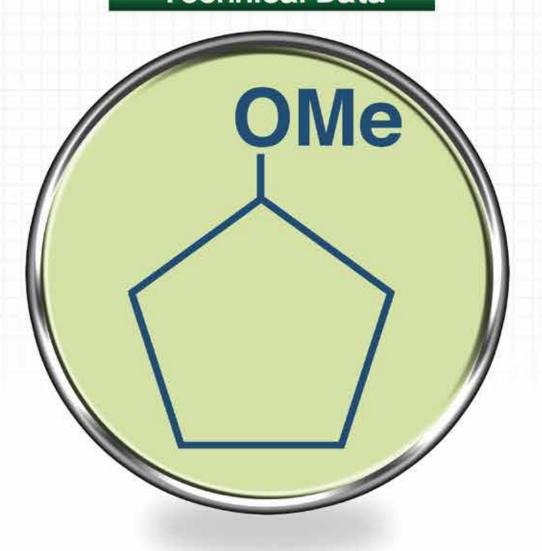
Novel hydrophobic ether solvent

Cyclopentyl methyl ether



Technical Data



ZEON CORPORATION

2) Solubility of CPME vs. Water 3 3) Azeotropic distillation of CPME 3 4) Distillation of CPME 3 4) Distillation of CPME 5 4 5 6 7 6 6 7 6 6 7 6 6		O D CO CCDUE	
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(2) Effects of CPME on Rubbers — 20 Vapor Pressure — 21 Vapor-liquid Equilibrium of Water-CPME — 21			
№ Vapor-liquid Equilibrium of Water-CPME ————21		(2) Effects of CPME on Rubbers	 20
№ Vapor-liquid Equilibrium of Water-CPME ————————————————————————————————————			
		■ Vapor-liquid Equilibrium of Water-CPME ————	<u> </u>
	(1)		





HIGH HYDROPHOBICITY

Easy separation and recovery from water, reducing emissions and wastewater

Wide applicability as a reaction, extraction and crystallization solvent, giving simple and One-pot syntheses

WIDE LIQUIDITY RANGE

Wide applications from lower to higher temperature, accelerating reaction rate

LOW HEAT OF VAPORIZATION

Saving energy for distillation and recovery

RESIST PEROXIDE FORMATION

Low exothermic decomposition energy of solvent containing it's peroxides

NARROW EXPLOSION AREA
STABLE TO ACIDS OR BASES

EASY DRYING

Physical Properties

	СРМЕ	MeTH	F	THF		Diethy ether		Dioxa	ne	МТВ	E
Relative density	0.86	0.85	*d	0.89	*a	0.71	*b	1.03	*a	0.70	*a
Vapor specific gravity (air=1)	3.45	2.97	*d	2.5	*a	2.6	*a	3.0	*a	3.0	*a
Boiling point [°C]	106	80.2	*d	66	*a	35	*a	101	*a	55	*a
Melting point [°C]	<-140	-136	*d	-108.5	*a	-116	*a	12	*a	-109	*a
Viscosity (20°C) [cP]	0.57	0.46 (25°C)	*d	0.55	*b	0.24	*b	1,31	d*		
Surface tension (20°C) [mN/m]	25.17			26.4	*b	17.3	*b	36.9	ď*		
Heat of vaporization (boiling point) [kcal/kg]	69.2	87.1	*d	98.1	*b	86.1	*b	98.6	d*		
Specific heat (20℃) [kcal/kg · k]	0.435			0.469	*b	0.584	*c	0.41	*b		
Dielectric constant (25°C)	4.76	6.97	*d	7.58	*b	4.20	*b	2.24	*b		
Azeotropic temperature with water [°C]	83	71	*d	64	*C	34	*b	88	*b		
Azeotropic composition (Solvent / Water, wt%)	83.7/16.3	89.4/10.6	*d	94.0/6.0	*C	98.7/1.3	*b	81.6/18.4	1 *b		
Solubility in water (23°C) [g/100g]	1.1	14 (20°C)	*d	∞	*a	6.9 (20°C)	*a	8	*a	4.2	*a
Solubility of water in solvent (23°C) [g/100g]	0.3	4 (20°C)	*d	∞	*b	1.2	*b	8	*b		
Flash point [°C]	-1	-11	*d	-14.5	*a	-45	*a	12	*a	-28	*a
Auto Ignition temperature [°C]	180	270	*e	321	*a	160-180	*a	180	*a	375	*a
Explosion range [vol%] Lower limit	1.1	1.5	*e	2	*a	1.7	*a	2	*a	1.6	*a
Upper limit	9.9	8.9	*e	11.8	*a	48	*a	22	*a	15.1	*a

ref:

^{*}a; International Chemical Safety Cards (ICSC)

^{*}b;溶剤ハンドブック、講談社 (1989) (Youzai hand book, Kodansha Ltd., 1989)

^{*}c;溶剤ポケットブック、オーム社 (2001) (Youzai pocket book, Ohmsha,Ltd., 2001)

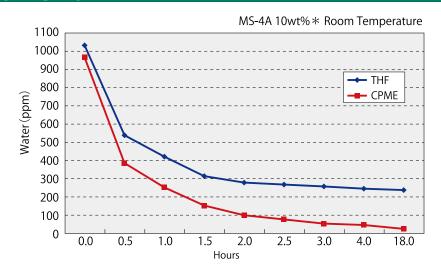
^{*}d; Org. Process Res. Dev., 2007, 11 (1), pp 156-159

^{*}e; Penn A Kem, Methyltetrahydrofuran, MSDS Date: 10/1/2010

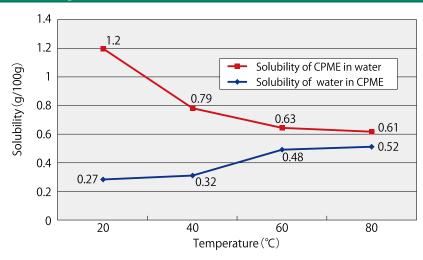


3 High Hydrophobicity

(1) Drying by Molecular sieves

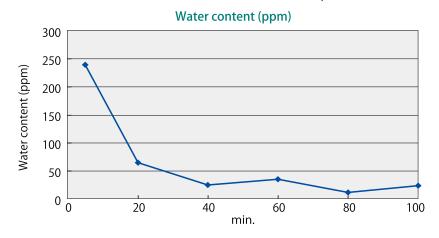


(2) Solubility of CPME vs. Water



(3) Azeotropic distillation of CPME with Dean-Stark Trap

Conditions: CPME saturated with water was refluxed with Dean-Stark Trap and then water content was monitored.



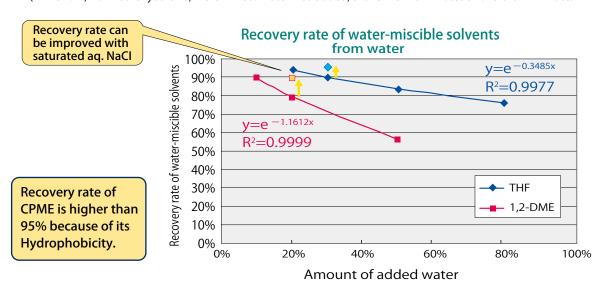
(4) Distillation of CPME saturated with water

Fractional Distillation of CPME saturated with water (500g)

Fraction	Boiling Point (°C)	Weigjt (gram)	CPME (%)	Water (ppm)
1	81-84	11	99.3	
2	84-105	10	99.9	617
3	105-106	454	99.9	92
Residue		24		
Total		498		

(5) Recovery of water-miscible solvents from water

Conditions: At room temperature, equal weight of CPME and water-miscible solvents (THF and 1,2-Dimethoxyethane) were mixed. Water was added, shaken for 10 minutes and left for 1 minute.



(6) Azeotropes with other solvents

Solvents with which CPME forms azeotropes

Solvent	Boiling Point of Solvent (°C)	Azeotropic temperature (°C)	Composition Solvent / CPME (wt%)
Water	100	83	16/84
Methanol	65	63	85/15
Dimethyl carbonate	90	90	67/33
Acetonitrile	82	82	63/37

Solvents with which CPME does not form azeotropes

	<u> </u>	
Ketones	Acetone MEK MIBK	
Hydrocarbons	n-Hexane n-Heptane* Toluene*	
Ethers	THF DME	
Alcohols	IPA	
Esters	Ethyl acetate	
Others	DMSO	

^{*} Hard to separate



(7) Azeotropes with water at different pressures

Pressure (kPa)	Azeotropic Temperature (°C)	Composition Water/CPME (wt%)
101.3	83.0	16.3/83.7
19.1	44.7	19.6/80.4
11.4	34.2	19.5/80.5
7.5	26.1	17.5/82.5

(8) Distribution of ketones between CPME and water

Conditions : Ketones were added to mixture of 10g of CPME and 5g of ion-exchange water. The mixtures were cooled down to 5° C and vigorously shaken. They were cooled down to 5° C again.

Acetone

MEK

MIBK

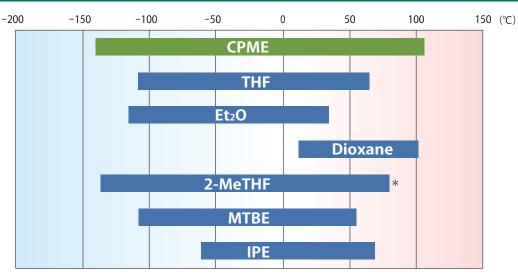
Amount	Weight Acet	
of Acetone	Layer of CPME	Layer of Water
1 g	0.49	0.51
2g	0.53	0.47
4g	0.58	0.42

Amount	Weight ratio of MEK			
MEK	Layer of CPME	Layer of Water		
1g	0.83	0.17		
2g	0.85	0.15		
4g	0.88	0.12		

Amount	Weight ratio of MIBK		
MIBK	Layer of CPME	Layer of Water	
1g	0.99	0.01	
2g	0.99	0.01	
4g	0.99	0.01	

The solubility of CPME in water was around 1%.

Liquidity range of ether solvents



ref.: International Chemical Safety Cards (ICSC) *: Org. Process Dev., 2007, 11(1), PP 156-159



Evaporation rate

Method: ASTM D 3539-87 Standard Test Methods for

Evaporation Rates of Volatile Liquids by Shell Thin-Film Evaporometer

Conditions: 23°C x50%RH

Solvent	Relative evaporation rate
СРМЕ	3.5
Butyl acetate	1

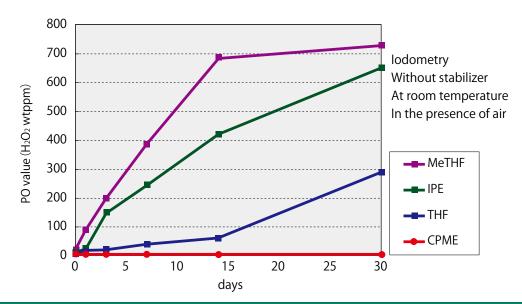




(1) Peroxide Formation of Ether Solvents

Conditions:

- 20ml of each sample in a brown bottle (capacity: 65 ml)
- CPME: Distilled over with benzophenone-sodium mixture
 THF (Tetrahydrofuran): commercially available dried product without stabilizers (Wako)
 MTBE (Methyl t-butyl ether): commercially available dried product without stabilizers (Aldrich)
 IPE (Diisopropylether): Distilled over with benzophenone-sodium mixture of a commercial product (Aldrich)
- Storage: at room temperature, in a dark place, in the presence of air Measurement times: after 0, 1, 3, 7, 14, 30 days Number of samples n = 2
- Titration method: Add ag.acetic acid, ag.Kl and titrate produced l2 with ag.sodium thiosulphate (lower limit: 1 ppm)



(2) SC-DSC Analysis

Conditions: Sealed cell type, in the air

Peroxide value (H ₂ O ₂ wt ppm)	Exothermic intiation temperature (°C)	Heat Generation (J/g)
4.5	128.4	61.4
30.0	122.0	67.0
150.0	129.5	77.9

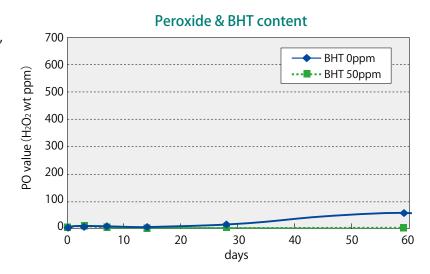
(3) ARC Test (Accelerating Rate Calorimeter)

- ●Pressurization by air to 5 atm: Temprature rise by 1.5°C at 106°C No heat generation afterward
- •Pressurization by nitrogen to 5 atm: No heat generation

(4) Effect of Stabilizer to Peroxide Formation

Conditions:

at 40°C, in the presence of air, in a dark place



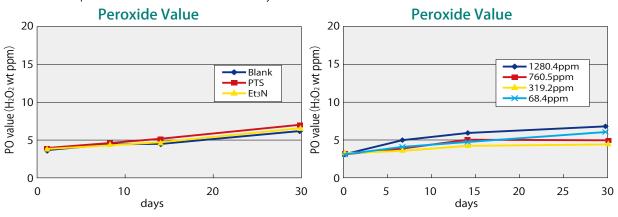
(5) Effects of Additives to Peroxide Formation

Conditions: at room temperature, in the presence of air, in a dark place

(1) Acid and Base: 100ppm each

Acid:PTS=p-Toluenesulfonic acid Base:Et3N=Triethylamine

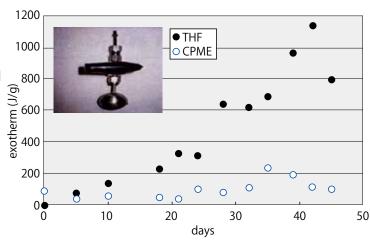
(2) Water



(6) Exothermic energy under O₂

Miyake, et al., 12th International Symposium on Loss Prevention and Safety Promotion in the Process Industries

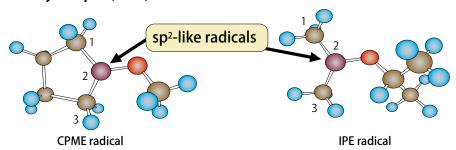
Conditions: CPME and THF without an antioxdant which were pressurized with 0.99MPa of O₂ in SUS316 closed vessel were stored at 40°C. The material was sampled at adequate interval time and the thermal analyses were carried out by using differential scanning calorimetry (DSC).





(7) MM3 simulation analysis

Kubo, H.; Sakakibara, K.; Yoshizawa, K.; Watanabe, K.; Yuzuri, T. The 85th Spring Meeting of Chemical Society of Japan (2005).



MM3 simulation

Ether radical	СРМЕ	IPE
Bond Angle (°)	115.6	119.9
Heat of Formation (kcal/mol)	44.2	40.9
Strain Energy (kcal/mol)	62.4	55.3

As a result of the simulation, the structural strain of CPME radical is calculated to be greater then that of IPE radical because of its five-membered ring structure. That should hardly cause formation of CPME radical itself. Therefore, the unstable radical of CPME is supposed to be the reason of low peroxides formation of CPME.

(8) Removal of Peroxide with aq. Na₂SO₃

Preparation:

CPME 1kg (Purity: 99.9% Peroxide: 5ppm)



stir in the air at 80°C for 4hr

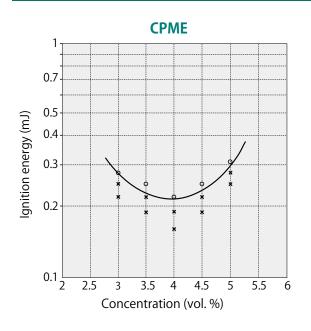
CPME 1kg (Purity: 99.5% Peroxide: 620ppm)

Conditions: 1kg of CPME containing peroxide washed with 500g of 5% Na₂SO₃ several times.

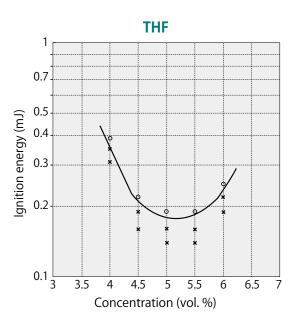
Number of washing (times)	Peroxide value (H ₂ O ₂ wt ppm)
0	620
1	116
2	95
3	7.5

9

(1) Minimum ignition energy *(E)*



0.19mJ<E<0.22mJ at about 4% concentration, 33°C



0.16mJ<E<0.19mJ at about 5% concentration, 23°C

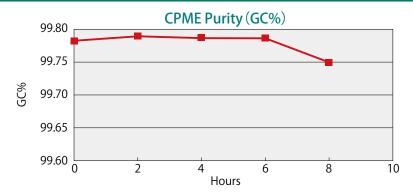
(2) Electrical resistivity

Solvent	Volume Resistivity (Ω•cm)	Temperature (°C)	Relative Humidity (%)
Cyclohexane	2.1×10 ¹⁴	9	58
Toluene	2.5×10^{13}	27	54
Xylene	2.8×10^{13}	28	54
CS ₂	7.5×10 ¹¹	10	57
CPME	5.0×10°	20	55
AcOBu	9.2×10^{8}	27.7	54
AcOEt	1.7×10^{7}	27.7	54
MeOH	$<4.0 \times 10^{6}$	27.7	54
EtOH	<4.0×10 ⁶	27.7	54
BuOH	<4.0×10 ⁶	27.7	54
Acetone	$<4.0 \times 10^{6}$	27.7	54
MEK	<4.0×10 ⁶		-
MIBK	<4.0×10 ⁶	27.7	54
CCI ₄	1.0×10 ¹⁴	9.5	57

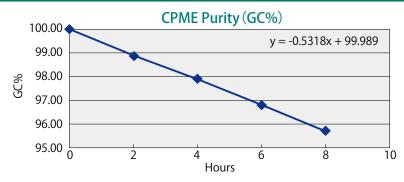


Stability to acids

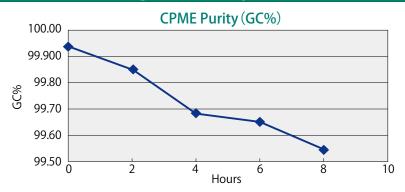
(1) 18% HCI (Heterogeneous system) (1/1 vol., at 40°C)



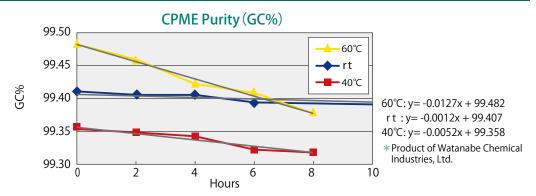
(2) 18% HCI (Heterogeneous system) (1/1 vol., at 100°C)



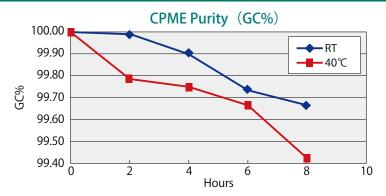
(3) 36% HCl (Homogeneous system) (1/1 vol., at 26°C)



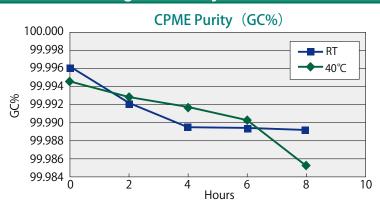
(4) 4N HCI-CPME* (Homogeneous system)



(5) 62% H₂SO₄ 〈Homogeneous system〉 (1/1 vol.)

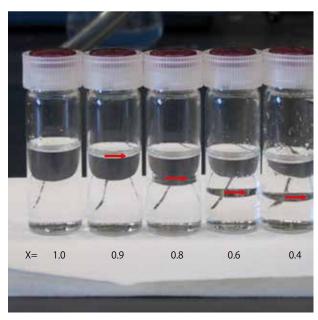


(6) conc.H₂SO₄ (Homogeneous system) (H₂SO₄/CPME=1/10 wt.)



(7) Compatibility of CPME with sulfuric acid

Water (ml)	conc.H ₂ SO ₄ (ml)	H ₂ SO ₄ (wt%)	
1	1	61.1	Compatible
1	0.9	58.7	Partially Compatible
1	0.8	56.1	Partially Compatible
1	0.6	49.3	Separate
1	0.4	39.8	Separate

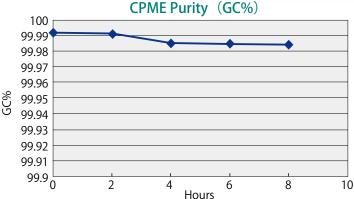


10 minutes after mixing Water:1ml+H₂SO₄:Xml+CPME:2ml

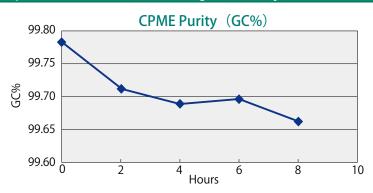


(8) 65% conc. HNO₃ (Homogeneous system) (HNO₃/CPME=1/10wt., 24°C)

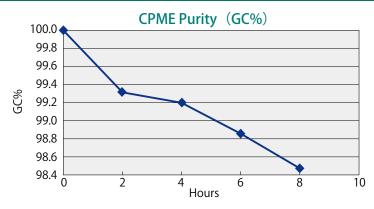
Conditions: 2g of 65% conc. HNO₃ was added slowly to 20g of CPME at 0° C, and stirred for 8 hours at room temperature (24°C).



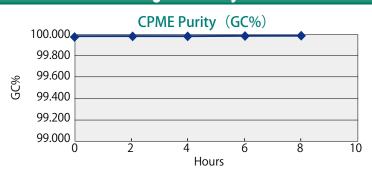
(9) 0.1M Camphor sulfuric acid (Homogeneous system) (at reflux temperature)



(10) Methyl trifluoromethanesulfonate (Homogeneous system) (MeOTf/CPME=1/10 wt., 25°C)



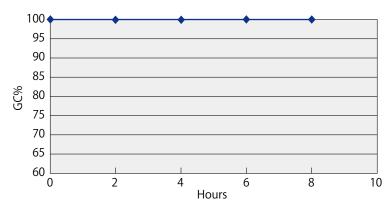
(11) Trifloroacetic acid (Homogeneous system) (TFA/CPME=1/10wt., 22°C)



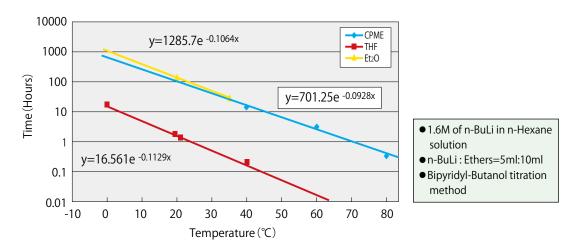
9 Stability to Bases

(1) 85% KOH \langle Heterogeneous system \rangle (KOH/CPME=35/100 wt., 110°C)

Conditions: 35g of 85% KOH pellets were added to 100g of CPME, and then refluxed for 8 hours under nitrogen at 110°C.



(2) Half-Lives of n-BuLi in Ethers





(1) Hydrogen solubility in solvents

Solvent	Temperature(°C)	Solubility(ml/l)
СРМЕ	18	110
THF	21	96
Acetone	18	99

(2) Oxygen solubility in solvents

Solvent	Temperature(°C)	Solubility(ml/l)
СРМЕ	16	330
THF	21	390





(1) Grignard Reaction

$$MgBr$$
 $+$ OH $+$

	Yield of Pro	Yield of Products(%)		ucts(%)	
Solvent	1	2	1	2	
THF	44.8	33.1	57.5	42.5	
THF+CPME	66.8	14.7	82.0	18.0	
СРМЕ	81.9	1.6	98.0	2.0	

Conditions: ● Grignard reagent concentration 1 mol/l

● Temperature: O°C * 1hr+reflux * 1h

● Work-up: 1N-HCI*rt

(2) LAH Reduction

COOEt + LiAlH₄
$$\frac{0^{\circ}C*0.5hrs}{Solv.}$$
 CH₂OH

Conditions: Substrate: LAH: Solvent=10mmol: 10mmol: 30ml

Solvents: Diethyl Ether or CPME

Work-up:

① Acid treatment Water $1ml \rightarrow 10\%HCl$ $20ml \rightarrow washing with water in 2times$

② Alkaline treatment Ethyl acetate $2ml \rightarrow 40\%NaOH 10ml \rightarrow water 5ml$

(yield: GC internal standard method)

Work-up	СРМЕ	Diethyl Ether	Remarks	
	Organic layer 83.5	Organic layer 66.3	CDME: I	
1	Aqueous layer 6.1	Aqueous layer 21.1	CPME is less soluble in water.	
	Total 89.6	Total 87.4	Soluble III Water.	
	Organic layer 93.7		CPME is less	
2	Aqueous layer 4.1		soluble in water.	
	Total 97.8		Joseph Materi	

(3) Other Reactions-1

The following reactions in CPME proceeded similarly to those in THF.

1) Synthesis of compound -2

Compound 1 (5.2 mg, 9.04 μ mol) was dissolved in 200 μ L of CPME in a nitrogen atmosphere and cooled to -78°C. Then 15.5 μ L (17.7 μ mol) of 1.14-M methyl lithium in diethyl ether solution was added dropwise and the reaction mixture was stirred for 5 minutes.

2 Synthesis of compound -4

Compound 3 (21.2 mg , 94.0 μ mol) was dissolved in 400 μ L of CPME in an argon atmosphere and cooled to 0°C. Then 190 μ L (190 μ mol) of 1.00-M isopropyl magnesium bromide in THF was added as the reaction mixture was stirred for 30 minutes at 0°C.

3 Synthesis of compound -6

Compound 5 (9.7 mg, 44.3 μ mol) was dissolved in 194 μ L of CPME in an argon atmosphere. Acetic anhydride (45.2 μ L, 443 μ mol) was added, and the reaction mixture was allowed to stand for 40 minutes at room temperature.



4 Synthesis of compound -8

Compound 7 (10 mg, 36.9 μ mol) was dissolved in 300 μ L of CPME. Then 100 mg of active manganese dioxide was added. The reaction mixture was allowed to stir for 12 hours at 50°C.

Results: The reaction proceeded until the ratio of raw materials/target materials = 1/1, regardless of whether THF or CPME was used as the solvent. With CPME, after additional stirring for 12 hours at 95°C, the reaction proceeded until the ratio of raw materials/target materials = 1/9.

5 Synthesis of compound -10

Compound 9 (60.9 mg, 131 μ mol) was dissolved in 1.2 mL of CPME in an argon atmosphere and cooled to 0° C. Then 11.8 mg (522 μ mol) of lithium borohydride was added, and the reaction mixture allowed to stir for 12 hours at 60° C.

(4) Other Reactions-2

In the following reactions in CPME, almost the same or better results were obtained in comparison with those in the other solvents.

1. Higher optical purity or selectivity were observed

- Asymmetric Michael alkylation
- Michael addition of R₂CuLi
- Alkylation of chiral amide
- Glycosylation
- Asymmetric hydrogenation of NaBH₄
- Hydrosilylation by Ru cat

2. Nucleophilic Reactions

- Amide synthesis by the reaction of acid chloride with amine
- Sillylation and desillylation
- Reaction of carbanion with aldehyde
- Debenzylation
- Alkylation of amine
- Selective methylation of phenols
- Bromination of alcohol with PBr₃
- Sulfonylchloride synthesis by the reaction of sulfonic acid with PCI₅

3. Reactions using metals

- Reaction of Ketone with NaBH₄
- Reaction of acetylenes with Ti (OR) 4
- Reaction using n-BuLi or Lithium Diisoprpyl Amide
- Radical cyclization of trichloroacetate using Cu cat
- Reduction of Ethyl benzoate using Lithium Aluminium Hydride
- Formation of sodium dispersion
- Intramolecular ene reaction using ZnCl₂





1. CPME showed superior extraction performance to Diethyl Ether when evaluated with the following 6 compounds.

● 2 grams of each compound was dissolved in 20mL of CPME or 40mL of diethyl ether, respectively, 10mL of distilled water was added, and liquid—liquid separation was carried out.

*Under these conditions, one extraction results with 20ml CPME gave results comparable to those obtained from two extractions with 40ml diethyl ether.

2. CPME showed similar extraction performance to Diethyl Ether when evaluated with the following 5 compounds.

● 2 grams of each compound was dissolved in 10mL of CPME or 10mL of diethyl ether, respectively, 10mL of distilled water was added, and liquid—liquid separation was carried out.

*Under these conditions, one extraction results with 10ml CPME gave results comparable to those obtained from two extractions with 10ml diethyl ether.

Conditions:

After materials were immersed in CPME at 50° C for 4hrs, their weight were measured. Test pieces: $2mm \times 50mm \times 25mm$

1) Effects of CPME on plastics

	Change of weight (wt%)	Appearance *
Polypropylene	6.3	Excellent
Polyethylene	3.7	Excellent
Hard polyvinylchloride	33.6	No Good
Soft polyvinylchloride	47.8	No Good
ABS resin	95.1	No Good
Polystyrene	dissolved	No Good
Phenol resin	-0.1	Fair
Epoxy resin	0.7	Fair
Polyacetal	-0.5	Fair
Nylon 66	-0.2	Fair
Polycarbonate	16.8	Fair
Polyacrylate	1.3	Fair
Polyurethane	78.4	Fair
Polyphenylensulfide	0.0	Excellent
PTFE	0.0	Excellent

2) Effects of CPME on Rubbers

	Change of weight (wt%)	Appearance*
SBR (styren-butadiene)	94	No Good
BR (butadiene)	100<	No Good
NBR (nitrile-butadiene)	44.0	No Good
Chloroprene rubber	84	No Good
Hydrogenated NBR	62.0	No Good
Silicone rubber	100<	No Good
Fluorinated rubber	10.7	Fair

*Appearance

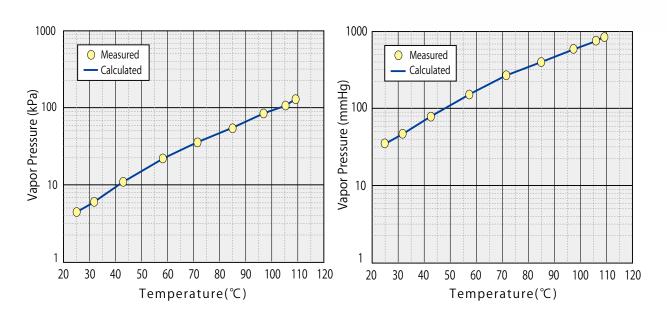
Excellent No change

Good Slight swelling without notable change Fair Swelling without notable change

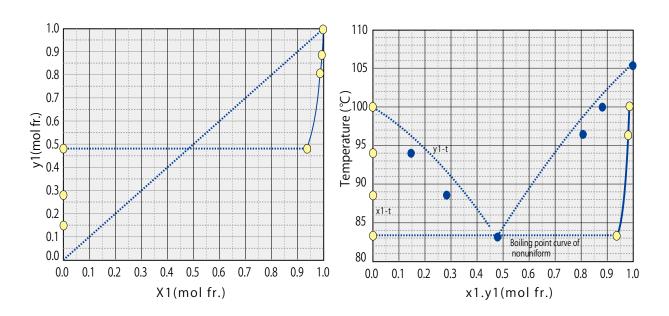
No Good Notable swelling, cracking of materials or Dissolution to CPME







Vapor-liquid Equilibrium of Water-CPME





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