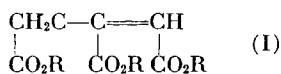


## Copolymerization of Trialkyl Aconitates with Butadiene and Some Vinyl Monomers\*

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Copolymerizations of trialkyl aconitates (I) with vinyl chloride,<sup>1</sup> vinyl acetate,<sup>2</sup> trialkyl cyanurate,<sup>3</sup> dichlorostyrene,<sup>4</sup> and butadiene<sup>5</sup> in free radical systems have been mentioned in the patent literature. There is



also a claim for the formation of homopolymers<sup>2</sup> by heating the esters for long periods with a peroxide catalyst.

Since it is a little surprising that this trisubstituted olefin with an allylic —CH<sub>2</sub>— polymerizes or copolymerizes, we have undertaken some further studies of such reactions and particularly have investigated some reactivity ratios by the method of Mayo and Lewis.<sup>6</sup>

### REACTIVITY RATIOS

Reactivity ratios for the copolymerization of trimethyl aconitate with butadiene, styrene, vinyl chloride, and acrylonitrile were determined. The polymerizations were run in bulk at 60° in the experiments using butadiene, styrene, and vinyl chloride. The polymerizations were run in *N,N*-dimethyl formamide (5.0 ml. per 10-g. charge) at 60° in the experiments using acrylonitrile. 4-oz. screw-cap bottles equipped with self-sealing gaskets were used in all experiments. The experimental method was essentially that of Mayo and Lewis.<sup>6</sup> The step-by-step procedure for the experiments is as follows:

1. The bottles containing 0.015 g. of benzoyl peroxide were weighed to the nearest one-hundredth of a gram.
2. The desired amount of trimethyl aconitate was added to each bottle while on the balance.
3. Sufficient comonomer was introduced into each bottle to make a total monomer charge of approximately ten grams.

\* The work discussed herein was performed as a part of the synthetic rubber research project sponsored by the National Science Foundation.

4. For the liquid monomers the bottles were flushed with dry, oxygen-free nitrogen and capped. For butadiene and vinyl chloride the bottles were allowed to stand for five to ten seconds before being capped.

5. The bottles were weighed again to determine accurately the weight of comonomer added.

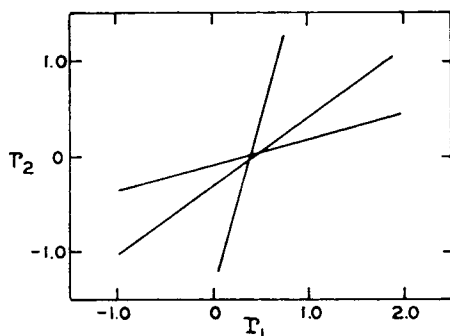


Fig. 1. Reactivity ratios for the copolymerization of butadiene with trimethyl aconitate.

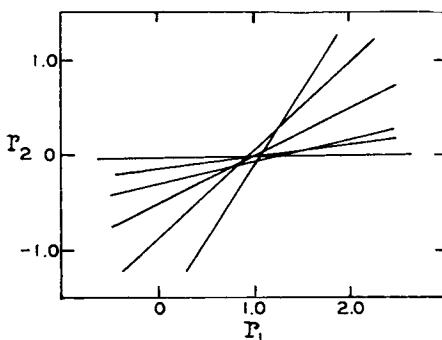


Fig. 2. Reactivity ratios for the copolymerization of styrene with trimethyl aconitate.

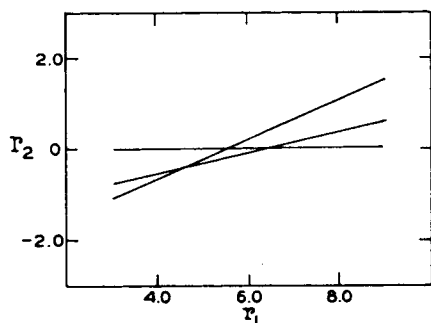


Fig. 3. Reactivity ratios for the copolymerization of acrylonitrile with trimethyl aconitate.

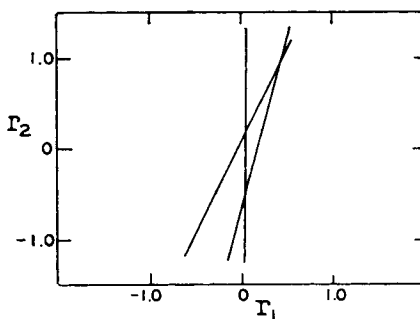


Fig. 4. Reactivity ratios for the copolymerization of vinyl chloride with trimethyl aconitate.

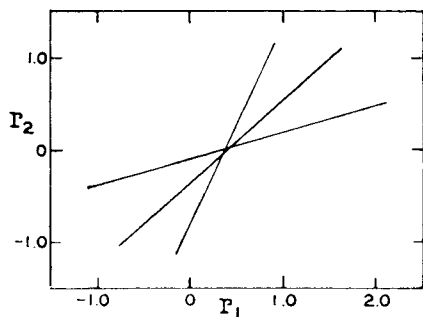


Fig. 5. Reactivity ratios for the copolymerization of butadiene with triethyl aconitate.

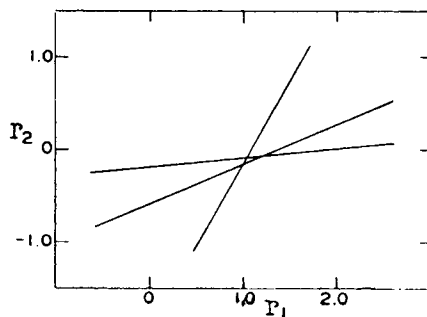


Fig. 6. Reactivity ratios for the copolymerization of styrene with triethyl aconitate.

6. The bottles were then allowed to remain in the 60° water bath for several hours until the desired conversions were obtained.

7. After removal from the bath and cooling the bottles to room temperature, the bottles were placed in a dry ice-acetone mixture for five minutes.

8. The bottles were opened and allowed to warm to room temperature. The contents were poured into vials containing 25 to 50 ml. of methanol to precipitate the copolymers.

9. The copolymers were reprecipitated three times from benzene into methanol. For the acrylonitrile-trimethyl aconitate copolymers *N,N*-dimethyl formamide was used as the solvent and a 50% aqueous solution of methanol was used as the precipitating agent.

10. Finally the copolymers were dried at room temperature at 0.1–0.5 mm. for 24 hours and weighed. They were submitted for elemental analyses in nitrogen filled vials.

The data for these experiments are found in Tables I–VII and Figures 1–6.

TABLE I  
REACTIVITY RATIO DATA FOR BUTADIENE AND TRIMETHYL ACONITATE  
BULK COPOLYMERIZATION AT 60°C.

No.	Charge				Product			
	Tri-methyl aconitate, g.	Butadiene, g.	Mole fraction <sup>a</sup>	$\frac{M_1}{M_2}$ <sup>b</sup>	Conversion, % <sup>c</sup>	Carbon, %	Mole fraction <sup>d</sup>	$\frac{m_2}{m_1}$ <sup>e</sup>
1	6.08	4.10	0.271	2.722	5.0	63.12	0.331	0.486
2	7.98	2.17	0.502	0.995	5.4	60.47	0.411	0.694
3	9.02	1.15	0.650	0.548	9.7	58.85	0.452	0.852

<sup>a</sup> Mole fraction of trimethyl aconitate in the charge.

<sup>b</sup>  $M_1/M_2$  = mole ratio, butadiene/trimethyl aconitate in the charge.

<sup>c</sup> Per cent conversion based on the weight of copolymer obtained.

<sup>d</sup> Mole fraction of trimethyl aconitate in the product.

<sup>e</sup>  $m_2/m_1$  = mole ratio, trimethyl aconitate/butadiene in the product.

TABLE II  
REACTIVITY RATIO DATA FOR ACRYLONITRILE AND TRIMETHYL ACONITATE  
SOLUTION COPOLYMERIZATION AT 60°C.

No.	Charge				Product			
	Tri-methyl aconitate, g.	Acrylonitrile, g.	Mole fraction <sup>a</sup>	$\frac{M_1}{M_2}$ <sup>b</sup>	Conversion, % <sup>c</sup>	Nitrogen, %	Mole fraction <sup>d</sup>	$\frac{m_2}{m_1}$ <sup>e</sup>
1	3.64	6.36	0.30	2.33	1.5	20.35	0.068	0.0730
2	2.69	7.31	0.40	1.50	2.0	19.04	0.087	0.0949
3	1.97	8.03	0.50	1.00	1.5	18.88	0.089	0.0977

<sup>a</sup> Mole fraction of trimethyl aconitate in the charge.

<sup>b</sup>  $M_1/M_2$  = mole ratio, acrylonitrile/trimethyl aconitate in the charge.

<sup>c</sup> Per cent conversion based on the weight of copolymer obtained.

<sup>d</sup> Mole fraction of trimethyl aconitate in the product.

<sup>e</sup>  $m_2/m_1$  = mole ratio, trimethyl aconitate/acrylonitrile in the product.

TABLE III

REACTIVITY RATIO DATA FOR STYRENE AND TRIMETHYL ACONITATE BULK COPOLYMERIZATION AT 60°C.

No.	Charge				Product			
	Tri-methyl aconitate, g.	Styrene, g.	Mole fraction <sup>a</sup>	$\frac{M_1}{M_2}$ <sup>b</sup>	Conversion, % <sup>c</sup>	Carbon, %	Mole fraction <sup>d</sup>	$\frac{m_2}{m_1}$ <sup>e</sup>
1	4.71	5.29	0.30	2.34	3.0	76.50	0.22	0.284
2	5.81	4.20	0.40	1.50	2.0	72.79	0.29	0.407
3	6.75	3.25	0.50	1.00	2.0	70.82	0.33	0.491
4	7.57	2.43	0.60	0.667	2.0	69.99	0.35	0.532
5	8.29	1.71	0.70	0.428	2.0	67.59	0.40	0.668
6	9.49	0.51	0.90	0.108	2.0	65.79	0.44	0.799

<sup>a</sup> Mole fraction of trimethyl aconitate in the charge.

<sup>b</sup>  $M_1/M_2$  = mole ratio, styrene/trimethyl aconitate in the charge.

<sup>c</sup> Per cent conversion based on the weight of copolymer obtained.

<sup>d</sup> Mole fraction of trimethyl aconitate in the product.

<sup>e</sup>  $m_2/m_1$  = mole ratio, trimethyl aconitate/styrene in the product.

TABLE IV

REACTIVITY RATIO DATA FOR VINYL CHLORIDE AND TRIMETHYL ACONITATE BULK COPOLYMERIZATION AT 60°C.

No.	Charge				Product			
	Trimethyl aconitate, g.	Vinyl chloride, g.	Mole fraction <sup>a</sup>	$\frac{M_1}{M_2}$ <sup>b</sup>	Conversion, % <sup>c</sup>	Chlorine, %	Mole fraction <sup>d</sup>	$\frac{m_2}{m_1}$ <sup>e</sup>
1	2.20	6.03	0.085	10.717	1	15.95	0.425	0.739
2	7.02	2.98	0.405	1.469	1	12.01	0.520	1.076
3	6.00	3.93	0.306	2.266	1	16.54	0.413	0.703

<sup>a</sup> Mole fraction of trimethyl aconitate in the charge.

<sup>b</sup>  $M_1/M_2$  = mole ratio, vinyl chloride/trimethyl aconitate in the charge.

<sup>c</sup> Per cent conversion based on the weight of copolymer obtained.

<sup>d</sup> Mole fraction of trimethyl aconitate in the product.

<sup>e</sup>  $m_2/m_1$  = mole ratio, trimethyl aconitate/vinyl chloride in the product.

TABLE V

REACTIVITY RATIO DATA FOR BUTADIENE AND TRIETHYL ACONITATE BULK COPOLYMERIZATION AT 60°C.

No.	Charge				Product			
	Tri-ethyl aconitate, g.	Butadiene, g.	Mole fraction <sup>a</sup>	$\frac{M_1}{M_2}$ <sup>b</sup>	Conversion, % <sup>c</sup>	Carbon, %	Mole fraction <sup>d</sup>	$\frac{m_2}{m_1}$ <sup>e</sup>
1	7.04	2.84	0.34	1.923	3.0	64.29	0.37	0.591
2	8.35	1.95	0.47	1.118	4.7	63.39	0.41	0.682
3	9.12	1.11	0.63	0.581	6.1	62.35	0.46	0.838

<sup>a</sup> Mole fraction of triethyl aconitate in the charge.

<sup>b</sup>  $M_1/M_2$  = mole ratio, butadiene/triethyl aconitate in the charge.

<sup>c</sup> Per cent conversion based on the weight of copolymer obtained.

<sup>d</sup> Mole fraction of triethyl aconitate in the product.

<sup>e</sup>  $m_2/m_1$  = mole ratio, triethyl aconitate/butadiene in the product.

TABLE VI  
REACTIVITY RATIO DATA FOR STYRENE AND TRIETHYL ACONITATE BULK  
COPOLYMERIZATION AT 60°C.

No.	Charge				Product			
	Tri-ethyl aconitate, g.	Styrene, g.	Mole fraction <sup>a</sup>	$\frac{M_1}{M_2}$ <sup>b</sup>	Conversion, % <sup>c</sup>	Carbon %	Mole fraction <sup>d</sup>	$\frac{m_2}{m_1}$ <sup>e</sup>
1	5.19	5.53	0.27	2.642	1.2	77.94	0.20	0.258
2	7.13	2.87	0.50	1.000	1	73.43	0.295	0.418
3	8.44	1.47	0.70	0.434	1	71.06	0.36	0.553

<sup>a</sup> Mole fraction of triethyl aconitate in the charge.

<sup>b</sup>  $M_1/M_2$  = mole ratio, styrene/triethyl aconitate in the charge.

<sup>c</sup> Per cent conversion based on the weight of copolymer obtained.

<sup>d</sup> Mole fraction of triethyl aconitate in the product.

<sup>e</sup>  $m_2/m_1$  = mole ratio, triethyl aconitate/styrene in the product.

TABLE VII  
REACTIVITY RATIOS AT 60°C.

Monomer $M_1$	Monomer $M_2$	$r_1$	$r_2$
Butadiene	Trimethyl aconitate	$0.40 \pm 0.03$	$0.00 \pm 0.015$
Styrene	Trimethyl aconitate	$1.10 \pm 0.01$	$0.00 \pm 0.01$
Acrylonitrile	Trimethyl aconitate	$5.50 \pm 0.50$	$-0.10 \pm 0.10$
Vinyl chloride	Trimethyl aconitate	$0.15 \pm 0.10$	$0.00 \pm 0.50$
Butadiene	Triethyl aconitate	$0.37 \pm 0.03$	$0.00 \pm 0.01$
Styrene	Triethyl aconitate	$1.10 \pm 0.10$	$-0.10 \pm 0.10$

These values indicate that: (1) the butadiene radical is more reactive toward trimethyl aconitate than toward butadiene monomer; (2) the styrene radical is equally reactive toward trimethyl aconitate and styrene monomer; (3) the acrylonitrile radical is less reactive toward trimethyl aconitate than toward acrylonitrile monomer; and (4) the vinyl chloride radical is more reactive toward trimethyl aconitate than toward vinyl chloride monomer. As expected for trimethyl aconitate since it is related to the 1,2-disubstituted ethylenes the reactivity ratio  $r_2$  was found to be zero within experimental error. This indicates that the rate of addition of the trimethyl aconitate free radical to trimethyl aconitate monomer is insignificant (in this case this rate is zero) when compared to the rate of addition of this radical to monomer  $M_1$ . Similar interpretations apply to the values for the reactivity ratios involving triethyl aconitate. In addition it is to be noted that the values for triethyl aconitate and butadiene and triethyl aconitate and styrene are the same for the corresponding values of trimethyl aconitate and butadiene and trimethyl aconitate and styrene within experimental error. This supports the theoretical considerations that the nature of the alkyl group should not influence the reactivity of the carbon-carbon double bond of trialkyl aconitates in free radical initiated copolymerizations.

# ATTEMPTED SOLUTION COPOLYMERIZATION OF ACONITIC ACID WITH VINYL MONOMERS

In all attempts to copolymerize aconitic acid with vinyl monomers (Table VIII), 1 g. of aconitic acid, 1.5 ml. of *N,N*-dimethyl formamide, and 5 g. of vinyl monomer were used, unless otherwise noted. The polymerization temperature was at 60°C. in 4-oz. screw-cap bottles. Initiation of the polymerization was effected with 0.015 g. of azobisisobutyronitrile.

TABLE VIII

ATTEMPTED SOLUTION COPOLYMERIZATION OF ACONITIC ACID WITH VINYL MONOMERS AT 60°C.

Polymer Number	Monomer	Charge ratio <sup>a</sup>	Polymerization time, hours	Conversion, %	Unreacted aconitic acid, % <sup>b</sup>
262	Acrylonitrile	100/0	3.0	9.0	—
263	Acrylonitrile	50/50	3.0	8.9	100
264	Methyl acrylate	100/0	9.5	80	—
265	Methyl acrylate	83/17	9.5	80	100
268	Vinyl acetate	100/0	17.0	46	—
269	Vinyl acetate	83/17	17.0	46	100

<sup>a</sup> Charge ratio by weight, monomer/acetic acid.

<sup>b</sup> Determined by titration with 1 *N* sodium hydroxide.

These data indicate that no aconitic acid was incorporated into the polymers which were produced and that it did not interfere with the homopolymerizations of these vinyl monomers.

## BUTADIENE-TRIMETHYL ACONITATE COPOLYMERS

Trimethyl aconitate readily copolymerizes with butadiene in a modified Mutual recipe<sup>7</sup> at 30 and 50°C. When butadiene (Phillips Special Purity research grade) and trimethyl aconitate were copolymerized at 50°C. in a charge weight ratio of 80 parts of butadiene to 20 parts of trimethyl aconitate, conversions of 63–66% were obtained in 3.5 hours. At 30°C., 24 hours was required to obtain the same conversions with the same charge weight ratio. As the molar ratio of butadiene/trimethyl aconitate approaches one, the rate of copolymerization increases rapidly. This effect is shown in Table IX.

TABLE IX

BUTADIENE-TRIMETHYL ACONITATE COPOLYMERS

(Mutual recipe, potassium persulfate initiation at 30°C.)

Charging ratio in grams butadiene/trimethyl aconitate	Polymerization time, hours	Conversion %
60/40	19.0	78
50/50	19.0	91
40/60	19.0	100

An evaluation sample of copolymer was prepared from an 80/20 charging ratio of butadiene and trimethyl aconitate. To the standard Mutual recipe there was added 0.02 part of Daxad 11 (Dewey and Almy Chemical Company) and 0.0625 g. of Hooker's lauryl mercaptan was used as a modifier for each 20 g. of monomer mixture used. This charging ratio gave a copolymer containing 25% of combined trimethyl aconitate. The copolymer is described in Table X.

TABLE X

Polymer code number.....	X-720	Illinois No. 331
Monomers.....	Butadiene/styrene	Butadiene/trimethyl aconitate <sup>a</sup>
Monomer charging ratio.....	71/29	80/20
Temperature of polymerization, °C....	50	50
Time, hours.....	—	3.5
Conversion, %.....	68	66
% combined trimethyl aconitate.....	—	25
Gel, % (benzene solvent).....	3	2
DSV (benzene).....	2.12	2.31
Mooney viscosity (ML-4 at 212°F.)...	48	60

<sup>a</sup> ANALYSIS. Found: C 78.5; H 9.8.

TABLE XI

Tread-type recipe		Carcass-type recipe	
	Parts by wt.		Parts by wt.
Polymer.....	100	Polymer.....	100
EPC black.....	40	Statex.....	30
Zinc oxide.....	5	Red lead.....	2.5
Sulfur.....	2	Sulfur.....	2
Benzothiazyl disulfide....	1.75	Benzothiazyl disulfide....	1
		Circosol-2XH.....	20
<i>Total</i> .....	<i>148.75</i>	<i>Total</i> .....	<i>155.5</i>

TABLE XII

## STRESS-STRAIN DATA AT 77°F., TREAD-TYPE RECIPE

	Min. cured at 292°F.	Polymer code number	
		X-720	Illinois No. 331
300% modulus, p.s.i.....	25	530	860
	50	1100	1440
	100	1600	1930
Tensile strength, p.s.i.....	25	2500	4090
	50	3960	3540
	100	4090	3560
Elongation, %.....	25	820	730
	50	670	530
	100	550	460
Set, %.....	25	25	19
	50	19	13
	100	16	11

The copolymer was compounded according to the recipes in Table XI.

In Tables XII to XVI are tabulated the results obtained in stress-strain,<sup>8</sup> oil resistance,<sup>9</sup> Gehman low temperature,<sup>10</sup> and Goodrich hysteresis<sup>11</sup> evaluations. The values of X-720 (standard GR-S 1000 rubber), Perbunan 18, and Perbunan 26 (butadiene-acrylonitrile rubbers) are given for com-

TABLE XIII

OIL RESISTANCE DATA AFTER SEVEN-DAY IMMERSION AT 77°F., TREAD-TYPE RECIPE

	X-720	Illinois No. 331	Perbunan 18	Perbunan 26
Minutes cured at 292°F.....	60	50	50	50
% swell by volume				
ASTM Oil No. 1.....	30	6	6	0
ASTM Oil No. 2.....	37	13	9	6
ASTM Oil No. 3.....	95	60	16	9
Isooctane.....	82	48	23	9
60/40 isooctane/toluene.....	231	163	100	69
Special solvent <sup>a</sup> .....	231	163	95	69

<sup>a</sup> Composed of 15% xylene, 20% toluene, 5% benzene, and 60% isooctane.

TABLE XIV

GEHMAN LOW-TEMPERATURE DATA, TREAD-TYPE RECIPE

	X-720	Illinois No. 331
Minutes cured at 292°C.....	60	50
Degrees of twist (10 sec. Twist)		
-60°C.	3	3
-55	7	5
-50	48	9
-45	121	11
-40	142	16
-35	150	37
-30	155	78
-25		113
-20		133
-15		143
-10		149
- 5		—
0	166	153
+25	167	160
Temperature, minus °C.		
$T_2$	29	15
$T_5$	44	26
$T_{10}$	47	30
$T_{100}$	52	42
F.p.	54	40
Gage, inches.....	0.077	0.076



parison. Illinois 331 is better than the standard GR-S copolymer in oil resistance properties, but it is not as good as Perbunan 18 or Perbunan 26. The tensile properties of the butadiene-trimethyl aconitate copolymer are

TABLE XV  
STRESS-STRAIN DATA AT 77°F., CARCASS-TYPE RECIPE

	Min. cured at 280°F.	X-720	Illinois No. 331
300% modulus, p.s.i. ....	20	230	300
	40	450	480
	80	630	610
Tensile strength, p.s.i. ....	20	1650	1710
	40	1890	1610
	80	1460	1240
Elongation, %.....	20	950	860
	40	650	630
	80	490	480
Set, %.....	20	33	25
	40	18	13
	80	5	4

TABLE XVI  
GOODRICH HYSTERESIS DATA, CARCASS-TYPE RECIPE

	Min. cured at 280°F.	X-720	Illinois No. 331
Shore A hardness. ....	60	40	39
	90	39	40
	120	40	40
Initial compression, %.....	60	32.0	31.8
	90	30.2	31.4
	120	31.4	31.5
Temperature rise, °F.....	60	29	20
	90	25	19
	120	25	20
Set, %.....	60	16.1	9.0
	90	7.2	5.9
	120	8.1	5.3

comparable to those of the standard GR-S copolymer, but inferior with regard to low-temperature flexibility properties. The temperature-rise properties of this butadiene-trimethyl aconitate copolymer were possibly better than those of the standard GR-S copolymer made at 50°C.

We are indebted to Dr. L. F. Martin, Head, Sugarcane Products Section, Southern Utilization Research Branch, United States Department of Agriculture, for generous supplies of pure trimethyl aconitate and triethyl aconitate. Mr. John W. Johnson, Jr., was aided in his graduate study by a Fellowship from National Medical Fellowships, Incorporated, through funds appropriated by the National Foundation for Infantile Paralysis.

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### Synopsis

Reactivity ratios have been determined for the copolymerization of trimethyl aconitate with butadiene, styrene, acrylonitrile, and vinyl chloride, and for triethyl aconitate with butadiene and styrene. The values obtained indicate that trialkyl aconitates are very reactive in free radical initiated copolymerizations. No copolymerizations of aconitic acid were realized. A copolymer of butadiene and trimethyl aconitate has been prepared in a modified Mutual recipe at 50°C. The copolymer shows fair oil resistance and tensile properties but the low-temperature properties are poor. The temperature rise of trimethyl aconitate copolymer is possibly better than that of standard GR-S made at 50°C.

### Résumé

On a déterminé les rapports de réactivité pour la copolymérisation de l'aconitate de triméthyle avec le butadiène, le styrène, l'acrylonitrile et le chlorure de vinyle, et de l'aconitate de triéthyle avec le butadiène et le styrène. Les valeurs obtenues indiquent que les aconitates d'alcyle sont très réactionnels dans les copolymérisations initiées par des radicaux. On n'a pu réaliser de copolymérisation de l'acide aconitique. Un copolymère de butadiène et d'aconitate de triméthyle a été préparé par un recipe Mutuel modifié à la température de 50°C. Le copolymère manifeste une bonne résistance aux huiles et de bonnes propriétés de tension; par contre les propriétés à basse température sont déficientes. L'échauffement du copolymère d'aconitate de triméthyle est vraisemblablement meilleur que celui du GR-S standard préparé à 50°C.

### Zusammenfassung

Es wurden Reaktivitätsverhältnisse für die Copolymerisation von Trimethyлаconitat mit Butadien, Styrol, Acrylonitril und Vinylchlorid, und von Triäthylaconitat mit Butadien und Styrol bestimmt. Die erhaltenen Werte zeigen an, dass Trialkylaconitate in durch Freiradikale initiierten Copolymerisationen sehr reaktiv sind. Es wurden keine Copolymerisationen von Aconitsäure erreicht. Ein Copolymer von Butadien und Trimethyлаconitat wurde nach einer abgeänderten Mutual-Vorschrift bei 50°C hergestellt. Das Copolymer zeigt annehmbare Öl-Widerstands- und Spannungseigenschaften, aber die Eigenschaften bei niedriger Temperatur sind schlecht. Die Temperaturerhöhung von Trimethyлаconitat-Copolymer ist möglicherweise besser als die von Standard GR-S, welches bei 50°C hergestellt wurde.

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