

Novel hydrophobic ether solvent

**Cyclopentyl methyl ether**

**CPME**

## **TECHNICAL DATA**

**Annex of June 2009**

The information contained herein is believed to be reliable, but no representations, guarantees or warranties of any kind are made as to its accuracy, suitability for particular applications or results to be obtained.

Please read the Material Safety Data Sheet (MSDS) carefully prior to handling.

This product was developed for the application in this brochure. In case of other applications, please handle under your confirmation of safety for the applications, or please talk to Zeon Corporation beforehand.



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# 0. Physical Properties

	CPME	THF	MeTHF	Et <sub>2</sub> O	Dioxane	MTBE	
Density (20°C) [g/cm <sup>3</sup> ]	0.86	0.89	0.85	0.71	1.03	0.74	
Vapor specific gravity (air = 1)	3.45	2.49	2.97	2.56	3.3	3.03	
Boiling point [°C]	106	65	80	34.6	101	55	
Melting point [°C]	<-140	-108.5	-136	-116.3	11.8	-108.7	
Viscosity (20°C) [cP]	0.55	0.55	0.6 (25°C)	0.2448	1.31	0.35	
Surface tension (20°C) [mN/m]	25.17	26.4	Unknown	17.3	33.74	19.8	
Vaporization energy (bp) [Kcal/kg]	69.2	98.1	89.7	86.08	98.6	81.7	
Specific heat (20°C) [Kcal/kg·K]	0.4346	0.469	Unknown	0.5385	0.41	0.51	
Refractive index (20°C)	1.4189	1.407	1.406	1.353	1.422	1.369	
Solubility parameter [cal/ml]	8.4	9.5	8.52	7.4	Unknown	Unknown	
Dielectric constant (25°C)	4.76	7.58	7	4.197	2.227	2.6	
Dipole moment [D]	1.27 (calcd.)	1.7	Unknown	1.12	0.45	1.4	
Azeotropic temperature with water [°C]	83 (a)	64	71 (b)	34.2	87.8	52.9 (c)	
Solubility of solvent in water (23°C) [g/100g]	1.1	Infinite	14	6.5	Infinite	4.8	
Solubility of water in solvent (23°C) [g/100g]	0.3	Infinite	4.4	1.2	Infinite	1.5	
Flash point [°C]	-1	-14.5	-11	-45	12	-28	
Auto Ignition Temperature [°C]	180	205	270	180~190	180	224	
LogPow	1.59	0.47	Unknown	0.89	-0.42	0.94	
Explosion range [vol%]	Lower limit	1.1	1.84	1.5	1.85	2	1.6
	Upper limit	9.9	11.8	8.9	48	22	8.4

## Azeotropic composition:

(a) CPME / water = 83.7 / 16.3

(b) MeTHF / water = 89.4 / 10.6

(c) MTBE / water = 96.5 / 3.5

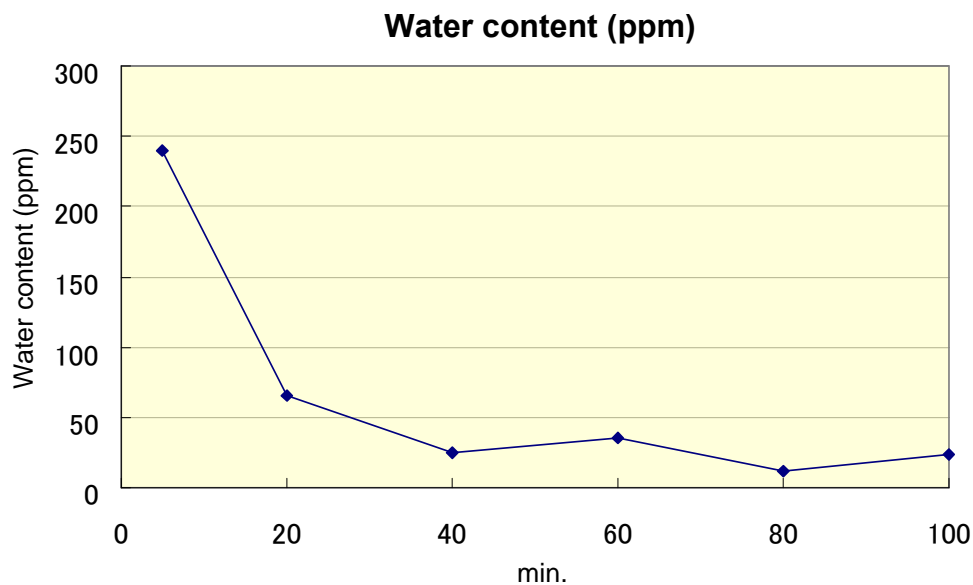
**MeTHF (2-Methyltetrahydrofuran)  
is added**

# 1. High Hydrophobisity

## 1) 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.



## 2) Distillation of CPME saturated with water

### Fractional Distillation of CPME saturated with water (500g)

Fraction	Boiling Point (°C)	Weight (gram)	CPME (%)	Water (ppm)	Peroxide (ppm)
1	81 - 84	11	99.3		
2	84 - 105	10	99.9	617	
3	105 - 106	454	99.9	92	5
Residue		24			
Total		498			

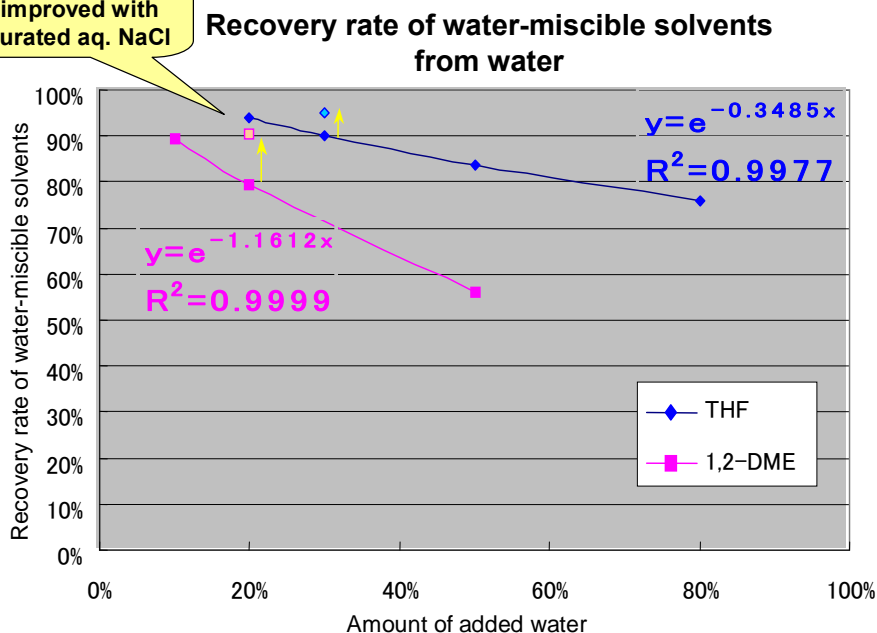
# 1. High Hydrophobicity

## 3) 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.

Recovery rate can be improved with saturated aq. NaCl



Recovery rate of CPME is higher than 95% because of its Hydrophobicity.

## 4) 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 / 33

### Solvents with which CPME does not form azeotropes

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

\* Hard to separate

# 1. High Hydrophobicity

## 5) 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

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

### MEK

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

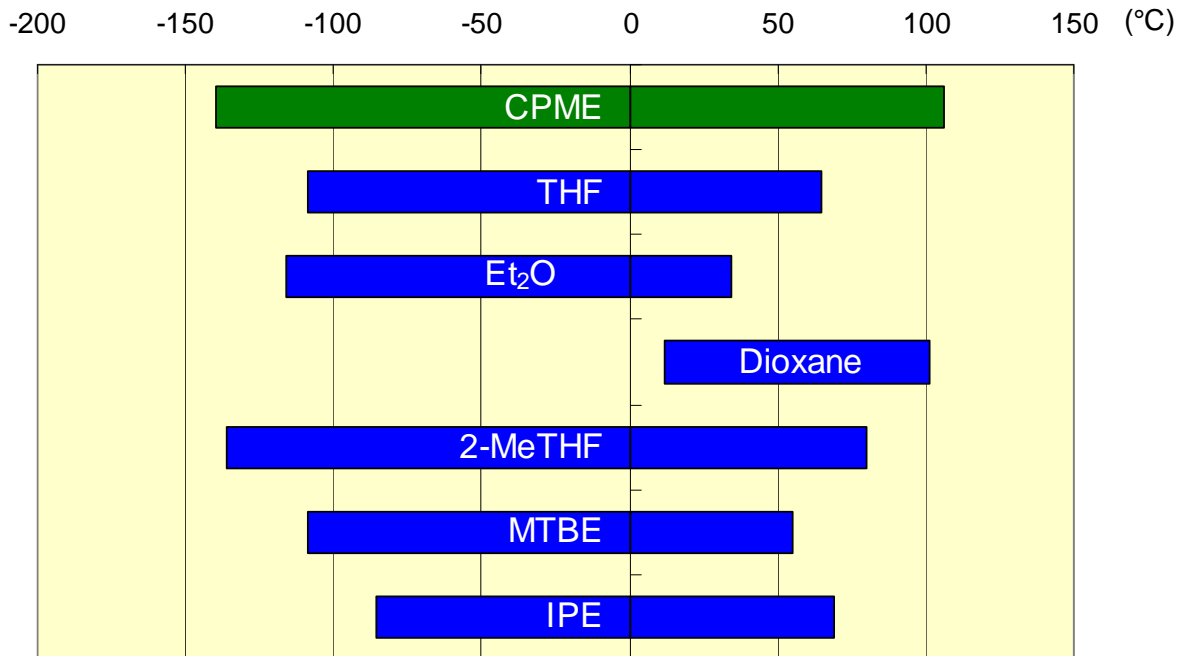
### MIBK

Amount of MIBK	Weight ratio of 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%.

## 2. Wide Liquidity Range

> Liquidity range of ether solvents



# 3. Low Heat of Vaporization

## > Evaporation rate

**Method:**

ASTM D 3539-87 Standard Test Methods for Evaporation Rates of Volatile Liquids by Shell Thin-Film Evaporometer

**Conditions:**

23°C x 50% RH

	Relative evaporation rate
CPME	3.5
Butyl acetate	1

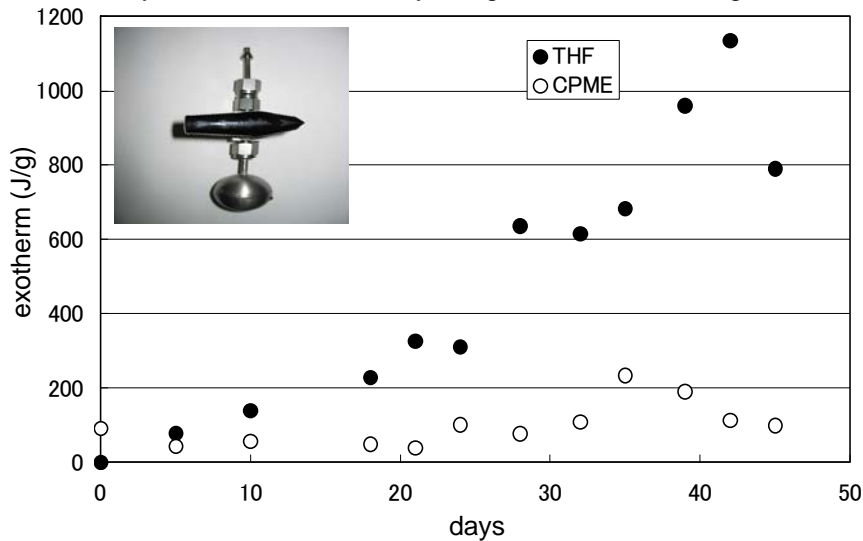
## 4. Resist Peroxide Formation

### 1) Exothermic energy under O<sub>2</sub>

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

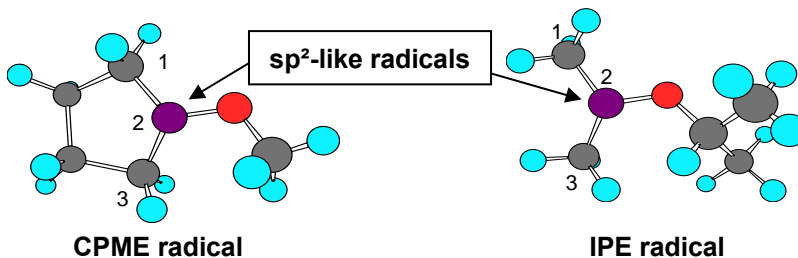
#### Conditions:

CPME and THF without an antioxidant which were pressurized with 0.99MPa of O<sub>2</sub> 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).



### 2) 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		CPME	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 than 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.

## 4. Resist Peroxide Formation

### 3) Removal of Peroxide with aq. $\text{Na}_2\text{SO}_3$

Preparation:

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



stir in the air at 80°C for 4hr

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

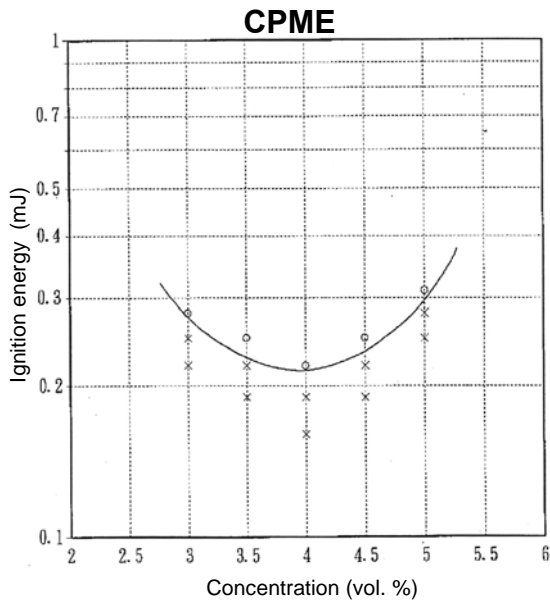
Condition:

1kg of CPME containing peroxide was washed with 500g of 5%  $\text{Na}_2\text{SO}_3$  several times.

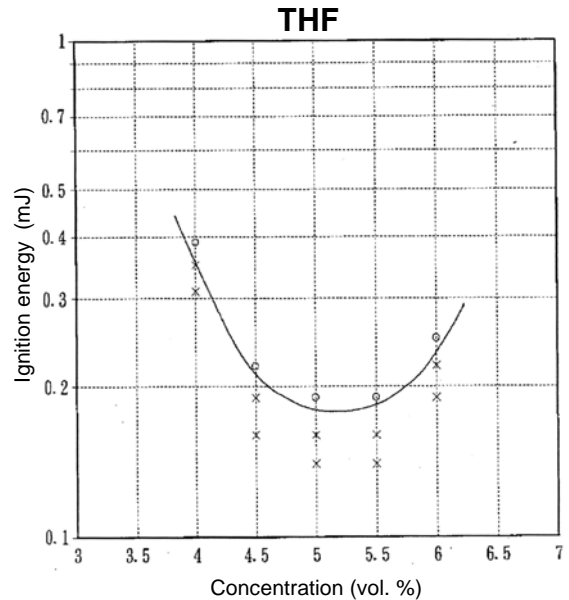
Number of washing (times)	Peroxide value ( $\text{H}_2\text{O}_2$ wt ppm)
0	620
1	116
2	95
3	7.5

## 5. Narrow Expansion Area (Static Electricity)

### 1) Minimum ignition energy ( $E$ )



**$0.19\text{mJ} < E < 0.22\text{mJ}$**   
at about 4% concentration, 33°C



**$0.16\text{mJ} < E < 0.19\text{mJ}$**   
at about 5% concentration, 23°C

### 2) Electrical resistivity

Solvent	Volume Resistivity ( $\Omega \cdot \text{cm}$ )	Temperature ( $^{\circ}\text{C}$ )	Relative Humidity (%)
Cyclohexane	$2.1 \times 10^{14}$	9	58
Toluene	$2.5 \times 10^{13}$	27	54
Xylene	$2.8 \times 10^{13}$	28	54
$\text{CS}_2$	$7.5 \times 10^{11}$	10	57
* CPME	$5.0 \times 10^9$	20	55
AcOBu	$9.2 \times 10^8$	27.7	54
AcOEt	$1.7 \times 10^7$	27.7	54
MeOH	$< 4.0 \times 10^6$	27.7	54
EtOH	$< 4.0 \times 10^6$	27.7	54
BuOH	$< 4.0 \times 10^6$	27.7	54
Acetone	$< 4.0 \times 10^6$	27.7	54
MEK	$< 4.0 \times 10^6$		-
MIBK	$< 4.0 \times 10^6$	27.7	54
$\text{CCl}_4$	$1.0 \times 10^{14}$	9.5	57

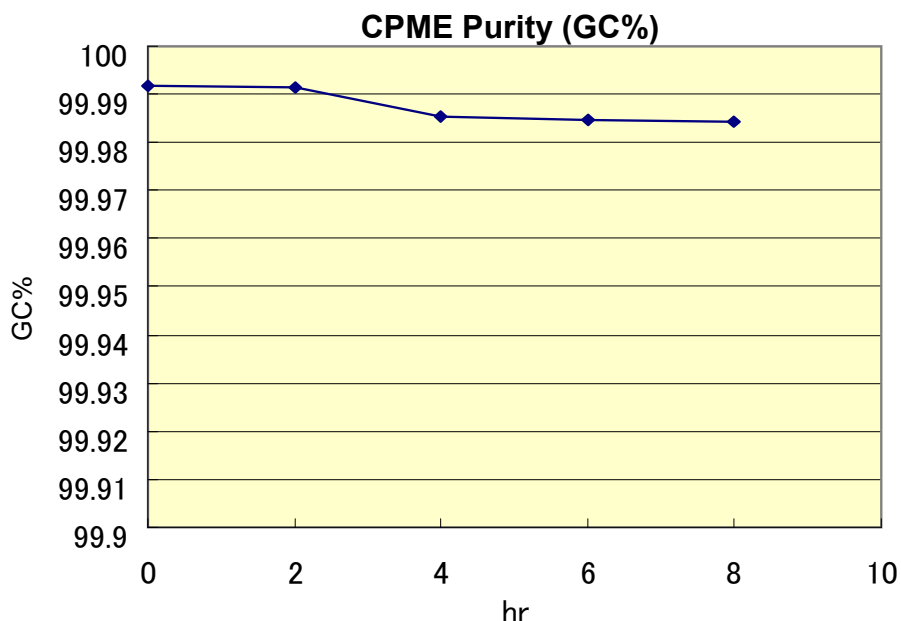
## 6. Relatively Stable to Acids and Bases

### 1) 65% conc. $\text{HNO}_3$ <Homogeneous>

( $\text{HNO}_3/\text{CPME} = 1/10$  wt.,  $24^\circ\text{C}$ )

#### Conditions:

2g of 65% conc.  $\text{HNO}_3$  was added slowly to 20g of CPME at  $0^\circ\text{C}$ , and stirred for 8 hours at room temperature ( $24^\circ\text{C}$ ).

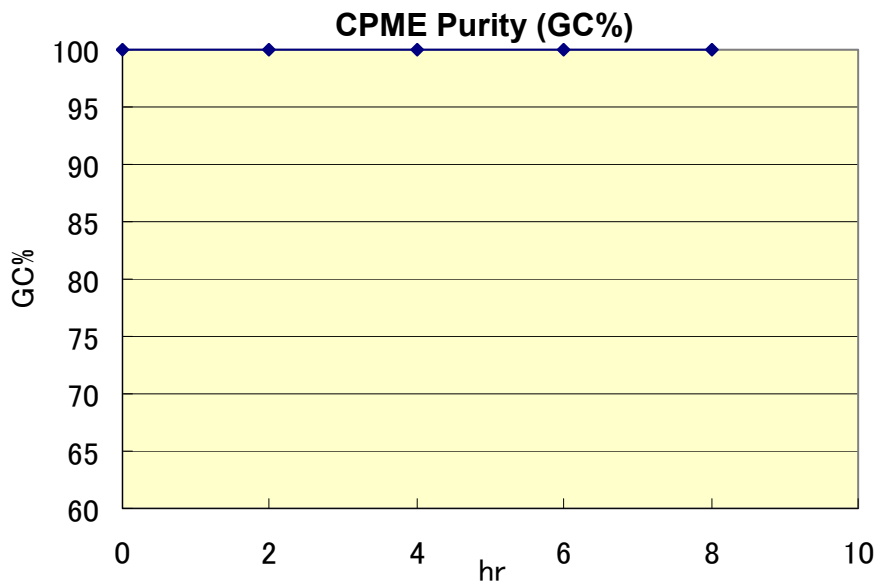


### 2) 85% KOH <Heterogeneous>

( $\text{KOH}/\text{CPME} = 35/100$  wt.,  $110^\circ\text{C}$ )

#### Conditions:

35g of 85% KOH pellets were added to 100g of CPME, and then refluxed for 8 hours under nitrogen at  $110^\circ\text{C}$ .



## 7. Miscellaneous

### 1) Hydrogen solubility in solvents

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

### 2) Oxygen solubility in solvents

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