on the reaction of 1,2-PrD + CO_2 with 2-cyanopyridine.

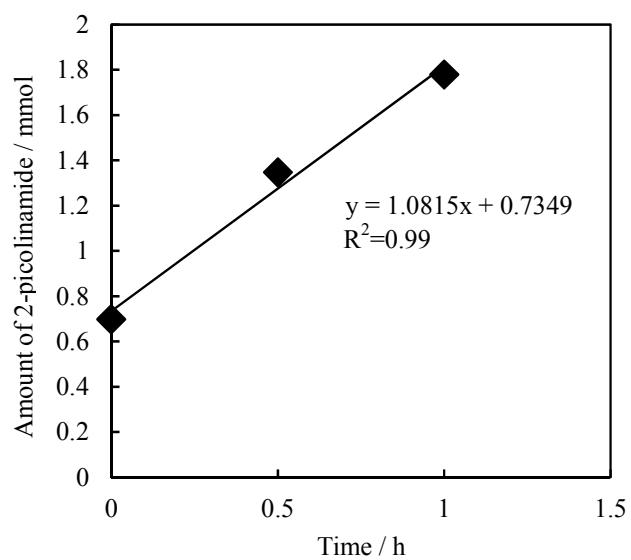


Figure S2. Measurement of 2-cyanopyridine hydration rate in the reaction of 1,2-PrD + CO₂ + 2-cyanopyridine + H₂O

Reaction conditions: CeO₂ 0.006 mmol, 1,2-PrD : 2-cyanopyridine : H₂O = 10 mmol : 100 mmol : 10 mmol, CO₂ 5 MPa, 413 K.

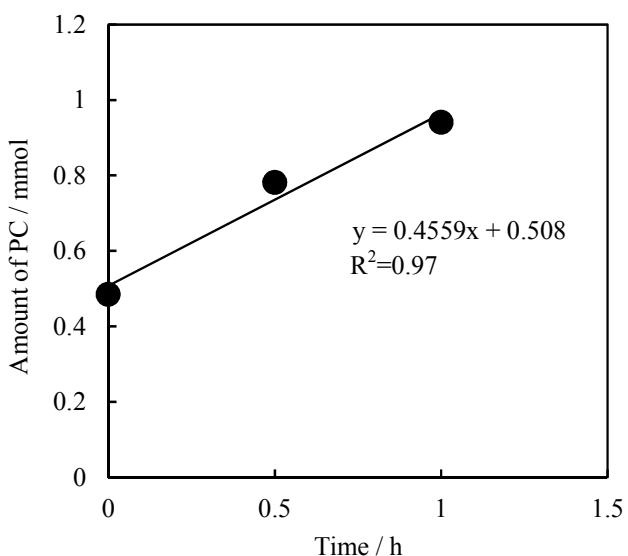


Figure S3. Measurement of PC formation rate in the reaction of 1,2-PrD + CO₂ + 2-cyanopyridine

Reaction conditions: CeO₂ 0.03 mmol, 1,2-PrD : 2-cyanopyridine = 10 mmol : 100 mmol, CO₂ 5 MPa, 413 K.

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