

# Cray Valley Products For Urethane Elastomers

Hydroxyl Terminated  
Functional Liquid Poly bd<sup>®</sup> Resins



**TOTAL**



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## INTRODUCTION

**P**oly bd<sup>®</sup> Resins are liquid, hydroxyl-terminated homopolymers of butadiene. Through the use of an isocyanate cure reaction to produce novel urethane products, they offer a route to castable, general-purpose elastomers. They can be reinforced with various fillers, and extended with a variety of low cost process oils.

The unique structure of the Poly bd<sup>®</sup> Resins provides properties which surpass both conventional polyether and polyester urethane systems, as well as conventional, general-purpose rubbers. These novel Poly bd<sup>®</sup> Resins can be used in preparing castable elastomers, caulks, sealants, membranes, sponges, foams, adhesives, coatings, propellant binders, potting and encapsulating compounds, as well as other rubber-fabricated materials.

Some of the outstanding features of Poly bd<sup>®</sup> urethane systems include:

- 1) The easy reaction of Poly bd<sup>®</sup> Resins with curing agents such as conventional di- and polyisocyanates provides an economical liquid or semi-solid route to general purpose rubbery elastomers.
- 2) The molecular structure of Poly bd<sup>®</sup> liquid polymers resembles that of polybutadiene rubber, hence these polymers can be reinforced with carbon black and other fillers in a similar manner.
- 3) Oil extendability is another outstanding feature of Poly bd<sup>®</sup> Resins. Poly bd<sup>®</sup> liquid Resins are compatible with a wide variety of hydrocarbon oils and resins. Oil extension, therefore, provides formulation flexibility in controlling properties of the uncured liquid systems such as pot life and gel time, as well as the properties of the cured products; such as tensile and tear strengths and elongation.

Oil extension, in combination with filler reinforcement, provides unique formulation flexibility for preparing low cost urethane products.

- 4) Improved low-temperature properties are also a feature. Many elastomeric products derived from Poly bd<sup>®</sup> Resins have brittle points as low as -70°C.
- 5) Poly bd<sup>®</sup> resins have excellent hydrolytic stability. The hydrophobic backbone of Poly bd<sup>®</sup> Resins imparts excellent hydrolytic stability to urethane products. Thus, elastomers, caulks, sealants, foams and other products can be made which are resistant to hydrolysis, even in boiling water.
- 6) Excellent electrical insulation properties also result from the use of Poly bd<sup>®</sup> Resins. A discussion of the benefits of Poly bd<sup>®</sup> Resins in electrical applications is found in a separate electrical applications bulletin.
- 7) Chemical resistance is imparted. Poly bd<sup>®</sup> Resins can be used to prepare polyurethane elastomers with excellent resistance to aqueous inorganic acids and bases.

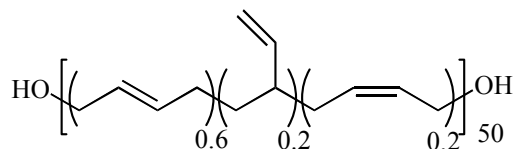
With these features, as well as others to be described, Poly bd<sup>®</sup> urethane systems combine the advantages of general-purpose rubbers and urethane elastomers within the same polymer network.

## Physical Properties of Poly bd<sup>®</sup> Resins

Typical physical properties of two Poly bd<sup>®</sup> Resins are summarized in Table 1. Their simplified structure is represented in Figure 1. The polybutadiene microstructure of both homopolymers is analogous to that of diene polymers prepared by emulsion techniques. In addition, these polymers possess predominantly primary, terminal hydroxyl groups of the allylic type. This structural combination accounts for their high reactivity, especially with aromatic diisocyanates. The hydroxyl functionality of Poly bd<sup>®</sup> R-45M Resin is typically in the range of 2.2 to 2.4 per chain, whereas that of Poly bd<sup>®</sup> R-45HTLO Resin is somewhat higher, typically 2.4 to 2.6 per chain.

Storage stability for both resins is excellent in the absence of air, and their low moisture content allows them to be used directly from containers without further pretreatment in many systems. The typical viscosity-temperature relationship is given in Figure 2.

**Figure 1: Poly bd<sup>®</sup> Resin Structure**



**Table 1 Typical Properties of Poly bd<sup>®</sup> Resins**

Poly bd <sup>®</sup> Resin	R-45HTLO	R-45M
Nonvolatile Material, Wt. %	99.9	99.9
Viscosity, mPa•s@ 30 °C	5000	4300
Hydroxyl Value, meq/g	0.86	0.73
Hydroxyl Number, mg KOH/g	48.2	41
Molecular Weight, No. Average	2800	2800
Water, Wt%	0.03	0.03
Specific Gravity @ 30°C	0.901	0.899
Iodine Number, g/100g	400	400
Polybutadiene Microstructure		
cis - 20%		
trans- 60%		
vinyl- 20%		

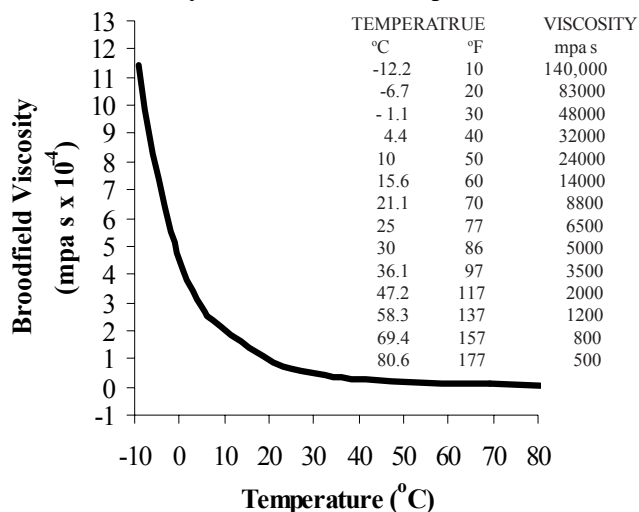
### Low Temperature Behavior of Poly bd<sup>®</sup> Resin Based Urethanes

Polyurethane elastomers based on Poly bd<sup>®</sup> Resins exhibit outstanding low temperature properties. This characteristic is attributable to the “rubbery” polybutadiene backbone. Many polyurethane elastomers derived from Poly bd<sup>®</sup> Resins have brittle points as low as -70 °C.

To demonstrate this characteristic, the physical properties of a Poly bd<sup>®</sup> Resin prepolymer system prepared from a 15% free NCO Poly bd<sup>®</sup> R-45HTLO/TDI prepolymer chain extended with equal equivalents of Voranol 220-530 and Pluracol TP-340 were measured over a temperature range of -30 ° to +60 °C. Table 2 shows a nearly constant elongation over this temperature range, while other properties behaved consistently with usual polyurethane systems. An elongation of 130% at -30 °C demonstrates that the Poly bd<sup>®</sup> based polyurethane elastomer is not brittle and, indeed, still possesses elastomeric properties at this temperature.

Retention of flexibility at low temperature is a prominent

**Figure 2: Poly bd<sup>®</sup> R-45HT Resin Viscosity as a Function of Temperature**



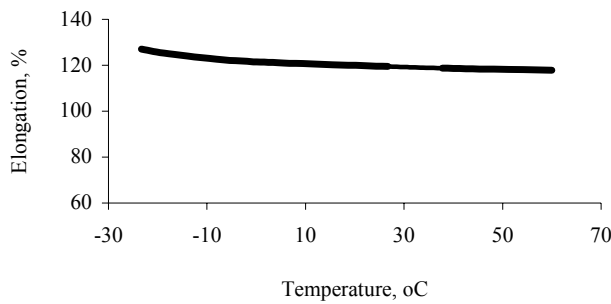
feature of Poly bd<sup>®</sup> Resin based systems. This feature allows for encapsulation of fragile electrical components in a medium that retains cushioning properties. Embedment stress at -40°C is significantly lower than many competitive materials. This same characteristic of the Poly bd<sup>®</sup> Resin also imparts excellent thermal cycling properties to components potted or encapsulated with a Poly bd<sup>®</sup> Resin based system. Figure 3 shows the elongation of the Poly bd<sup>®</sup> polyurethane described in Table 2 to be essentially constant over a wide temperature range.

### Hydrolytic Stability

**Table 2 Elongation Over a Temperature Range from -30° to 60 °C**

Temp., °C	Tensile (psi)	Elongation (%)	Modulus (psi)		
			50%	100%	Tear
60	2060	117	960	1775	--
RT	3250	122	1748	2800	275
-15	5090	125	2800	4390	--
-30	6080	130	3500	5250	423

**Figure 3: Elongation vs. Temperature**



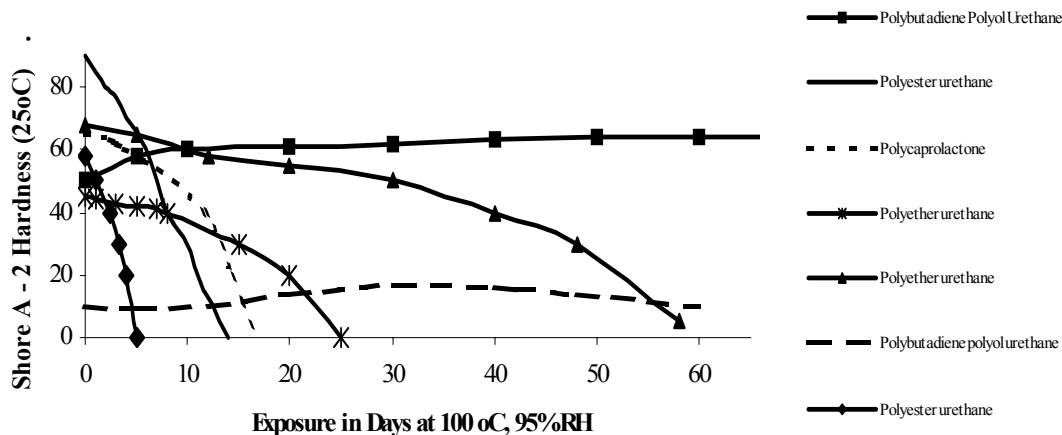
The hydrocarbon backbone in Poly bd<sup>®</sup> Resin imparts hydrolytic stability to the finished product, surpassing that of any other type of polyurethane. Electrical insulation properties are maintained in the presence of moisture, even at high temperatures. Poly bd<sup>®</sup> Resin systems far exceed the 28 day requirement of the Naval Avionics test. By measuring hardness vs. time at 100 °C and 95% relative humidity, it can be shown that Poly bd<sup>®</sup> based systems are essentially unaffected by moisture at high temperature, whereas other typical urethanes actually liquify (revert) during the test period, as shown in Figure 4.

### Hydrolytic Stability of Poly bd<sup>®</sup> Resins in Combination with Polyether Polyols

Polyurethane elastomers based on Poly bd<sup>®</sup> Resins exhibit outstanding hydrolytic stability. Again this characteristic is inherent in the nature of the hydrocarbon polybutadiene backbone of the Poly bd<sup>®</sup> Resin. There are no ester or ether linkages to hydrolyze.

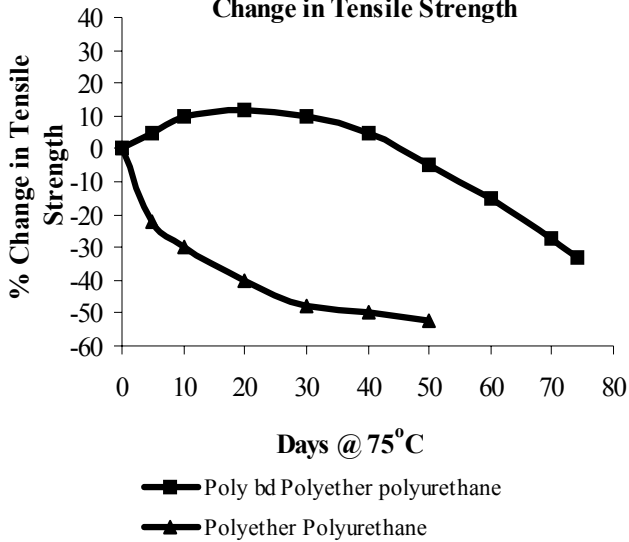
The addition of even moderate amounts of Poly bd<sup>®</sup> Resins to polyether based polyurethanes markedly improves the hydrolytic stability of the cured elastomer. Figures 5, 6 and 7 show the test results on two comparable elastomer systems. One contains 24.4 weight percent Poly bd<sup>®</sup> Resin, whereas the other is based only on the polyether polyol with no Poly bd<sup>®</sup> Resin.

**Figure 4: Comparative Hydrolytic Stability of Conventional vs. Polybutadiene Polyol Urethanes**

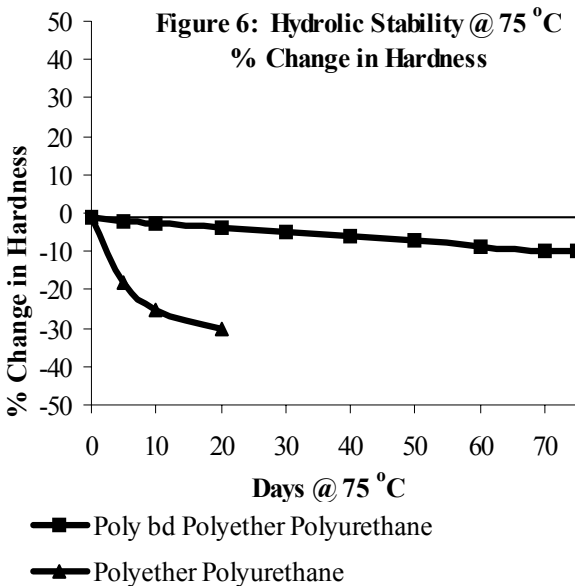


Ref: Gahimer, F. H., and Nieska, "Navy Investigates Reversion Phenomena of Two Elastomers," Insulation, August (1968), p 39 - 44

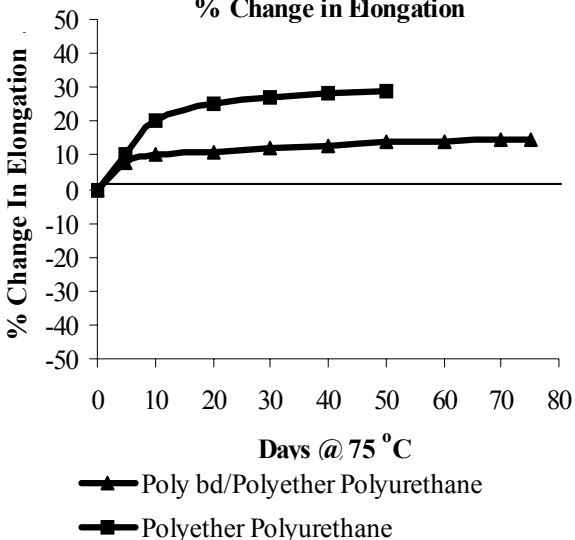
**Figure 5: Hydrolytic Stability @ 75 °C %  
Change in Tensile Strength**



**Figure 6: Hydrolytic Stability @ 75 °C  
% Change in Hardness**



**Figure 7: Hydrolytic Stability @ 75 °C  
% Change in Elongation**



Test specimens were immersed in water at 75 °C for the period shown. Elastomers containing Poly bd® Resin had excellent retention of all physical properties after 75 days. Note that hardness and tensile strength are virtually unchanged after 50 days immersion.

### Chemical Resistance

Poly bd® Resins can be used to prepare polyurethane elastomers with excellent resistance to aqueous inorganic acids and bases. This characteristic is also attributable to the hydrophobic nature of the polybutadiene backbone.

To demonstrate this resistance, an elastomer prepared from a 9% free NCO Poly bd® R-45HTLO/TDI prepolymer cured with 2-ethyl-1, 3-hexanediol was immersed in the test media shown for one week at 71 °C. The results are summarized in Table 3. The physical properties showed minimal change after exposure. Note also that there was no significant dimensional or weight change.

These data indicate that Poly bd® Resin can be used to formulate chemically resistant urethane elastomers. Physical properties of the elastomer can be varied by techniques discussed later in this bulletin. The added chemical resistance obtained from the hydrocarbon Poly bd® polymer will vary of course, depending upon the concentration in a given formulation.

### Low Exotherm/ Ambient Temperature Cure

Poly bd® Resin based systems can be cured at ambient temperatures by reaction with di- or polyisocyanates. Pot life/cure time can be readily adjusted by using typical urethane catalysts. Even when employing catalysts to increase reaction rates, exotherms are low, usually in the range of 15 ° to 40 °C (300-g mass).

### Minimal Shrinkage

Poly bd® Systems cured at ambient temperature exhibit little or no shrinkage.

### Toxicity

Poly bd® Resins are high molecular weight polymers and, therefore, are not expected to pose significant toxicity/health risks during handling and use. However, appropriate protective equipment and good personal hygiene should be employed as with any chemical.

**Table 3: Chemical Resistance of Poly bd® Resin Based Urethanes**

	<b>Untreated</b>	<b>20% H<sub>2</sub>SO<sub>4</sub></b>	<b>20% NaOH</b>
Tensile Strength, psi	1450	1420	1470
Elongation, %	315	335	335
Modulus, psi			
100%	775	765	770
200%	1020	1010	1020
300%	1340	1280	1350
Tear Strength, pli	224	227	228
Hardness, Shore A	89	89	89
Dimensional change, %	--	+0.10	+0.40
Weight Change, %	--	+0.18	0

Refer to MSDS for each Poly bd® resin before handling.

For typical applications, low vapor pressure polymeric isocyanates are recommended. Toxicity and proper handling information on this component of the formulation can be obtained from the manufacturer. Material Safety Data Sheets should be obtained from each manufacturer for all ingredients prior to formulation.

### **Formulating Flexibility**

Unlike preformulated potting and encapsulating compounds, Poly bd® Resins provide fabricators or formulators a wide degree of flexibility to develop products suitable for their particular needs. Desired physical properties can be achieved by formulating with short chain diols, fillers and extenders.



## Urethane Chemistry- One Step Urethane Reactions and Two Step Urethane and Urea - Urethane

Poly bd<sup>®</sup> formulations can be divided into two categories – one-step urethanes and two-step urethanes. One-step systems are based upon the direct reaction of Poly bd<sup>®</sup> Resin with an isocyanate. The one-step systems offer the advantages of versatility, simplicity, and low cost fabrication techniques for preparing urethanes having a wide range of physical properties. Such applications as caulks, sealants, elastomers and foams are possible via these systems.

Two-step Poly bd<sup>®</sup> Resin systems are based upon the intermediate formulation of a prepolymer which can be further chain-extended and crosslinked with additional diols and diamines to form the final polyurethane. These systems usually provide higher performance urethanes and have the advantages of lowering the overall toxicity of the system. Again, a variety of products are possible.

### One -Step Urethane Reactions

Poly bd<sup>®</sup> liquid polymers offer distinct advantages in preparing one-step urethane products. The primary, allylic nature of the hydroxyl groups, combined with their controlled functionality (hydroxyl functionality estimated between 2.2 – 2.6 per chain depending upon particular Poly bd<sup>®</sup> Resin grade) enables products to be rapidly and completely cured at ambient or elevated temperatures. The cure reaction of Poly bd<sup>®</sup> Resin with 4, 4' methylenebis (phenyl isocyanate) is illustrated in Figure 8.

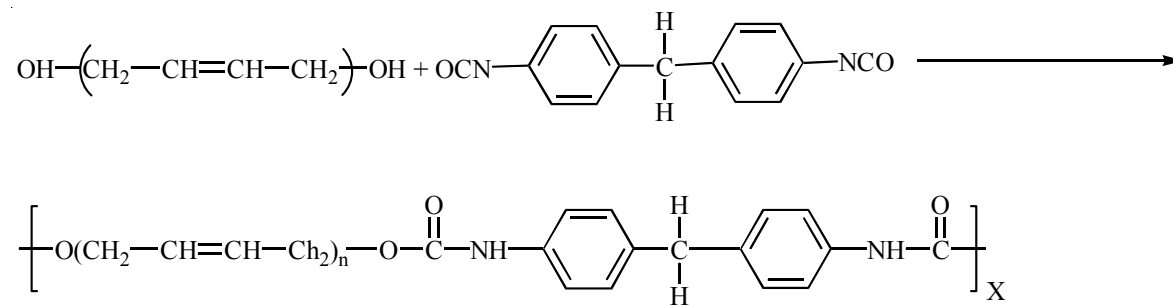
### Procedure for Reacting Poly bd<sup>®</sup> Liquid Polymers with Isocyanates

When producing simple, unfilled elastomers from Poly bd<sup>®</sup> Resins and isocyanates, the liquid polymer and the isocyanate are blended with good mixing. The mixture is poured into a mold or applied to a substrate and permitted to cure at ambient or elevated temperature. To prepare castings which are bubble-free, vacuum degassing is recommended. Refer to appendices for additional details concerning calculations and procedures.

Table 4 shows typical properties of unfilled gumstocks prepared by reacting Poly bd<sup>®</sup> Resins with commercial isocyanates. The use of NCO/OH ratios lower than unity results in softer, lower modulus materials. At NCO/OH ratio levels above 1.0 lower modulus material may also be prepared. However, these materials will gradually increase in hardness with time since the free NCO groups can undergo further reaction with moisture to give urea structures, or can form allophanate crosslinks (especially at elevated temperatures).

The properties of the unfilled urethanes in Table 4 resemble those of unreinforced SBR vulcanizate. Whereas the tensile and tear strengths are low, the gumstocks show good compression set, impact resilience and low temperature properties.

**Figure 8: One-Step Urethane Elastomers**



**Table 4: Properties of Typical One-Step Unfilled Urethanes Based on Poly bd<sup>®</sup> Resin**

Formulation (pbw)	1	2
Poly bd <sup>®</sup> R-45HTLO resin	100	100
Toluene diisocyanate	70	-
Isonate 143L	-	12.5
DBTDL	0.2	0.2
<b>Physical Properties of Gumstocks</b>		
Tensile Strength, psi	140	179
Ultimate Elongation, %	120	101
Modulus, psi, 100%	90	179
Tensile Set, %	0	--
Hardness, Shore A	45	53
Tear Strength, pli	20	16
Compression Set, % Method B, 22hrs. @ 158°F	5.5	--
Compression Set, % Method B, 22hrs. @ 212°F	30	--
Impact Resilience Goodyear Haealey Rebound, %	71	--
Low Temperature Brittleness (B) at -70°C	Passed-F	Passed
Chemical Resistance - Volume Change, +% 10% NaOH, 72hrs. @ 216°F	4.4	--
10% HCL, 72 hrs. @ 216°F	3	--

Unfilled urethane systems may be cured at ambient temperatures, but cure times can be up to 24 hours when no catalyst is used. However, using either typical urethane catalysts and/or elevated temperatures, cure rates are greatly accelerated. Catalysts such as dibutyltin dilaurate and 1, 4-diazo [2.2.2] bicyclooctane have been used successfully with these systems. Catalyst concentrations are usually in the range of 0.05-0.5 weight %, depending upon the particular system. The tin catalysts are recommended for fast cures at ambient temperatures in approximately 12 – 16 hours, at 75 °C. in 8-12 hours, and at 125 °C. in 15-30 minutes.

#### **Filled, One -Step Urethanes**

Poly bd<sup>®</sup> Resin based urethanes have a polymer structure similar to conventional diene rubbers. Just as those systems respond to carbon black reinforcement, Poly bd<sup>®</sup> Resin based urethane systems can be reinforced with various carbon blacks, as well as a variety of other fillers. Although carbon blacks

must be masterbatched into conventional elastomers with the aid of high-shear mixers for efficient dispersion, they may be dispersed readily into liquid Poly bd<sup>®</sup> Resins before curing with minimum energy requirements. Thus, new lower cost fabrication techniques are now offered to rubber goods manufacturers.

The properties of representative carbon black reinforced elastomers are shown in Table 5 (Formulations 1 and 2). Comparison of these data with those of Table 5 illustrates that tenfold increases in tensile strengths are obtainable along with general increases in elongation and other strength properties.

A three roll mill was used to prepare the dispersions in Table 5. The use of high shear compounding equipment, such as Hochmeier or Cowles mixers, results in the lowering of values at least 50%.

#### **Calcium Carbonate**

Calcium carbonate is used quite extensively to extend general-purpose elastomers. Common designations for calcium carbonates include limestone, chalk, whiting and ground oyster shell.

In general, calcium carbonates are quite soft and may be used at rather high levels. In fact, addition of calcium carbonate enhances the extrusion properties of general purpose goods. Elastomers prepared using this filler are suitable for many caulk and sealant applications where high elongation and moderate tensile properties are required.

Various levels and types of calcium carbonates were investigated to determine the effects on Poly bd<sup>®</sup> Resin based elastomers. The results of this investigation are shown in Table 5, Formulations 3-7.

#### **Silica**

In general, silicas contribute a greater increase in tensile strength than other non-carbon black fillers. Silicas also have a profound stiffening effect as indicated by the increased modulus and hardness and the decrease in ultimate elongation and tear strength shown in Table 5, Formulations 8 and 9.

## Clay

Clays, like calcium carbonates, are low cost, high bulking fillers widely used in the rubber industry. In general, clays give only a moderate degree of reinforcement, only fair abrasion resistance but a relatively high stiffening effect. Clays are used as fillers in stocks requiring hardness and high modulus; e.g., shoe soles and heels, mats, and floor tiles.

The use of clays as non-black fillers for Poly bd<sup>®</sup> Resin based elastomers has been investigated. Data describing the use of clay in Poly bd<sup>®</sup> based elastomers are shown in Table 5, Formulations 10 and 11.

## Zinc Oxide

In addition to reinforcement, zinc oxide also provides resilience and heat conductivity. Its use as a reinforcing filler in general purpose elastomers is rather limited, however, due to high density and cost.

The data in Table 5, Formulations 4 and 12, shows zinc oxide to have a reinforcing effect on Poly bd<sup>®</sup> Resin based elastomers. Increasing the zinc oxide content improves tensile and tear strength, increases hardness and modulus but decreases elongation. The

effect of oil extension on the physical properties of zinc oxide reinforced Poly bd<sup>®</sup> Resin based elastomers is discussed in the last section of this page

## Combinations of Zinc Oxide and Carbon Black

Zinc oxide can be effectively employed as a reinforcing filler in conjunction with carbon black. There is a progressive increase in tensile, modulus, tear, and hardness as the carbon black loading is increased. However, at a constant carbon black level, increasing the concentrations of zinc oxide also increases tensile, tear, modulus and hardness until maximum values are reached, after which there is a decrease in overall physical properties. The abrasion resistance of elastomers filled with combinations of zinc oxide and carbon black are generally superior to those filled using carbon black alone. It is important to note that at a constant carbon black level, increasing the concentration of zinc oxide decreases the workable pot life after the isocyanate component is added; i.e., gelation occurs more rapidly.

In addition to carbon black, other fillers may be incorporated into Poly bd<sup>®</sup> Resin urethane systems to alter physical properties and reduce costs.

**Table 5: Properties of Representative Filled Poly bd<sup>®</sup> Elastomers**

Formulation (pbw)	1	2	3	4	5	6	7	8	9	10	11	12
Poly bd <sup>®</sup> R-45HTLO Resin	100	100	100	100	100	100	100	100	100	100	100	100
Toluene diisocyanate	7	7	7	7	7	7	7	7	7	7	7	7
DBTDL	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Elftex-5 (HAF-Fluffed)	30											
Regal 600 (ISAF-L3)		50										
Calcium Carbonate			100									
Zinc Oxide				100								150
Calcene (CaCO <sub>3</sub> )					100							
Calcene NC (CaCO <sub>3</sub> )						50						
Calcene CO (CaCO <sub>3</sub> )							100					
Silica Filler								100	50			
Clay										50	100	
<b>Physical Properties</b>												
Tensile Strength, psi	1700	1740	350	510	510	680	560	1800	1100	520	560	770
Elongation, %	570	460	320	420	610	630	700	60	170	280	270	210
Modulus, psi												
100%	210	280	240	190	330	210	260		800	310	430	470
200%					420	310	340			460	560	750
300%	777	1030	330	420	450	450	380					
Tear Strength, pli	139	179	53	94	128	89	99	52	78	55	72	60
Hardness, Shore A	53	58	55	52	59	55	58	90	76	62	71	71

Note: All Poly bd systems used in thermal cycling conditions in air should contain 1 phr of an antioxidant such as Rabox46 or Irganox 1076. The antioxidant should be added at a temperature above its melting point.

Although the degree of reinforcement using calcium carbonate or zinc oxide fillers is not as great as with carbon black, the non-black systems can be more highly loaded (300phr and higher is feasible), and in combination with oil extension (see Table 6) can provide starting points for a wide variety of low cost caulks, sealants, elastomers and foams. In addition, a wide variety of fillers, including clays and talcs can be employed with these systems either alone or in combination to provide the formulator versatility in compounding for specific properties.

### Conclusions

In most cases, non-carbon black fillers contribute less reinforcement to Poly bd<sup>®</sup> Resin based elastomers than carbon black fillers. However, there are several advantages inherent in using non-black fillers. Some of the more prominent advantages are:

- Ease of dispersion
- Lower viscosity build
- Light color
- High loadings – low cost.

### Oil Extension

Poly bd<sup>®</sup> Resins, because of their hydrocarbon backbones, are compatible with conventional hydrocarbon oils, chlorinated oils, asphalts and other related low cost materials. Such mixture can be cured with conventional diisocyanates to yield oil-extended elastomers.

**Table 6: Effect of Oil-Extension on zinc Oxide Reinforced Poly bd<sup>®</sup> Based Elastomers**

Formulation (pbw)	1	2	3
Poly bd <sup>®</sup> R-45M resin	100	100	100
NCO:OH	1:1	1:1	1:1
DBTDL	0.2	0.1	0
Process Oil (A)		25	50
Zinc Oxide	300	300	300
<b>Physical Properties</b>			
Tensile Strength, psi	1030	750	510
Ultimate Elongation, %	160	240	650
Modulus, psi			
100%	890	570	220
200%	--	730	330
300%	--	--	370
Tear Strength, pli	93	89	92
Hardness, Shore A	82	73	49

(A) Naphthenic oil, viscosity 120cp @ 100 °F

The quantity of oil which can be incorporated into a Poly bd<sup>®</sup> Resin system is a function of the type of oil and the type of filler, if present. Cured Poly bd<sup>®</sup> Resin systems may be formulated which incorporate in excess of 100 parts oil per 100 parts Poly bd<sup>®</sup> Resin and do not “bleed” oil from the final product.

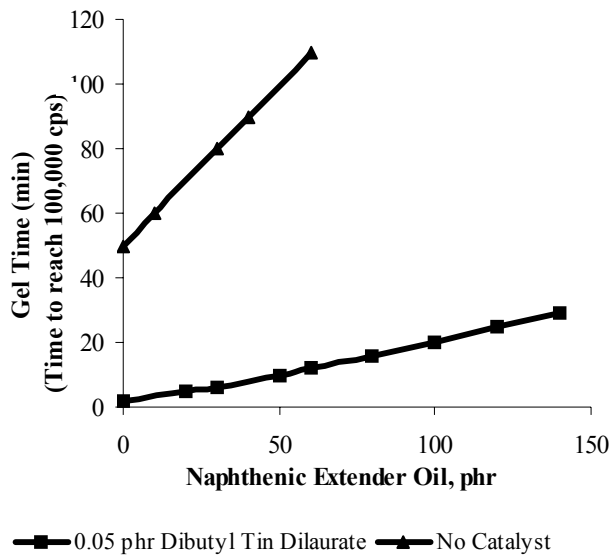
Table 6 illustrates the effect of oil extension on a zinc oxide-reinforced Poly bd<sup>®</sup> Resin system. With the addition of up to 50 parts oil, there is a moderate decrease in tensile strength and modulus, and elongation is greatly improved while the tear strength is unaffected. No bleeding of oil was noted. This data demonstrates that the combination of oil extension and filler reinforcement provides a means to improve overall properties of Poly bd<sup>®</sup> Resin based urethanes while reducing formulation cost.

Oil extension provides many other advantages such as further improvement in hydrolytic stability, control of premix viscosities, gel time, cure time, and the ability to attain higher filler loading. The use of materials such as chlorinated waxes and oils also provides fire retardant properties to the finished product.

The effect of oil extension on gel time is shown in Figure 9 for an unfilled one-step urethane cured with Isonate 143L. The addition of 60 parts oil extended the gel time in an uncatalyzed reaction from 50 to 110 min. With a tin catalyzed system, the trend to lengthen gel time is maintained. However, the relative effect is diminished.

The addition of an electrical grade hydrocarbon oil or ester plasticizer will serve to extend the formulation by a dilution effect. These materials are generally utilized to reduce viscosity and lower cost. Increasing levels of oil or plasticizer will tend to increase elongation and lower tensile and tear strengths. Very high levels of oil or plasticizer (up to 80 parts per 20 parts by weight of polymer) will result in a jelly-like cured material with minimal physical properties but still retaining electrical insulation characteristics. The addition of liquid extenders will also extend pot life and cure times.

**Figure 9: Gel Time vs. Extender Oil**



The selection of the extender oil will depend on compatibility parameters as well as the viscosity and volatility characteristics desired. Hydrocarbon process oils of the aromatic and naphthenic types are very compatible and can be used at relatively high levels. Process oils that have a high paraffinic component are the least compatible.

Plasticizers are also effective as extenders for Poly bd<sup>®</sup> Resin and they tend to be lower in viscosity than process oils. Aromatic ester plasticizers such as dioctyl and diundecyl phthalate as well as triisononyl mellitate can be used. Linear esters such as di-tridecyl adipate are also useful.

A list of generally compatible extenders for Poly bd<sup>®</sup>

Resin includes:

Dioctyl Phthalate

Diundecyl Phthalate

Tricresyl Phosphate

Chlorinated Paraffin

Aromatic Process Oil

Naphthenic Process Oil

Alkyl Naphthalenes

Asphalt

Coal Tar

Linseed Oil

Tung Oil

Detergent Alkylate

### Asphalt Extension

In addition to extender oils and fillers common to rubber processing, asphalt modification provides the basis for formulating many low-cost elastomeric caulks, sealants and coatings. Tables 7 and 8 show the properties of a series of asphalt-extended products prepared by adding a mixture of carbon black, Poly bd<sup>®</sup> R-45HTLO Resin and polymeric, PAPI 901 (f=2.6) or PAPI 27 (f=2.3), or modified MDI, Isonate 143L, products to either AC-20 or AC-5 straight run asphalt. The 1:1 NCO/OH ratio used includes the isocyanate equivalency of the asphalt. This data indicates that, while the tensile strengths of the elastomers are only moderately affected, the elongations change significantly with the level of asphalt and the functionality of the isocyanate.

**Table 7 Poly bd<sup>®</sup> Resin/Asphalt AC - 20 Urethanes**

Formulation (pbw)	1	2	3	4	5	6	7	8	9	10
Poly bd <sup>®</sup> R-45HTLO resin	100	100	100	100	100	100	100	100	100	100
Asphalt AC-20	100	200	300	300	200	200	300	400	100	400
Carbon Black Sterling R	6	9	12	15	6	9	12	15	6	15
DBTDL, drops	2	2	2	2	2	2	2	2	2	2
PAPI 27	13.26	14.73	16.2	17.67	--	--	--	--	--	--
PAPI 901	--	--	--	--	13.02	14.46	15.91	17.36	--	--
Isonate 143L	--	--	--	--	--	--	--	--	14.21	18.94
<b>Physical Properties</b>										
Tensile Strength, psi	189	222	319	226	224	340	354	309	265	256
Elongation, %	183	303	437	413	291	600	720	785	506	>800
Tensile Set, %	4	7	18	13	5	17	17	25	8	56
Modulus, psi										
100%	158	132	108	89	158	127	100	80	125	72
200%	--	161	122	107	190	145	106	91	160	81
300%	--	--	169	145	220	164	122	101	185	88
Tear Strength, pli	34	44	46	35	42	54	48	40	54	42
Hardness, Shore A	49	48	50	43	44	49	45	45	41	25



### Short Chain Reinforcing Diols

Physical properties of Poly bd<sup>®</sup> Resin based elastomers can be improved by the addition of auxiliary polyols to the formulation. A wide variety of short chain diols may be employed, however, Voranol 220-530 (phenyl diisopropanolamine) was found to be most effective.

The use of a short chain diol in conjunction with the required additional isocyanate increases the urethane concentration in the final polymer backbone. This combination leads to increased hydrogen bonding between polymer chains and thus higher strength properties in the cured elastomer. The same effect is

noted when short chain diamines or mixed alcohol diamines are employed as auxiliary reactants with Poly bd<sup>®</sup> Resin.

The Voranol 220-530 chemically reinforces the cured Poly bd<sup>®</sup> elastomer, increasing hardness and improving tensile and tear strengths. Table 9 shows the effect of using increasing levels of Voranol 220-530 in a Poly bd<sup>®</sup> system. Other effective short chain diols are 2-ethyl-1,3-hexanediol and bis-hydroxyethyl dimerate.

Isocyanates such as PAPI27, PAPI 901 or others may be used with Voranol 220-530 systems, but the Isonate 143L produced elastomers with better physical properties.

**Table 8 Poly bd<sup>®</sup> Resin/Asphalt AC -5 Urethanes**

Formulation (pbw)	1	2	3	4	5	6	7	8	9	10
Poly bd <sup>®</sup> R-45HTLO resin	100	100	100	100	100	100	100	100	100	100
Asphalt AC-5	100	200	300	300	200	200	300	400	100	400
Carbon Black Sterling R	6	9	12	15	6	9	12	15	6	15
DBTDL, drops	2	2	2	2	2	2	2	2	2	2
PAPI 27	13.26	14.73	16.2	17.67	--	--	--	--	--	--
PAPI 901	--	--	--	--	13.02	14.46	15.91	17.36	--	--
Isonate 143L	--	--	--	--	--	--	--	--	14.21	18.94
<b>Physical Properties</b>										
Tensile Strength, psi	174	174	202	219	214	275	292	249	466	230
Elongation, %	170	287	403	570	300	562	688	650	593	930
Tensile Set, %	3	5	11	12	4	11	12	14	9	35
<b>Modulus, psi</b>										
100%	140	105	76	59	133	111	79	60	111	56
200%	--	134	97	74	173	128	88	72	142	65
300%	--	--	134	97	207	146	100	90	165	75
Tear Strength, pli	34	33	35	29	207	146	100	90	165	75
Hardness, Shore A	43	41	39	34	42	56	39	34	37	25

**Table 9 Effects of Voranol 220-530 Content NCO/OH ratio = 1.05**

Formulation (pbw)	1	2	3	4	5	6	7	8
Poly bd <sup>®</sup> R-45HTLO resin, g	100	100	100	100	100	100	100	100
Voranol 220-530	--	22.2	4.45	8.89	11.85	17.78	26.67	35.56
DBTDL, drops	4	4	4	4	4	4	4	4
Cyanox 2246, g	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Isonate 143L	12.76	15.45	19.14	25.53	29.78	38.29	51.05	63.81
Equivalent ratio - Poly bd <sup>®</sup> /Voranol 220-530	--	4/1	2/1	1/1	3/4	1/2	1/3	1/4
Tensile Strength, psi	179	242	377	902	1192	2021	2695	3476
Tear Strength, pli	16	40	46	114	150	231	308	411
Elongation, %	101	151	195	238	245	300	325	297
<b>Modulus, psi</b>								
100%	179	192	124	247	465	1073	1609	2182
200%	--	--	195	382	763	1492	2126	2814
300%	--	--	--	--	--	2010	2641	3784
Hardness, Shore A	53	56	62	75	82	--	--	--
Hardness, Shore D	--	--	--	--	--	43	51	53

Oil extension and filler reinforcement can be used in combination with short chain reinforcing diols to allow maximum property and cost modification.

### Two-Step Urethanes and Urea-Urethanes

Previous sections described the preparation of one-step urethane and urea-urethane products based on Poly bd<sup>®</sup> Resins. In many conventional urethane applications, it is desirable or essential to utilize a two-step reaction sequence, wherein an isocyanate-terminated prepolymer is first formed and subsequently converted to a high molecular weight elastomer by further reaction with glycols, diamines, or other chain-extending agents.

Isocyanate prepolymers are widely used to produce high performance elastomers in castable, millable, and moldable forms. Other applications include foams, and one and two component coatings, caulks, and sealants.

The chemistry of Poly bd<sup>®</sup> Resin based prepolymer systems is depicted in Figure 10.

Prepolymers of Poly bd<sup>®</sup> Resins can be prepared with excellent storage characteristics when the recommended reaction and handling precautions are observed. See Appendix C for further detailed information.

Reaction of isocyanate-terminated prepolymers with diol or diamine extenders produces solid elastomers. In addition to the normal chain extension reaction,

cross-linking occurs in these systems because the functionality of the Poly bd<sup>®</sup> Resins is in excess of two. Accordingly, when a diol extender is employed, crosslinking occurs via urethane and allophanate formation. With diamine extenders, further crosslinking is possible through urea and biuret formation.

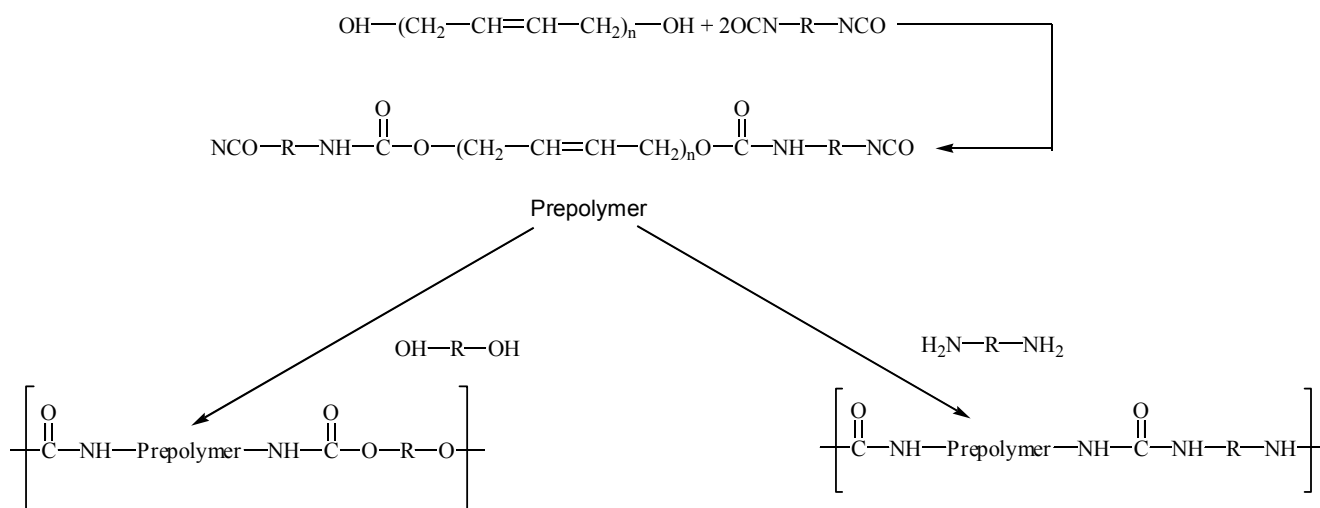
### Two-Step Urethanes

Use of the prepolymer technique to prepare Poly bd<sup>®</sup> Resin urethanes provides a method for preparing elastomers having improved physical properties over unmodified or unreinforced one-step Poly bd<sup>®</sup> Resin systems. A wide variety of chain-extending diols can be employed and this choice will affect the cure rate and the final physical properties of the urethane. Two particularly good diols for use with Poly bd<sup>®</sup> Resin based prepolymers are 2-ethyl 1,3 hexanediol, and Voranol 220-530. Properties of some typical products are summarized in Tables 10 and 11.

As can be seen from this data, maximum properties for both systems in tensile and tear strengths occur at NCO/OH ratios of near unity. However, as the ratio is changed, other properties such as elongation and hardness can be altered. Also, at decreased NCO/OH the degree of thermoplasticity of the system increases so that production of thermoplastic or millable gumstock is feasible.

Variation of the free NCO content of the prepolymer provides a further means of changing the physical properties of the cured gumstocks.

**Figure 10: Two-Step Urethanes and Urea-Urethanes**



**Table 10 Two Step Urethane Elastomers From Poly bd<sup>®</sup> R-45M Resin -TDI Prepolymer and 2-Ethyl-1, 3-Hexandiol**

<b>Formulation (pbw)</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>
Percent Free NCO of prepolymer	6.39	6.56	6.56	6.56	8.71	8.71	8.71	8.71
Reaction NCO/OH Ratio	0.8	0.9	1	1.1	0.8	0.9	1	1.1
<b>Formulation (pbw)</b>								
R-45M Prepolymer	100	100	100	100	100	100	100	100
2-Ethyl-1, 3-hexanediol	13.9	12.6	11.4	10.4	19	16.9	15.2	13.8
DBTDL	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
<b>Elastomer Properties</b>								
Tensile Strength, psi	310	550	1140	990	340	1300	2270	2200
Tear Strength, pli	55	105	140	145	90	230	215	210
Elongation, %	950+	635	460	335	1130	615	470	410
Modulus, psi								
100%	95	185	365	480	195	555	770	865
200%	110	235	495	665	220	680	1010	1145
300%	125	280	650	890	235	795	1270	1470
Hardness, Shore A	45	58	70	74	45	80	84	88

**Table 11 Two Step Urethane Elastomers from Poly bd<sup>®</sup> R-45M Prepolymers and Voranol 220-530**

<b>Formulation (pbw)</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>
Poly bd <sup>®</sup> R-45M Prepolymer	100	100	100	100	100	100	100	100
Tinuvin 327	--	--	--	1	--	--	--	--
Cyanox 2246, g	--	--	--	1	--	--	--	--
DBTDL Catalyst	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Voranol 220-530	15	21.3	29.6	36	26.7	24	21.3	19.3
NCO/OH ratio	1	1	1	1	0.83	0.94	1.04	1.15
Percent NCO of Prepolymer	6	8.9	11.9	14.5	8.9	8.9	8.9	8.9
<b>Physical Properties</b>								
Tensile Strength, psi	1280	3140	2970	1920	1090	3170	3140	2160
Tear Strength, pli	195	295	340	290	275	345	295	305
Elongation, %	400	495	400	400	705	595	495	575
Modulus, psi								
100%	570	1040	1485	1690	805	990	1040	880
200%	765	1340	1950	1650	890	1235	1340	1120
300%	970	1685	2200	1670	910	1475	1685	1310
Hardness, Shore D	34	42	51	50	40	42	42	40



## Applications For Poly bd Resin Systems

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### Applications for Poly bd<sup>®</sup> Resin Systems

A wide range of properties is attainable for Poly bd<sup>®</sup> Resin based urethanes. These features, combined with their versatility in formulating with compatible oils and inexpensive fillers, enhance their value for many elastomer applications. General-purpose rubber parts can be easily and inexpensively processed from Poly bd<sup>®</sup> Resin based formulations. These products can vary from completely castable liquids to millable gumstocks. Oil and asphalt-extended Poly bd<sup>®</sup> Resin based systems permit the manufacture of low-cost systems for elastomer coatings, linings, adhesives, caulks and sealants.

Compounding flexibility permits viscosity control for sprayable coatings and adhesives, with excellent moisture and chemical resistance. Other advantages of Poly bd<sup>®</sup> Resin based 100% polymer systems over many conventional systems include the elimination of the use of solvents and solvent removal equipment and capability for room temperature cures or very fast oven cures. The liquid Poly bd<sup>®</sup> Resin prepolymer systems, cured with Voranol 220-530, provide excellent high performance coatings having good abrasion, impact and low temperature ductility properties.

Both rigid and flexible products can be produced by chemical blowing techniques identical to those used in foaming polyether and polyester urethanes. Foamed products can be produced with blowing techniques used in sponge rubber production. In addition, Poly

bd<sup>®</sup> Resin based systems can be mechanically frothed to obtain medium density, closed cell, flexible and non-flexible products.

Potting and encapsulating compounds exhibit excellent electrical properties. Further information on the electrical properties of these materials is available on request.

The ability of Poly bd<sup>®</sup> Resins to accept high filler loadings and to be extended with asphalts and oils allows the formulation of many low-cost, good performance caulks and sealants.

A list of applications where Poly bd<sup>®</sup> urethane elastomers are typically used includes:

- Electrical/Electronic Potting & Encapsulation
- Construction
- Asphalt Extended Membranes
- Waterproofing Membranes
- Roofing
- Mastics
- Highway Sealants
- Architectural Sealants
- Insulating Glass Sealants
- Adhesives
- Rubber Parts
- Military/Aerospace
- Automotive
- Encapsulants
- Sealants

## Appendix A

In all urethane calculations the reactants should be expressed in terms of equivalent weight. The basis for this approach is that one equivalent of OH or other

active H will react with one equivalent of NCO. In general, an NCO/OH equivalent ratio of approximately 1 will provide the best physical properties. The following relationships for equivalent weight (EW) will be helpful in urethane calculations.

For Hydroxyl Compounds:  $EW = \frac{\text{Molecular Weight}}{\text{Functionality}}$

$$EW = \frac{56100}{\text{Hydroxyl Number}}$$

$$EW = \frac{1000}{\text{Hydroxyl Value}}$$

$$EW = \frac{1700}{\% \text{Hydroxyl}}$$

$$\begin{aligned} \text{Also: Hydroxyl Value} &= \frac{\text{milliequivalent OH}}{\text{g}} \\ &= \frac{\text{Hydroxyl Number}}{56.1} \end{aligned}$$

For Isocyanate Compounds:  $EW = \frac{\text{MW of NCO Compound}}{\text{Functionality}}$

$$EW = \frac{4200}{\% \text{NCO in compound}}$$

Examples of Urethane Formulation Stoichiometry follow:

### General Formula

$$\frac{\text{Weight NCO Compound}}{100 \text{ g Hydroxyl Compound}} = \frac{(\text{Hydroxyl Value}) (\text{EW NCO compound}) (\text{NCO/OH ratio})}{10}$$

1. Find the weight of Isonate 143L (EW = 143) needed to prepare a gumstock from Poly bd R- 45HTLO Resin (Hydroxyl Value = 0.83 meq/g) at an NCO/OH ratio of 1.1.

$$\frac{\text{Weight Isonate 143L}}{100\text{g Poly bd R-45HTLO Resin}} = \frac{(0.83)(143)(1.1)}{10} = 13.0\text{g}$$

For each 100g of R-45HTLO Resin, 13.0g of Isonate 143L would be required.

2. Find the weight of Isonate 143L needed to make a Poly bd Resin system reinforced with Voranol 220-530, at a Poly bd/Isonol equivalent of 0.5 and NCO/OH ratio of 1.1.

$$\begin{aligned} \text{Equivalents Poly bd R-45HTLO Resin} &= \frac{\text{Weight Poly bd R-45HTLO Resin}}{\text{EW Poly bd R-45HTLO Resin}} \\ &= \frac{100\text{g}}{1000/0.83 \text{ meq/g}} = 0.083 \text{ equivalents} \end{aligned}$$

$$\begin{aligned} \text{Equivalents Voranol 220-530} &= (2) (\text{equivalents Poly bd R-45HTLO Resin}) = (2) (0.083 \text{ eq}) \\ &= 0.166 \text{ equivalents} \end{aligned}$$

$$\begin{aligned} \text{Weight Voranol 220-530} &= (\text{Equivalents Voranol 220-530}) (\text{EW Voranol 220-530}) \\ &= (0.166\text{eq}) (104.5\text{g/eq}) = 17.3\text{g} \end{aligned}$$

$$\begin{aligned} \text{Weight Isonate 143L required} &= (\text{total OH equivalents}) (\text{EW Isonate 143L}) (\text{NCO/OH}) \\ &= (0.083\text{g} + 0.166\text{g}) (143 \text{ eq/g}) (1.1) = 39.2\text{g} \end{aligned}$$

Formula would be:

Poly bd R-45HTLO Resin 100g  
 Voranol 220-530 17.3g  
 Isonate 143L 39.2g

3. Prepolymers of Poly bd<sup>®</sup> Resins may be prepared at various % free NCO by use of the following equation:

$$\frac{\text{weight isocyanate}}{100\text{g Poly bd Resin}} = \frac{100 (\text{EW Isocyanate})}{\text{EW Poly bd Resin}} \times \frac{100(\text{EW NCO}) + (\text{EW Poly bd Resin}) (\% \text{Free NCO})}{100 (\text{EW NCO}) - (\text{EW Isocyanate}) (\% \text{Free NCO})}$$

## Appendix B – Preparation of Elastomers

### 1. One – Step Urethanes

- A. Weigh out the components of the desired formulation; e.g., Poly bd<sup>®</sup> Resin, filler(s), extender oil, anti-oxidants, and catalyst.
- B. Charge the weighed components to a suitable mixing device and mix until the ingredients are well dispersed. Mixing time may vary with the efficiency of the mixing device.

It is advisable to degas the “masterbatch” during the mixing steps under vacuum. It may also be necessary to dry fillers at elevated temperatures to remove moisture.

- C. Add the calculated amount (See Appendix A) of di- or polyisocyanate to the mixture to give the desired NCO/OH ratios between 1.0 and 1.1
- D. Mix the completed formulation until homogenous and pour or pump into molds or apply to desired substrate.
- E. If multi-component mixing-metering equipment is utilized, the filled Poly bd<sup>®</sup> Resins and isocyanate components may be metered separately into the mixing head of the metering device.
- F. Permit the formulation to cure, Longer cure times are required at lower temperatures. The elastomers described in this bulletin were arbitrarily cured at 80 – 100 °C for 1.5 hours unless otherwise stated. No differences in properties were noted between elastomers which were cured at ambient or elevated temperatures.

Catalyst concentration and/or level of oil extension permit wide latitude in pot lives and cure times of specific formulations as previously discussed.

Moreover, the type of filler greatly affects workable pot life and cure time. For example, zinc oxide catalyzes the reaction and, therefore, shortens pot life. But acids fillers such as silicas may have a retarding effect on pot life and ultimate cure time.

### 2. One –Step Urea- Urethanes

Procedure is identical as for the one –step urethanes, except that diamines are added to the Poly bd<sup>®</sup> Resin formulation during the initial mixing cycle.

### 3. Two – Step Urethanes

The weighed prepolymer is mixed with weighed amounts of catalyst, filler and polyol, such as Voranol 220-530, or more Poly bd<sup>®</sup> Resin (or diamine if urea-urethanes are desired) and cured at ambient or elevated temperature.

\*Note: A three-roll paint mill was used to prepare the filled formulations presented in this bulletin. However, experimental work has shown that fillers can be satisfactorily dispersed with conventional mixers.

## Appendix C – Preparation of Prepolymers

Prepolymers from Poly bd<sup>®</sup> Resins can be prepared with excellent shelf– life when the following reaction and storage precautions are observed:

1. The Poly bd<sup>®</sup> Resin must be thoroughly degassed. It is recommended that the resin is heated at 100°C at low vacuum for a minimum of 30 minutes with efficient stirring.
2. The addition of benzoyl chloride (0.03% based on Poly bd<sup>®</sup> Resin) prior to the addition of the diisocyanate greatly enhances the stability of the prepolymer product. The same amount of benzoyl chloride may be added to the prepolymer at the completion of the reaction to further insure shelf stability.
3. When storing the prepolymer, containers with plastic lids should be avoided. Narrow-mouthed solvent cans which can be plugged and then capped are the most satisfactory containers. The product should always be purged with nitrogen before sealing.

The procedure below has been employed successfully in preparing Poly bd<sup>®</sup> prepolymers. The equipment used was a five- gallon 316 stainless steel jacketed autoclave equipped with an anchor type stirrer, thermowell, charging port, vacuum and nitrogen lines, and a bottom flush valve. The autoclave was heated by circulating a lubricating oil heat transfer fluid through a 6 kW electric heater and then through the autoclave jacket. Cooling was effected by circulating the oil through a water cooled heat exchanger and through the autoclave jacket.

## Procedure

The Poly bd<sup>®</sup> Resin was charged to the autoclave, and the autoclave sealed. The agitator was turned on, the vessel evacuated to 20 mm mercury absolute pressure, and heating begun.

About two hours time was required to heat the charge from 20 to 110 °C, with a maximum jacket temperature of about 115 °C. The autoclave internal pressure was 5 mm mercury absolute pressure at the 110 °C internal temperature. The autoclave contents were degassed and stirred at 110 °C and 5 mm mercury absolute pressure for thirty minutes. The autoclave contents were then cooled to 40 °C over a two-hour period.

The autoclave internal pressure was increased to 55 mm mercury absolute pressure and benzoyl chloride added.

After stirring five minutes, the diisocyanate was added. The addition required about three minutes. The exothermic reaction increased the internal temperature from 40 °C to 50 °C over a twelve-minute time interval.

The autoclave contents were then heated to 60 °C with stirring, at 50 mm mercury absolute pressure, over a one hour period.

The reactor contents were maintained at 60 °C/ 50 mm mercury, with stirring for two hours.

A second portion of benzoyl chloride was then added with stirring, and the product withdrawn into three two–gallon screw-top cans. The cans were thoroughly flushed with purified nitrogen before sealing.

For low NCO pre-polymers (6% Free NCO) it is recommended that 1% Ralex 46 also be added for package stability.

## Appendix D

<b>Tradename</b>	<b>Chemical Name</b>	<b>Supplier</b>
Voranol 220 - 530	(phenyl diisopropanolamine)	The Dow Chemical Company
Isonate 2143L	Polyfunctional liquid isocyanate	The Dow Chemical Company
PAPI	Polymethylene polyphenyl isocyanate	The Dow Chemical Company
Ralox 46	Antioxidant	Raschiq Industries
Tinuvin 327	U.V. Stabilizer	Ciba Corporation
Lead Octoate	Catalyst	Nudex Chemical Company
DBTDL Catalyst	Dibutyltin Dilaurate	Atofina Chemicals
Dabco	Triethylene Diamine	Air Products & Chemicals, Inc.
Pluracol TP - 340	Urethane Triol	BASF Corporation

## Appendix E

The following testss were used to determine properties of Poly bd<sup>®</sup> elastomers described in this brochure.

<b>Physical Propertiy Tested</b>	<b>Method</b>
Tensile Strength	ASTM D - 412 - 61T
Ulimate Elongation	ASTM D - 412 - 61T
Tensile Modulus	ASTM D - 412 - 61T
Tear Strength	ASTM D - 642 - 51 (Die C)
Shore Hardness	ASTM D - 676 - 59T
Flexural Cut Growth Resistance	ASTM D - 1052 - 55
Low Temperature Brittleness	ASTM D - 746
Compression Set	ASTM D - 395
Oil and Solvent Resistance	ASTM D - 471 - 64T
Abrasion Index	ASTM D - 1630 - 61T
Impact Resilence	ASTM D - 1054 -55

# Notes



# CRAY VALLEY

Resins par excellence

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