

# Resin curing agent capable of thin film curing

## RIKACID™ TBN-100

### ■ Summary

- Powder acid anhydride with reduced volatilization during heat curing.
- Epoxy curing coating is provided by combining TBN-100 and epoxy resin with accelerator, and applying it to substrate, and vaporization drying solvent, and heat curing.

### ■ Feature

- The curing coating is superior in thermal resistance and initial transparency and heat yellowing resistance.
- The curing coating is superior in water vapor barrier property.
- The curing coating is superior in adhesion to various substrate.
- The curing coating is superior in flexibility.
- The curing coating is superior in solvent resistance and alkali resistance.
- Curing coating having high surface hardness is provided.
- Solvent solubility is relatively good.

### ■ Typical properties

Table 1. Typical properties of TBN-100

	RIKACID™ TBN-100	Remarks
Appearance	White powder	—
Equivalent of acid anhydride	450~550	—
Softening point[°C]	90~95	Melting point measuring instrument
Registration situation of law	Not registered	—

※These values are not guaranteed.

## ■ Solvent solubility of TBN-100

The solubility in various solvents is shown in Table 2 and Table 3, and the viscosity of each 50wt% product at room temperature (25°C) is shown in Table 4.

Table 2. Solubility at 25°C. (○: dissolved, ×: insoluble)

Solvent	TBN-100 concentration							
	40wt%		50wt%		60wt%		70wt%	
	Solubility	Dissolution time(h)	Solubility	Dissolution time(h)	Solubility	Dissolution time(h)	Solubility	Dissolution time(h)
MEK <sup>※1</sup>	○	0.5	○	1.0	○	1.5	○	6.0
Ethyl acetate	○	0.5	○	1.0	○	1.5	○	6.0
Butyl acetate	○	1.0	○	3.0	×	6.0	×	6.0
PGMEA <sup>※2</sup>	○	3.0	○	3.0	×	6.0	×	6.0
BCA <sup>※3</sup>	○	6.0	×	6.0	×	6.0	×	6.0

Table 3. Solubility at 50°C. (○: dissolved, ×: insoluble)

Solvent	TBN-100 concentration							
	40wt%		50wt%		60wt%		70wt%	
	Solubility	Dissolution time(h)	Solubility	Dissolution time(h)	Solubility	Dissolution time(h)	Solubility	Dissolution time(h)
MEK <sup>※1</sup>	○	0.5	○	1.0	○	1.5	○	6.0
Ethyl acetate	○	0.5	○	1.0	○	1.5	○	6.0
Butyl acetate	○	1.0	○	3.0	×	6.0	×	6.0
PGMEA <sup>※2</sup>	○	3.0	○	3.0	○	4.0	○	4.0
BCA <sup>※3</sup>	○	6.0	○	6.0	×	6.0	×	6.0

### <Evaluation method>

TBN-100 and each solvent were added to the sample bottle so as to have a predetermined concentration, and the mixture was stirred at each temperature (25°C, 50°C), and whether or not the solution was soluble was visually confirmed.

Table 4. Solution viscosity (E type viscometer, 25°C, TBN-100 concentration: 50wt%)

Solvent	Viscosity[mPa·s]
MEK <sup>※1</sup>	30
Ethyl acetate	59
Butyl acetate	102
PGMEA <sup>※2</sup>	263
BCA <sup>※3</sup>	1300

※1 MEK: Ethyl methyl ketone (Nacalai Tesque, Inc.)

※2 PGMEA: Propyleneglycol monomethyl ether acetate (Nacalai Tesque, Inc.)

※3 BCA: 2-(2-Butoxyethoxy)ethyl acetate (Butyl carbitol acetate)(Nacalai Tesque, Inc.)

## ■ Curing condition of RIKACID™ TBN-100

We show a curing condition of TBN-100 (in Figure 1). It can be cured at 140°C or more / 30 to 60 minutes.

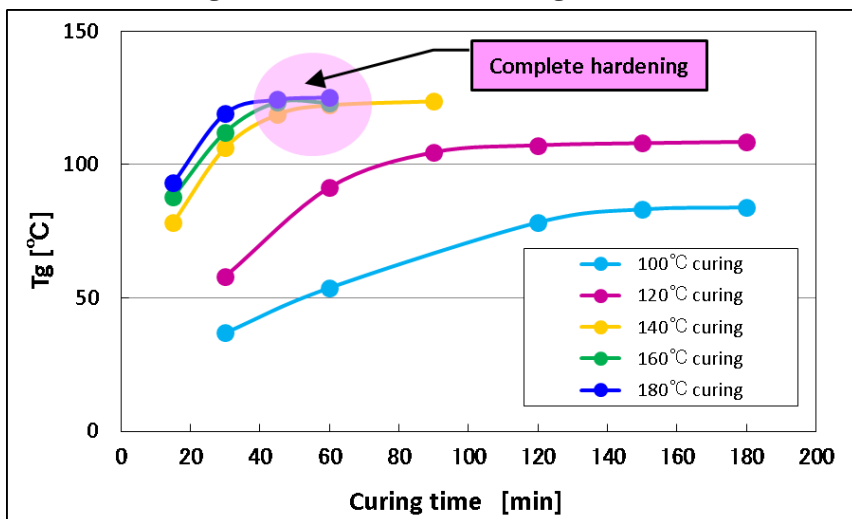


Fig 1. The relationship of Tg and curing time

<Formulation> [p.b.w, Equivalent ratio = 1.0]

Epoxy Resin JER™ 828: Bisphenol A epoxy resin (Mitsubishi Chemical Corporation.) 100

Curing agent RIKACID™ TBN-100 255

Solvent Ethyl methyl ketone (MEK, Nacalai tesque Co., Ltd.) 255

Accelerator HISHICOLIN™ PX-4ET: Quaternary phosphonium salt (NIPPON CHEMICAL INDUSTRIAL CO., LTD.) 1

<Curing condition>

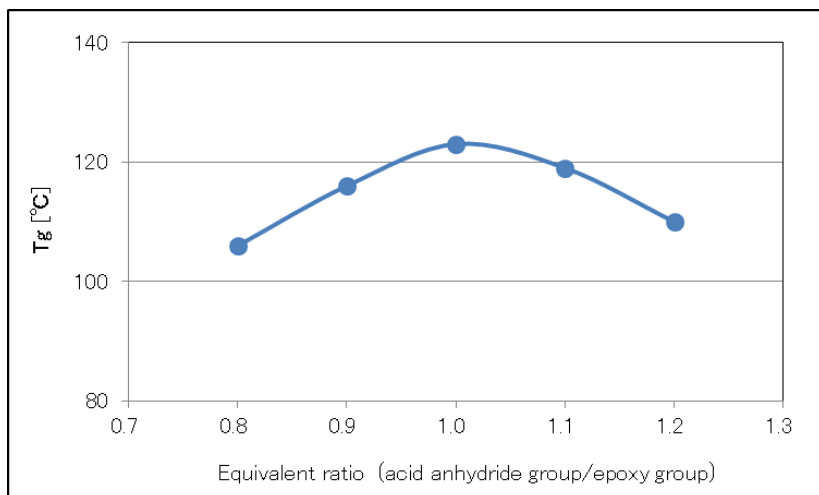
100°C/10min(Dry) + 100~180°C/15~180min(Curing)

<Test condition>

DSC, Heating rate: 20°C/min Sample: 100μ m(Film thickness)

## ■ Depend on equivalent ratio

We show a equivalent ratio of TBN-100 (in Figure 2). The most suitable equivalent ratio of TBN-100 is 1.0.



<Foemulation>

The formulation is the same as figure 1.

<Curing condition>

100°C/10min(Dry) + 150°C/3h(Curing)

<Test condition>

DSC, Heating rate: 20°C/min

Sample: 100μ m(Film thickness)

Fig 2. The relationship of Tg and equivalent ratio

## ■ Typical properties of cured epoxy resin

Table 5 shows the epoxy cured film properties and Table 6 shows the water vapor permeability of the film.

Table 5. Typical properties of cured epoxy resin

Formulation No.		①	②	Remarks	
Formulation [p.b.w] Equivalent ratio = 1.0	jER™ 828※4	100	100	Epoxy equivalent = 185	
	RIKACID™ TBN-100	255	-	Equivalent of acid anhydride = 472	
	RIKACID™ MH-700※5	-	89	Equivalent of acid anhydride = 164	
	MEK※6	255	-	Solvent	
	HISHICOLIN™ PX-4ET※7	1	1	Accelerator	
Curing condition		100°C/10min(Dry) + 150°C/1h(Curing)			
Typical properties of cured epoxy resin	Gel time (140°C)[sec]	220	280	Based on JIS C2161 B	
	Tg [°C]	121	30	DSC	
	YI <sub>1</sub> (After curing)	0.02	3.2	Δ YI = YI <sub>2</sub> -YI <sub>1</sub>	Thickness: 100 μ m
	YI <sub>2</sub> (After 150°C/5days aging)	0.61	8.4		
	Resistance for Thermal Yellowing Δ YI	0.59	5.2		
	Pencil hardness	H	5B	Based on JIS K5600-5-4	
	90° Bending resistance (PET※8)	○	×	○ : There is no change × : There is change	Thickness: 30 μ m
	Alkali resistance (5%NaOH)	○	×		
	Solvent resistance (NMP)	○	×		

※4 jER™ 828: Bisphenol A epoxy resin (Mitsubishi Chemical Corporation)

※5 RIKACID™ MH-700: 4-methyl cyclohexane-dicarboxylic anhydride/cyclohexane-dicarboxylic anhydride = 70/30(wt%) (New Japan Chemical Co., Ltd.)

※6 MEK: Ethyl methyl ketone (Nacalai tesque Co., Ltd.)

※7 HISHICOLIN™ PX-4ET: Quaternary phosphonium salt (NIPPON CHEMICAL INDUSTRIAL CO., LTD.)

※8 PET: Polyethylene terephthalate, 「COSUMOSHINE™ A4300」 (TOYOBO CO., LTD.)

Preparation of curing agent: TBN-100 was dissolved in MEK at room temperature(25°C) to give a 50wt% solution.

Table 6. Water vapor permeability of the film

Resin type	jER™ 828/TBN-100※9	Polyurethane※10	PC※10
Water vapor permeability (g/m <sup>2</sup> ·24h·atm)	4.0	850	24

※9 Preparation of curing agent: TBN-100 was dissolved in MEK at room temperature(25°C) to give a 50wt% solution.

Curing condition: 100°C/10min(Dry) + 150°C/1h(Curing)

→ Separation of the film from glass substrate → 60°C/8h(Dry)

Measure condition: 40°C, 90%RH

Film thickness: 25μ m

※10 Reference: 「Testing method and evaluation result of each dynamic characteristic of the plastic material」  
Plastics, 51(6), 119 (2000)

Measure condition: 40°C, 90%RH

Film thickness: Polyurethane(25μ m), PC(50μ m)

Table 7 shows the adhesion to various substrates.

Table 7. Adhesive property (Cross-cut peeling test)

Formulation No.		①	②	Remarks
Formulation [p.b.w] Equivalent ratio = 1.0	jER™ 828※11	100	100	Epoxy equivalent = 185
	RIKACID™ TBN-100	255	-	Equivalent of acid anhydride = 472
	RIKACID™ MH-700※12	-	89	Equivalent of acid anhydride = 164
	MEK※13	255	-	Solvent
	HISHICOLIN™ PX-4ET※14	1	1	Accelerator
Curing condition	100°C/10min(Dry) + 150°C/1h(Curing)			-
Thickness[μm]		30		-
Initial adhesion	Steel plate※15	○	×	Based on JIS K5600-5-6 ○: There is no detachment ×: There is detachment
	PC※16	○	×	
	PET※17	○	×	
	PI※18	○	×	
	Copper plate※19	○	×	
Boiling adhesion (100°C/1h)	PC※16	○	×	
	PET※17	○	×	

※11 jER™ 828: Bisphenol A epoxy resin (Mitsubishi Chemical Corporation)

※12 RIKACID™ MH-700: 4-methyl cyclohexane-dicarboxylic anhydride/cyclohexane-dicarboxylic anhydride = 70/30(wt%) (New Japan Chemical Co., Ltd.)

※13 MEK: Ethyl methyl ketone (Nacalai tesque Co., Ltd.)

※14 HISHICOLIN™ PX-4ET: Quaternary phosphonium salt (NIPPON CHEMICAL INDUSTRIAL CO., LTD.)

※15 Substrate: Extension steel plate (Nippon Testpanel Co., Ltd.)

※16 PC: Polycarbonate (Commercial product)

※17 PET: Polyethylene terephthalate, 「COSUMOSHINE™ A4300」 (TOYOBO CO., LTD.)

※18 PI: Polyimide, 「Kapton™ 100EN」 (DU PONT-TORAY CO., LTD.)

※19 HULL CELL CATHODE PLATE (YAMAMOTO-MS Co., Ltd.)

## ■ Inquiry

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