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EVALUATION OF ALTERNATE VANADIUM COMPOUNDS FOR USE IN FIBERGLAS--ETC(U)
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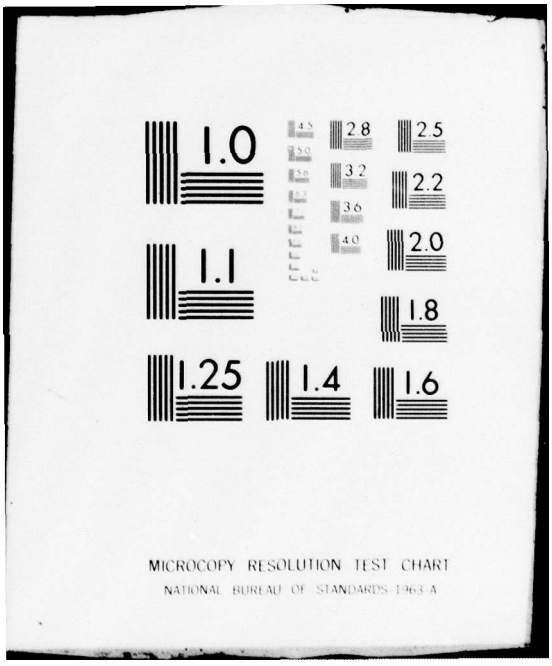
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CR 79.013

CIVIL ENGINEERING LABORATORY
Naval Construction Battalion Center
Port Hueneme, California

Sponsored by
NAVAL FACILITIES ENGINEERING COMMAND

EVALUATION OF ALTERNATE VANADIUM COMPOUNDS FOR USE IN FIBERGLASS-REINFORCED PLASTIC SOIL SURFACING

September 1979

An Investigation Conducted by
THE DOW CHEMICAL COMPANY
Walnut Creek, California

N68305-77-C-0005

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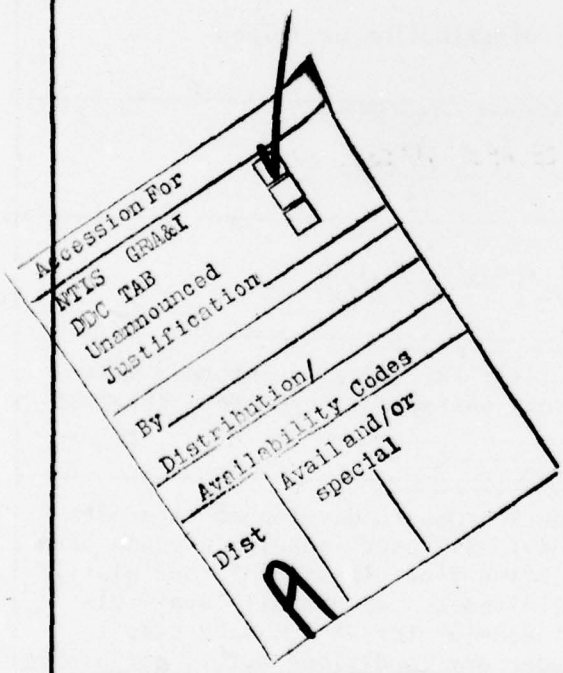

EVALUATION OF ALTERNATE VANADIUM COMPOUNDS FOR USE IN FIBERGLASS-REINFORCED PLASTIC SOIL SURFACING

By

Theo John West

THE DOW CHEMICAL COMPANY

Walnut Creek, California

March 28, 1979

ABSTRACT

The purpose of this investigation was to develop an alternate vanadium compound for the previously used vanadium neodecanoate (Vanadium Ten Cem) in formulating fiberglass-reinforced plastic surfacings. The study was limited to commercially available products. It was found that Accelerator VN-2 can be used in place of Vanadium Ten Cem under dry conditions but is not usable under wet conditions. Vanadyl and vanadium acetylacetonate and vanadyl naphthenate were also evaluated and were ineffective under wet conditions and inferior to Accelerator VN-2 under dry conditions. The flexural strengths of laminates prepared using Accelerator VN-2 at 32°F and 75°F substantially exceeded contract specifications.

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GLOSSARY

Port Hueneme Resin	Resin received from Port Hueneme, Selectron RS50338 (PPG Industries, Inc.) Resin SR3704 plus 400 ppm dinitrophenol and 200 ppm p-toluhydroquinone.
Styrene	Styrene, 99% inhibited 10-15 ppm p-tert-butylcatechol (Aldrich Chemical Company, Inc.).
CHP	Cumene hydroperoxide (Lucidol Division, Pennwalt Corporation).
DDM	LUPERSOL® DDM, a 60 percent solution of methyl ethyl ketone peroxide in dimethyl-phthalate (Lucidol Division, Pennwalt Corporation).
DMA	N,N-Dimethylaniline.
DMT	N,N-Dimethyl-p-toluidine (Aldrich Chemical Company, Inc.).
VN-2	Accelerator VN-2 (Interstab Chemicals, Inc.).
VY	Vanadyl acetylacetonate (Mooney Chemicals, Inc.).
VAY	Vanadium acetylacetonate (Mooney Chemicals, Inc.).
VAN	Vanadyl naphthenate (ICN K&K Laboratories, Inc.).
MEKP	Methyl ethyl ketone peroxide.
FABMAT® C-4020	Fiberglass mat composed of woven roving and chopped strand (Fiber Glass Industries, Inc.).
phr	Parts per hundred parts of resin.

SECTION I

INTRODUCTION

The object of the work is to test and evaluate an alternate vanadium compound for use in constructing fiberglass-reinforced plastic soil surfacing. The work was funded by the Civil Engineering Laboratory, Port Hueneme, California, under Phase II of Contract No. N68305-77-C-0005.

The Government has previously developed a resin formulation having a five year shelf life using: a polyester resin, cumene hydroperoxide and a promoter composed of N,N-dimethyl-p-toluidine and vanadium neodecanoate. The manufacturer of the vanadium neodecanoate (Vanadium Ten Cem) has discontinued this product. This compound is essential to the Marine Corps capability to construct expedient flexible surfacings in amphibious landing operations under all environmental conditions. Previous work by S. S. Drake[1], et al., H. E. Filter[2], et al., and T. J. West[3] has shown that resin cure to the required strength, under adverse environmental conditions, has only been possible by using Vanadium Ten Cem as a catalyst.

One of the objectives of this investigation was to investigate a vanadium compound, Accelerator VN-2, produced by Akzo Chemie and distributed in this country by Interstab Chemicals, Inc. The work of S. S. Drake[1] had shown that this compound might be a viable replacement for the Vanadium Ten Cem. Also, suppliers of other organic vanadium compounds were contacted and samples of promising commercial products were obtained. The short nature of this contract (200 hours of work) precluded an intensive evaluation. Therefore, the products evaluated were limited to those that would have representative valences and solubility in order to obtain as wide a range of chemical properties as possible.

The work is specifically addressed to the development of a vanadium compound to be used in the construction of a fiberglass-reinforced plastic surfacing for soil where the ambient air temperature is between 32°F and 120°F and the fiberglass matting is completely wet with water (such as from a rainstorm) and the underlying soil is completely saturated with water.

[1]Civil Engineering Laboratory, "Improved Chemical Components for Formulating Fiberglass-Reinforced Plastic Soil Surfacings," by S. S. Drake, H. E. Filter, D. L. Stevens, CR 77.017, (May 1977, The Dow Chemical Company).

[2]Kirtland AFB, "Expedient Surfacing Materials," by H. E. Filter and S. S. Drake, AFWL-TR-73-70, (October 1973, The Dow Chemical Company).

[3]Civil Engineering Laboratory, "Fabrication of Fiberglass Reinforced Plastic Surfacing Under Wet Conditions," by T. J. West, CR 78.018, (September 1978, The Dow Chemical Company).

SECTION II

TEST PROCEDURES

A. REACTIVITY STUDIES

Reactivity testing consisted of determining the gel time, time to peak exotherm, temperature at peak exotherm, degree of cure by hardness (Barcol #934). Determinations were made with a Sunshine gel tester and with a time/temperature recorder.

Samples for the Sunshine gel tests were prepared by thoroughly mixing 10 grams of properly catalyzed resin and pouring the mixture into the Sunshine gel tube to the specified level. The switch controlling the spindle and timer was turned on and the gel time was recorded from the timer on the Sunshine tester.

Samples for the exothermic test were prepared by mixing 20 grams of properly catalyzed resin and pouring the resin in a 2-inch diameter aluminum foil dish. An iron-constantan thermocouple was imbedded in the resin and attached to a temperature recorder. From the recorder output, time to peak exotherm and temperature rise were determined.

B. PREPARATION AND MECHANICAL TESTING OF GLASS LAMINATES

Laminates were prepared using FABMAT C-4020 at a resin to glass ratio of 60 to 40 by weight. This was done using two plies of 6 inch by 9 inch FABMAT C-4020. The first ply of fiberglass was placed in an adjustable frame made of 0.5 inch thick by 2 inches wide Teflon of the appropriate lengths to contain the mat and was located over a Mylar sheet. The frame was held in place by lead weights. This frame was used to insure that when the laminates were prepared under wet conditions, the water did not run off. The predetermined amount of resin and catalyst at the appropriate temperature was then distributed over the fiberglass mat by pouring, and then rolled with a parallel grooved roller until the resin was forced into the mat. In the case of the laminates prepared under wet conditions, the resin was allowed to exotherm before adding the next layer of FABMAT C-4020 and repeating the procedure. This was done to simulate field fabrication. The laminates prepared under wet conditions were cured under from 1 mm to 3 mm (0.039 in. to 0.118 in.) of water, depending on the saturation of the mats. Each laminate was cured at the appropriate temperature and then cut into test strips and tested on an Instron tester for flexural strength as specified in the ASTM procedure.

SECTION III

REACTIVITY STUDIES OF VANADIUM COMPOUNDS

The resin used in this work was Selectron RS50338 obtained from the Civil Engineering Laboratory, Port Hueneme, California. This vinyl ester resin is stabilized by the addition of 400 ppm of dinitrophenol and 200 ppm of p-toluhydroquinone resulting in a resin shelf-life of greater than five years. This long shelf-life results in low resin reactivity and precludes the use of normal catalyst systems (see the work of Drake[1] et al. and Filter[2] et al.). In view of the short nature of the Phase II contract (200 hours), the catalyst component study was limited to vanadium compounds. The following were tested: Accelerator VN-2, vanadyl acetylacetonate, vanadium acetylacetonate and vanadyl naphthenate. Catalyst optimization studies were made for all of the compounds tested using cumene hydroperoxide and DMT (N,N-dimethyl-p-toluidine). In the case of VN-2 methyl ethyl ketone peroxide (LUPERSOL DDM), t-butyl perbenzoate and dimethyl aniline were also tested.

The catalyst optimization studies for cumene hydroperoxide (CHP) and Accelerator VN-2 are given in Table 1. When VN-2 and cumene hydroperoxide are used as the sole catalyst components the reactivity is low but good cures are attained (see Figure 1). No clearly defined optimum ratio of CHP to VN-2 was found; but good cures were obtained at 6.7:1 and 4:1 (Barcol 44), and satisfactory Barcol hardness was obtained at 2:1. The Accelerator VN-2 has a wider range than Vanadium Ten Cem but since the composition range for both products is adequate, no advantage results. The gel time can also be controlled by the use of N,N-dimethyl-p-toluidine (DMT) with VN-2 as in the case of Vanadium Ten Cem (Table 1). The addition of DMT to the VN-2 is necessary for a fast cure. The ratio of VN-2 to DMT is not critical (see Table 1 and Figure 2). The Accelerator VN-2 is more sensitive to DMT than Vanadium Ten Cem and although a good cure can be obtained over a broad range, the use of a VN-2 to DMT ratio of 1:1 appears to be the most satisfactory.

The gel time can also be controlled by manipulating the CHP/VN-2/DMT ratio as well as the total catalyst concentration and further controlled by using Lupersol DDM (methyl ethyl ketone peroxide) as a retarder (Table 2). The influence of VN-2 to DMT at a ratio of 1:1 is shown in Figure 3. Promoter contents below 0.3 phr results in too long an exotherm time, although a satisfactory cure is obtained. A CHP/VN-2/DMT ratio of 1/0.15/0.15 appears to be a reasonable ratio of catalyst components (Figure 4). Increasing the ratio to 1/0.25/0.25 or higher results in increased reactivity (decreased gel time) but the resin cured too violently. The use of this catalyst composition results in a 25 percent increased catalyst requirement as compared to Ten Cem at 75°F for a 10 minute gel time (Figure 5). The Accelerator VN-2 is more temperature sensitive than Vanadium Ten Cem. For example, by decreasing the temperature to 32°F, a 160 percent increase in catalyst is required to achieve the same gel time of 10 minutes when compared to Ten Cem (Figure 5). Good cure was obtained at 32°F and at 120°F (see Table 3) although again more catalyst was required for the VN-2, 81 percent more than for Ten Cem.

Catalyst studies were also made of Accelerator VN-2 with methyl ethyl ketone peroxide and t-butyl perbenzoate. The t-butyl perbenzoate was ineffective (see Table 4) and the methyl ethyl ketone peroxide was not as active as cumene hydroperoxide (see Table 4).

The influence of water on the reactivity of VN-2 was determined by adding 20 grams of water to the 20 grams of resin in the aluminum dish and allowing the resin to cure under water. As was expected, it was found that this resulted in increased time to exotherm, due to increased heat capacity, and also decreased peak temperature. What was not expected was that the resin did not cure at the water interface (see Table 1). In order to confirm the deleterious effect of water on the VN-2 catalyst system, a laminate was attempted with Fabmat C-4020 containing 74 percent of a 1 percent Triton X100 solution. The resin did not cure (see Table 5), but satisfactory cures were obtained under these conditions using Vanadium Ten Cem in the previous work by West[3].

Catalyst optimization studies were made for cumene hydroperoxide with vanadyl acetylacetonate (V^{+4}), vanadium acetylacetonate (V^{+3}), and vanadyl naphthenate. None of these compounds had as broad a ratio of CHP to promoter as Accelerator VN-2. In every case the optimum ratio was higher, e.g., 1:0.5 for vanadyl acetylacetonate (Table 6 and Figure 6), 1:0.5 for vanadium acetylacetonate (Table 7 and Figure 6), and 1:1 for vanadyl naphthenate (Table 8 and Figure 7). In the case of vanadium naphthenate, the activity was sufficiently great that a gel time of six minutes or less could be obtained without using N,N-dimethyl-p-toluidine. Neither the vanadyl or vanadium acetylacetonate were sensitive to the DMT level (see Figure 8) and CHP/V compd/DMT ratios of 1/0.5/0.2 and 1/0.5/0.35, respectively, gave good cures and control of gel time. The vanadyl naphthenate, however, was very sensitive to DMT and good cures could not be obtained at a catalyst ratio greater than 1/1/0.1 (see Figure 8 and Table 8). Tests were made of the effect of water by adding 20 grams of water to 20 grams of resin in an aluminum foil dish. As in the case of Accelerator VN-2, the resin did not cure at the resin water interface with any of these vanadium compounds (see Tables 6, 7, and 8).

The effect of temperature on the reactivity and cure was determined at 32°F, 75°F, and 120°F for vanadyl and vanadium acetylacetonate (see Table 3). These results together with those for Accelerator VN-2 are presented in Figure 9. The results for VN-2 and Vanadium Ten Cem are compared in Figure 10. As can be seen in Figure 9, at 32°F and 120°F there is little difference (14%) in the amount of catalyst required at these temperatures between the Accelerator VN-2 and the vanadium and vanadyl acetylacetonates. At room temperature, about 37 percent more catalyst is required with vanadyl acetylacetonate as compared to VN-2; and about 10 percent more vanadium acetylacetonate catalyst is required than VN-2 catalyst. Although satisfactory cures did result with all of the vanadium compounds when used with CHP and DMT, the exotherm was significantly less for the vanadium and vanadyl acetylacetonates; and with vanadium naphthenate the exotherm was at least 60°C less than for Accelerator VN-2 and Vanadium Ten Cem. This indicates that in the preparation of laminates, trouble might be encountered in obtaining a good cure.

SECTION IV

PHYSICAL AND MECHANICAL PROPERTIES

Fiberglass reinforced composites were made using RS50338 resin and Fabmat C-4020 at the recipe of 60 percent resin and 40 percent fiberglass. Laminates were made at 32°F and 75°F using Accelerator VN-2. The results obtained are presented in Table 5. Laminates were prepared whose flexural strengths exceeded those specified in the contract. However, it was found that if the Sunshine gel time was in the order of ten minutes, the flexural strengths were less than 25,470 psi which did not meet specification. This is not true when Vanadium Ten Cem was used. The reason for this is due to the difference in the temperature behavior of Ten Cem and VN-2. In the case of VN-2, it was observed that the exotherm in the Fabmat C-4020 did not follow the same rate behavior as in the Sunshine gel time or the surplus gel time specimens (see Figure 11). The exotherm rate decreases more rapidly in the laminate specimens, because of the additional heat capacity, than it does in the gel-time specimens; thus a poor cure is obtained. This was not the case with Ten Cem where this behavior does not occur.

SECTION V

MISCELLANEOUS

A. EFFECT OF STYRENE

The effect of the addition of styrene to Port Hueneme resin (RS50338) was also investigated using Accelerator VN-2. The addition of 20 percent styrene to the Port Hueneme resin resulted in only a slight decrease in reactivity (see Table 1 and Figure 12) but with no change in slope. In the previous investigation using Vanadium Ten Cem, the addition of styrene to Port Hueneme resin resulted in a pronounced change in catalyst activity as shown by a change in slope of the gel time vs. catalyst concentration.

B. SOURCE AND PROPERTIES OF THE COMPOUNDS TESTED

Interstab Chemicals, Inc.
500 Jersey Avenue
P. O. Box 638
New Brunswick, N.J. 08903

Accelerator VN-2 is a clear light green liquid containing 0.2% Vanadium having a density of 1.15-1.16. The flash point is 92°C (C.O.C.), and the material is stable under normal storage and handling conditions according to Interstab. It was not tested for shelf life due to the short nature of the contract.

Mooney Chemicals, Inc.
2310 Scranton Road
Cleveland, Ohio 44113

Acetylacetonates

	Vanadium	Vanadyl
Formula	$V(C_5H_7O_2)_3$	$VO(C_5H_7O_2)_2$
Color of crystals	Green-brown	Prossian Blue
% Vanadium	14.7	19.3
Molecular Weight	348.25	265.15
Melting Point	182°C	250°C
Solubility g/100 ml, 25°C		
Benzene	16.3	0.9
Methyl alcohol	14.6	6.4

ICN K&K Laboratories, Inc.
121 Express Street
Plainview, N.Y. 11803

Vanadyl naphthenate, black viscous liquid, 3% vanadium.

SECTION VI

CONCLUSIONS

Accelerator VN-2 can be used in place of Vanadium Ten Cem under dry conditions to fabricate fiberglass-reinforced plastic soil surfacings. It is not usable under wet conditions nor does it have the rate predictability or the versatility of Vanadium Ten Cem when used under dry conditions. None of the other vanadium compounds tested could be used under wet conditions and they were inferior to Accelerator VN-2 under dry conditions. Vanadium Ten Cem has the unique property of being effective under adverse environmental conditions. This property was not shown by any of the vanadium compounds evaluated.

SECTION VII

RECOMMENDATIONS

A viable substitute for Vanadium Ten Cem for use under wet conditions has not been found. An obvious approach would be to continue the evaluation of vanadium compounds. However, in view of the unique properties of Vanadium Ten Cem combined with the urgency to have a resin system in the near future, the following recommendations are made:

1. The structure of Vanadium Ten Cem (vanadium neodecanoate) be determined.

2. Develop a method of synthesis for the vanadium neodecanoate of the proper valence.

3. Then manufacture the product either "in house" or contract it to a custom manufacturer.

TABLE 1

ACTIVITY OF ACCELERATOR VN-2

Test No.	Catalyst - phr			Gel Time Minutes	Exotherm		Barcol Hardness Top/Bottom	Remarks
	CHP[1]	VN-2[2]	DMT[3]		△T°C	Time Minutes		
1	2.0	2.0	--	11.9	110	18.5	9-10/10-15	
2	2.0	1.40	--	14.5	118	23.5	30-36/10-22	
3	2.0	1.0	--	17.4	143	27.8	35-40/32-40	
4	2.0	0.5	--	29.4	146	47.5	41-46/43	
5	2.0	0.3	--	47.1	136	74.0	43-45/42	
6	1.0	0.5	0.5	3.45	>182	4.5	Shattered Hard	
7	1.0	0.5	0.25	9.3	165	12.4	Shattered Hard	
8	1.0	0.25	0.25	7.1	174	9.8	Shattered Hard	
9	1.0	0.25	0.125	17.4	162	25.0	Shattered Hard	
10	1.0	0.25	0.375	6.4	183	7.6	Shattered Hard	
11	1.0	0.094	0.094	30.1	127	64.2	Shattered Hard	
12	1.0	0.094	0.094	34.4	125	61.2	48/45	
13	1.0	0.156	0.156	12.0	169	18.5	48/45	
14	0.938	0.188	0.094	31.0	147	53.3	49/44-47	
15	1.875	0.375	0.188	10.9	175	13.7	48	
16	2.813	0.562	0.281	6.1	183	7.5	48	
17	2.0	0.30	0.20	7.5	172	10.3	48	
18	0.938	0.141	0.0937	30.4	129	53.7	47/43-46	
19	1.25	0.188	0.125	15.2	164	28.7		
20	1.0	0.15	0.10	26.3	146	43.3	46/43	
21	1.666	0.25	0.25	5.85	>186	7.7	48	
22	1.0	0.15	0.15	15.9	152	25.6	45/43	
23	0.729	0.109	0.109	35.7	116	63.7	43/43	
24	0.625	0.156	0.156	20.3	151	29.8	40-47/44-47	
25	0.50	0.25	0.25	12.4	155	16.7	43-45/43	

(Continued)

TABLE 1 (Continued)

Test No.	Catalyst - phr CHP[1]	VN-2[2]	DMT[3]	Gel Time Minutes	Exotherm △T°C	Exotherm Time Minutes	Barcol Hardness Top/Bottom	Remarks
			Next two tests with 20 grams of water in dish					
26	0.75	0.188	0.188	15.2	90	36.0	0/32-38	Layer on top that did not cure but hard underneath.
27	1.25	0.312	0.312	6.9	127	12.8	0/32-45	" " " " " " "
			Next two tests RS50338 + 20% Styrene					
28	0.688	0.172	0.172	18.7	161	31.7	45	
29	1.00	0.25	0.25	8.5	186	11.8	45	
	CHP[1]	VN-2[2]	DMA[4]					
30	1.00	0.25	0.25	11.7	175	17.3	48	
31	1.00	0.50	0.50	6.3	184	7.7	48	
32	1.00	0.156	0.156	18.8	153	33.2	49/43-49	

[1]CHP - Cumene Hydroperoxide

[2]VN-2 - Accelerator VN-2

[3]DMT - N,N-dimethyl-p-toluidine

[4]DDM - LUPERSOL® DDM - 60% methyl ethyl ketone peroxide in dimethylphthalate.

TABLE 2

METHYL ETHYL KETONE PEROXIDE AS RETARDER

Test No.	Catalyst - phr			Gel Time Minutes	Exotherm		Barcol Hardness Top/Bottom	Remarks
	CHP[1]	VN-2[2]	DMT[3]	DDM[4]		△T°C	Time Minutes	
1	2.812	0.562	0.212	0.2	5.4	>186	7.0	Hard	
2	2.812	0.562	0.212	0.6	4.3	>186	5.5	Hard	
3	2.812	0.562	0.212	1.219	3.6	>186	4.5	Hard	
4	2.812	0.562	0.212	1.969	7.1	>186	8.3	Hard	
5	2.812	0.562	0.212	2.812	8.0	>186	9.7	Hard	
6	1.666	0.25	0.25	1.0	37.0	171	51.2	Hard	
7	1.666	0.25	0.25	0.625	12.5	177	14.4	Hard	
8	1.666	0.25	0.25	0.469	5.7	>186	6.8	Hard	
9	1.0	0.156	0.156	0.188	13.2	151	22.8	49/46	
10	1.0	0.156	0.156	0.375	18.0	182	22.7	48/43	
11	1.0	0.156	0.156	0.562	43.5	172	50.8	48	
12	3.0	0.45	0.45	1.0	7.7	>186	8.0	Hard	
13	3.0	0.45	0.45	1.25	14.0	>186	14.5	Hard	
14	3.0	0.45	0.45	1.5	19.0	>186	21.0	Hard	

[1]CHP - Cumene hydroperoxide

[2]VN-2 - Accelerator VN-2

[3]DMT - N,N-dimethyl-p-toluidine

[4]DDM - LUPERSOL® DDM; 60% methyl ethyl ketone peroxide in dimethylphthalate.

TABLE 3

EFFECT OF TEMPERATURE

Test No.	Temp. °F	Catalyst - phr			Gel Time Minutes	Exotherm		Barcol Hardness Top/Bottom	Remarks
		CHP[1]	VN-2[2]	DMT[3]		△T°C	Time Minutes		
1	32	2.188	0.328	0.328	40.1	150	36.4	36	
2	32	3.125	0.469	0.469	21.5	174	22.5	30	
3	32	4.375	0.656	0.656	12.4	171	11.7	30	
4	32	4.375	0.656	0.438	22.7	167	15.5	Shattered	
5	32	3.125	0.469	0.312	46.0	121	46.5	45/36	
6	120	0.625	0.094	0.094	13.0	---	---	---	
7	120	0.844	0.127	0.127	5.6	---	---	---	
		Vanadium Acetylacetonate							
		CHP[1]	VAY[4]	DMT[3]					
8	32	3.75	1.875	1.312	7.5	177	7.7	32-38/20	
9	32	2.438	1.219	0.853	22.5	154	24.7	44/20-30	
10	120	0.812	0.406	0.284	4.3	---	---	---	
11	120	0.536	0.266	0.188	9.4	---	---	---	
		Vanadyl Acetylacetonate							
		CHP[1]	VY[5]	DMT[3]					
12	32	3.438	1.719	1.719	~2.2	---	~2.2	---	
13	32	2.875	1.438	1.438	5.3	141	7.8	42/8	
14	32	2.5	1.25	1.25	15.0	71	22.2	36/0	
15	120	0.562	0.281	0.281	7.4	---	---	---	
16	120	0.375	0.188	0.188	14.5	---	---	---	

[1]CHP - Cumene hydroperoxide

[2]VN-2 - Accelerator VN-2

[3]DMT - N,N-dimethyl-p-toluidine

[4]VAY - Vanadium Acetylacetonate in methyl alcohol (1.4% Vanadium)

[5]VY - Vanadyl Acetylacetonate in methyl alcohol (1.4% Vanadium)

TABLE 4

EFFECT OF METHYL ETHYL KETONE PEROXIDE AND t-BUTYL PERBENZOATE

Test No.	Catalyst - phr DDM[1]	VN-2[2]	DMT[3]	Gel Time Minutes	Exotherm △T°C	Exotherm Time Minutes	Barcol Hardness Top/Bottom	Remarks
1	2.0	2.0	---	13.4	87	19.7	20-27/0	
2	2.0	1.0	---	15.6	113	21.3	32-35/17-28	
3	2.0	0.5	---	25.1	114	38.2	41-45/35-41	
4	2.0	0.25	---	60.5	53	111.0	34-42/26-38	
5	1.0	0.25	0.2	16.9	156	19.2	43-45/39-44	
6	1.0	0.25	0.5	33.8? Viscous but not a gel				
7	1.0	0.25	0.25	>30.0	---	≈40.0	45-48/39	
8	1.0	0.25	0.2	26.5	166	29.7	45-47/33-39	
9	1.0	0.25	0.15625	9.5	166	11.5	41-45/32-36	
10	1.0	0.25	0.125	9.1	158	12.2	43-45/33-39	
11	1.0	0.25	0.0625	11.5	129	19.5	45-46/38-40	
12	1.0	0.25	0.20	14.3	171	16.5	42-44/38	
13	0.75	0.1875	0.0938	9.2	141	13.5	43-45/36-39	
14	2.0	0.5	0.25	7.1	185	7.7	45/43	
	Next two tests made with t-butyl perbenzoate							
	TBP[4]	VN-2	DMT					
15	2.0	0.5	---	Did not gel				
16	2.0	2.0	---	Did not gel				

[1]DDM - LUPERSOL® DDM; 60% methyl ethyl ketone peroxide in dimethylphthalate

[2]VN-2 - Accelerator VN-2

[3]DMT - N,N-dimethyl-p-toluidine

[4]t-butyl perbenzoate

TABLE 5

PROPERTIES OF LAMINATES

Test No.	Catalyst - phr CHP[1]	VN-2[2]	DMT[3]	DDM[4]	Gel Time Minutes Surplus	Exotherm Minutes	Resin %	Barcol Hardness Top/Bottom	Flexural Strength PSI
1	0.84	0.21	0.21	---	13	63	57	20-22/8	18,850
2	1.20	0.30	0.30	---	6.5	17	59	45-50/48-50	50,640
3	1.80	0.27	0.27	---	5	14.3	60	42-45/50-52	52,350
4	1.13	0.17	0.17		14	65	57	26/33	25,470
5	1.40	0.21	0.21	---	11	43	59	33/33	32,920
				Next test all components at 32°F					
6	4.636	0.695	0.695	---	---	15	57	40/-	40,910
				Next test mat contains 74% of a 1% solution of Triton X100					
7	3.0	0.45	0.45	1.2	8.5	Did not cure			

[1]CHP - Cumene hydroperoxide

[2]VN-2 - Accelerator VN-2

[3]DMT - N,N-dimethyl-p-toluidine

[4]DDM - LUPERSOL® DDM; 60% methyl ethyl ketone peroxide in dimethylphthalate

TABLE 6

ACTIVITY OF VANADYL ACETYLACETONATE

Test No.	Catalyst - phr CHP[1]	VY[2]	DMT[3]	Gel Time Minutes	Exotherm △T°C	Exotherm Time Minutes	Barcol Hardness Top/Bottom	Remarks
1	2.0	2.0	---	9.6	109	16.8	38-43/28-32	
2	2.0	1.0	---	15.8	104	35.3	44-46/42	
3	2.0	0.5	---	42.5	---	>70	0	
4	2.0	0.3	---	>91.0	---	>120	0	
5	1.0	0.1	0.1	Did not gel in 60 minutes				
6	1.0	0.5	0.2	9.6	114	27.0	43-45/42-43	
7	1.0	0.25	0.125	54.8	---	---	0	
8	0.625	0.3125	0.3125	36.1	>60	---	0	
9	1.0	0.5	0.2	12.1	95	36.8	43/41	Check of 7
10	1.0	0.5	0.5	7.8	130	18.3	43/41	
11	0.75	0.375	0.375	18.3	64	58.5	35-39/23-25	
12	1.0	0.5	0.2	12.7	100	43	45/40	Recheck of 9 after 12 days
				Next test with 20 grams of water in dish				
13	0.9375	0.4688	0.4688	13.0	23	43	Goo on top and 0 Barcol on bottom	

[1]CHP - Cumene hydroperoxide

[2]VY - Vanadyl acetylacetonate in methyl alcohol (1.4% Vanadium)

[3]DMT - N,N-dimethyl-p-toluidine

TABLE 7

ACTIVITY OF VANADIUM ACETYLACETONATE

Test No.	Catalyst - phr CHP[1]	VAY[2]	DMT[3]	Gel Time Minutes	Exotherm △T°C	Exotherm Time Minutes	Barcol Hardness Top/Bottom	Remarks
1	2.0	2.0	---	6.5	112	12.8	35/22	
2	2.0	1.0	---	14.4	99	36.2	39-43/40	
3	2.0	0.6875	---	25.4	38	72.0	6-10/0	
4	1.0	0.5	0.2	16.3	105	40.5	42-43/34-41	
5	1.0	0.5	0.35	15.1	122	33.5	42-45/44-46	
6	1.0	0.5	0.35	14.6	123	33.8	43-46/41-42	
7	1.0	0.5	0.5	14.8	118	36.3	43-47/43	
8	1.344	0.672	0.469	7.8	146	15.2	44-47/40-42	
				Next test with 20 grams of water in dish				
9	1.125	0.5625	0.5625	9.1	33	32.0	Tacky on top and Barcol 0 on the bottom.	

[1]CHP - Cumene hydroperoxide

[2]VAY - Vanadium acetylacetonate in methyl alcohol (1.4% vanadium)

[3]DMT - N,N-dimethyl-p-toluidine

TABLE 8

ACTIVITY OF VANADYL NAPHTHENATE

Test No.	Catalyst - phr CHP[1]	VAN[2]	DMT[3]	Gel Time Minutes	Exotherm ΔT°C	Exotherm Time Minutes	Barcol Hardness Top/Bottom	Remarks
1	2.0	2.0	---	6.1	100	33.5	42-43/37-38	
2	2.0	1.0	---	24.6	---	>70	0/0	Next day 20-22/15-18
3	2.0	1.5	---	9.7	---	>120	0/0	Next day 20-22/23-26
4	2.0	2.5	---	3.3	90	16.0	33-34/25-32	
5	1.0	1.0	0.5	1.9	120	3.8	0/0	
6	1.0	1.0	0.25	3.4	121	7.5	0/0	
7	1.0	1.0	0.1	7.6	99	28.0	40/34	
8	0.75	0.75	0.1875	6.2	105	14.7	26-38/3-6	
9	0.625	0.625	0.0625	16.6	41	67.0	24-25/0	
				Next two tests with 20 grams of water in dish				
10	2.0	2.0	---	6	---	39	Next day 0/25	
11	1.0	1.0	0.1	7.6	23	33.0	0/0	

[1]CHP - Cumene hydroperoxide

[2]VAN - 5 parts Vanadium Naphthenate + 1 part toluene

[3]DMT - N,N-dimethyl-p-toluidine

FIGURE 1

CATALYST COMPONENT STUDY
EFFECT OF ACCELERATOR VN-2

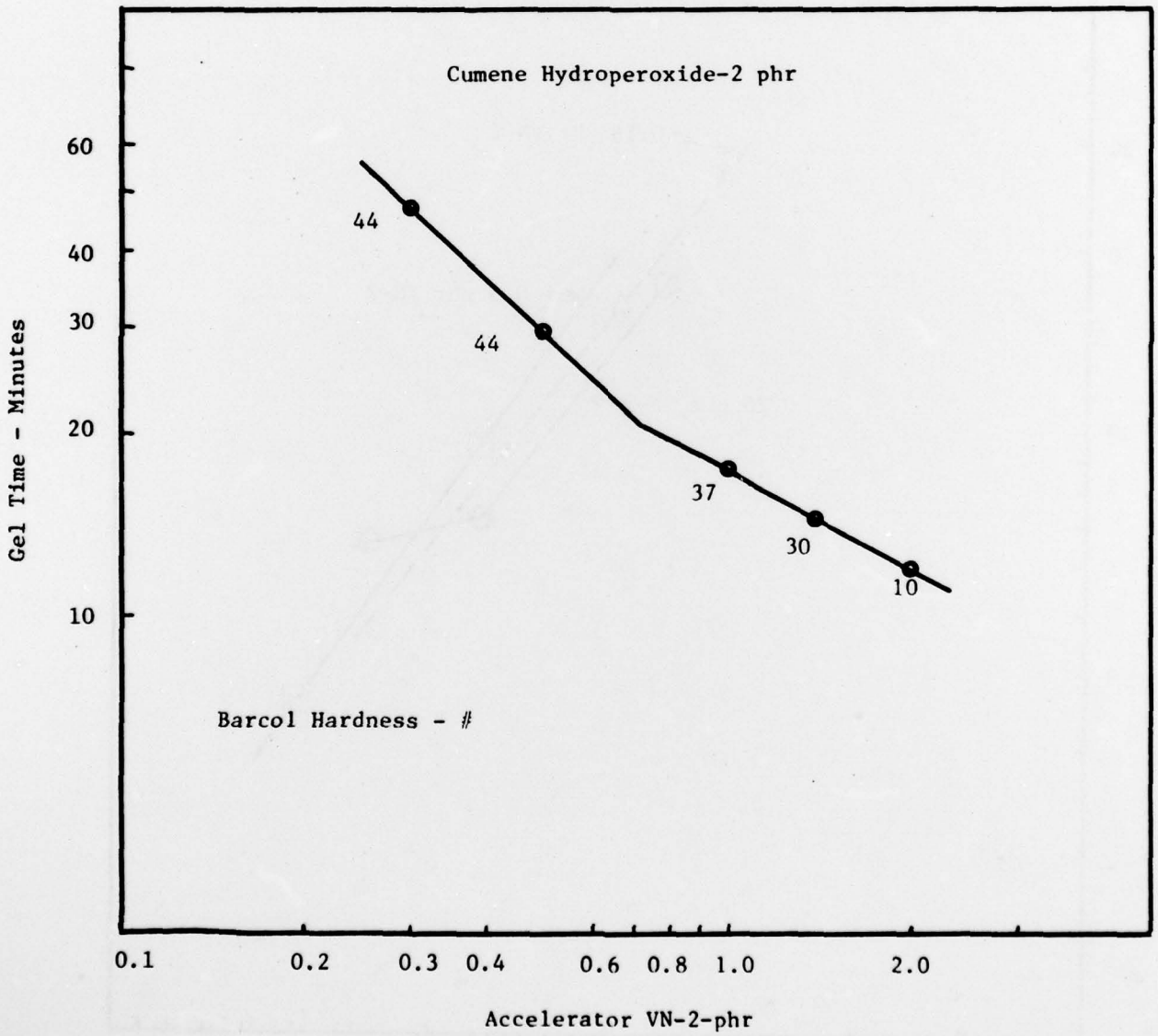

FIGURE 2

CATALYST COMPONENT STUDY
EFFECT OF N,N-DIMETHYL-P-TOLUIDINE ON ACCELERATOR VN-2

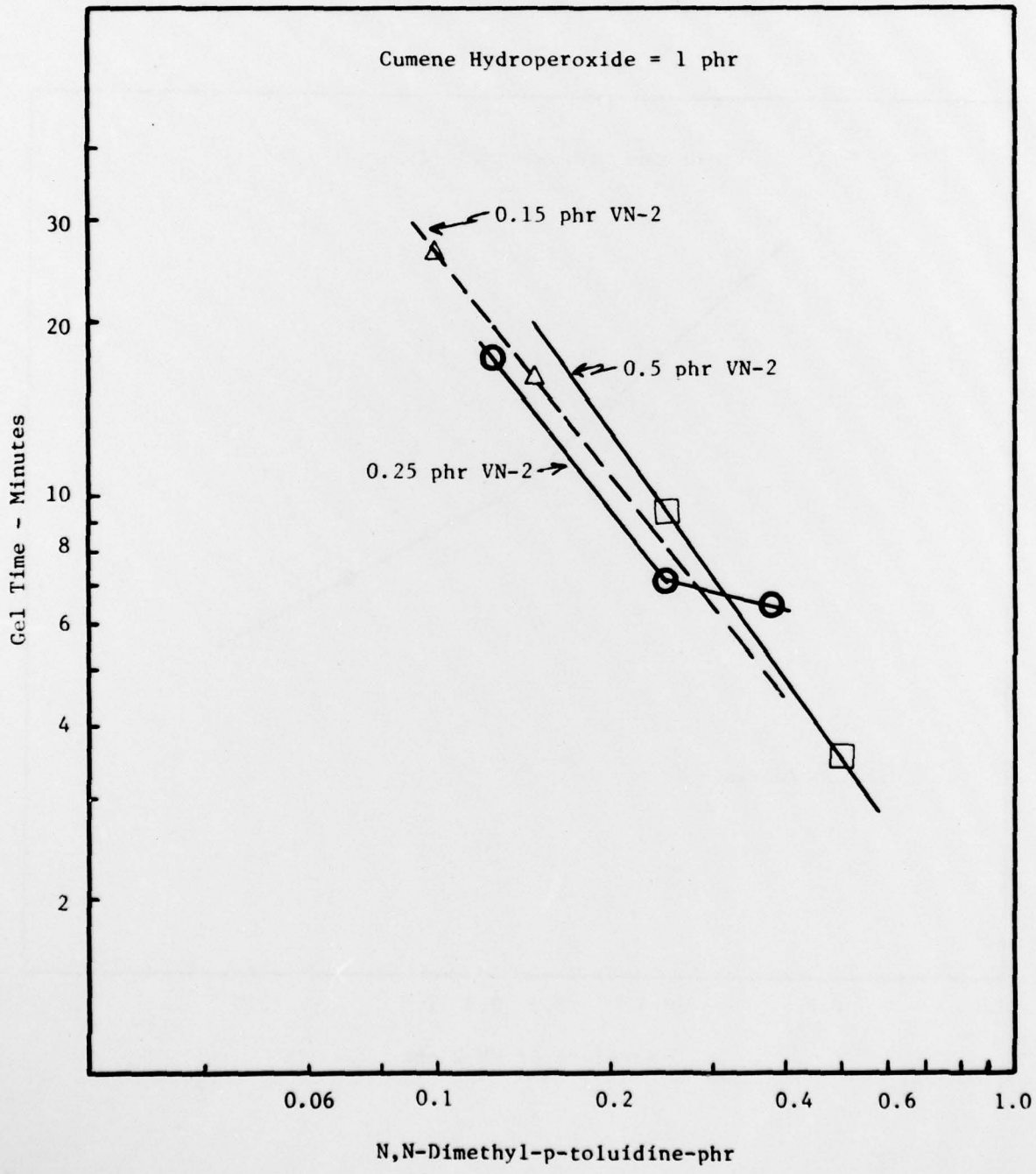

FIGURE 3

EFFECT OF PROMOTER ON REACTION RATE

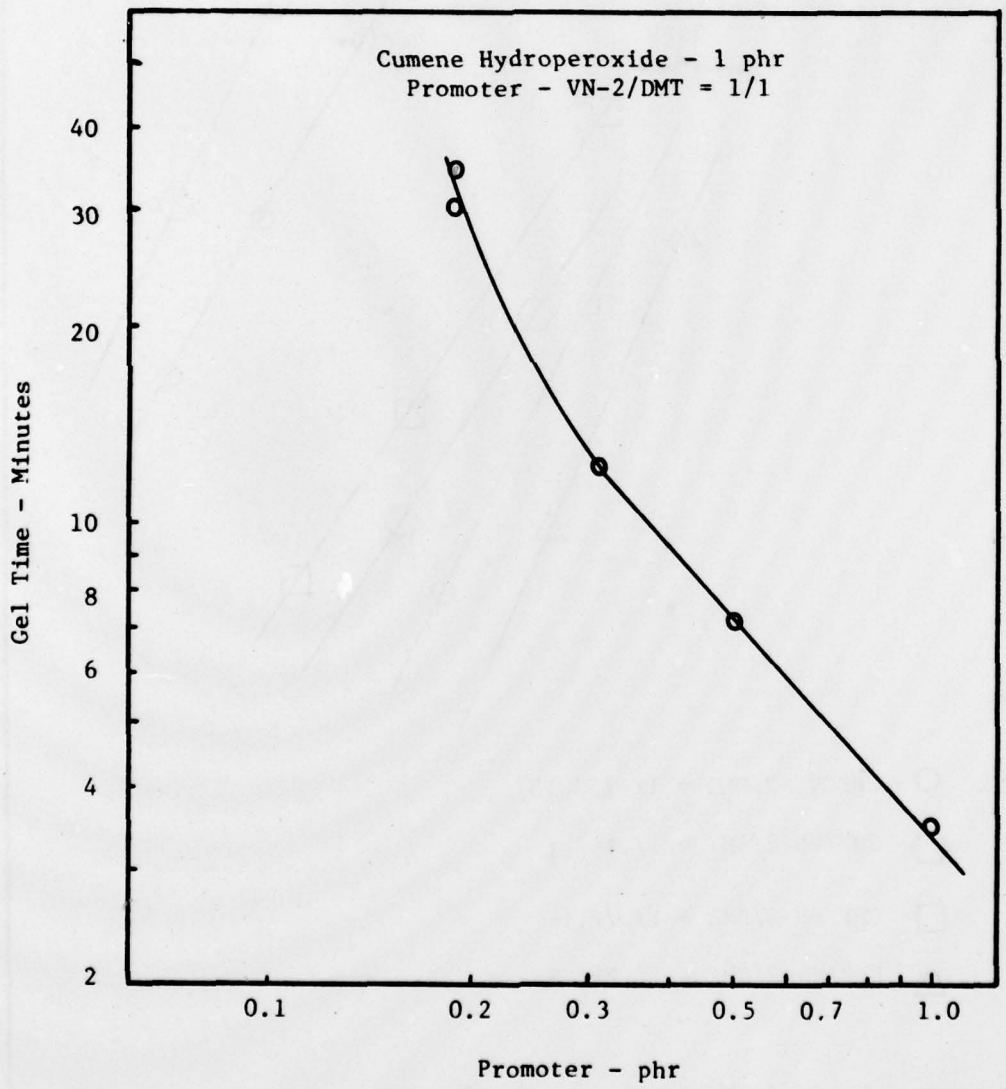

FIGURE 4

INFLUENCE OF PROMOTER ON REACTIVITY

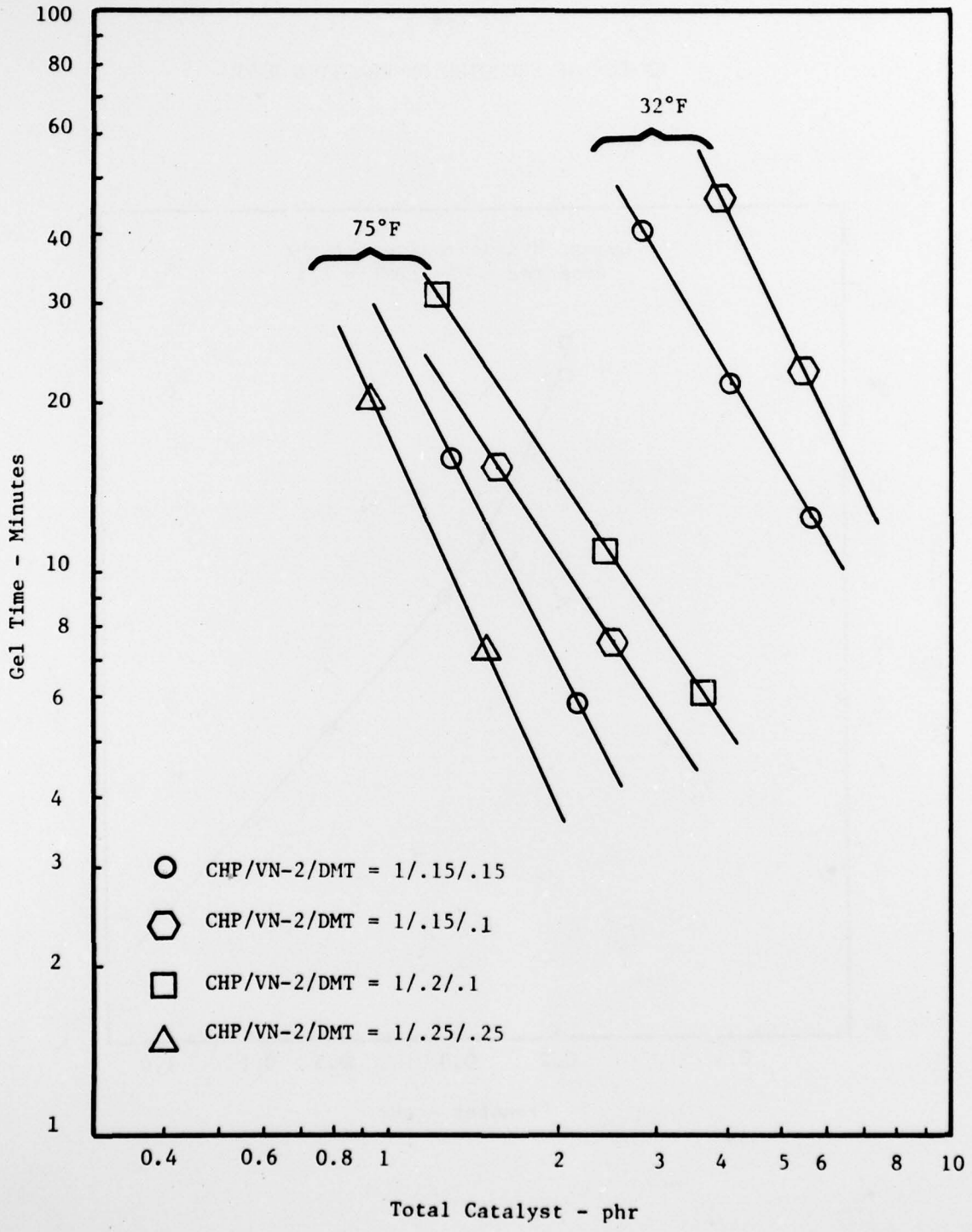

FIGURE 5

SELECTION OF CATALYST CONCENTRATION
VS. TEMPERATURE OF APPLICATION
(Constant Gel Time, 10 Minutes)

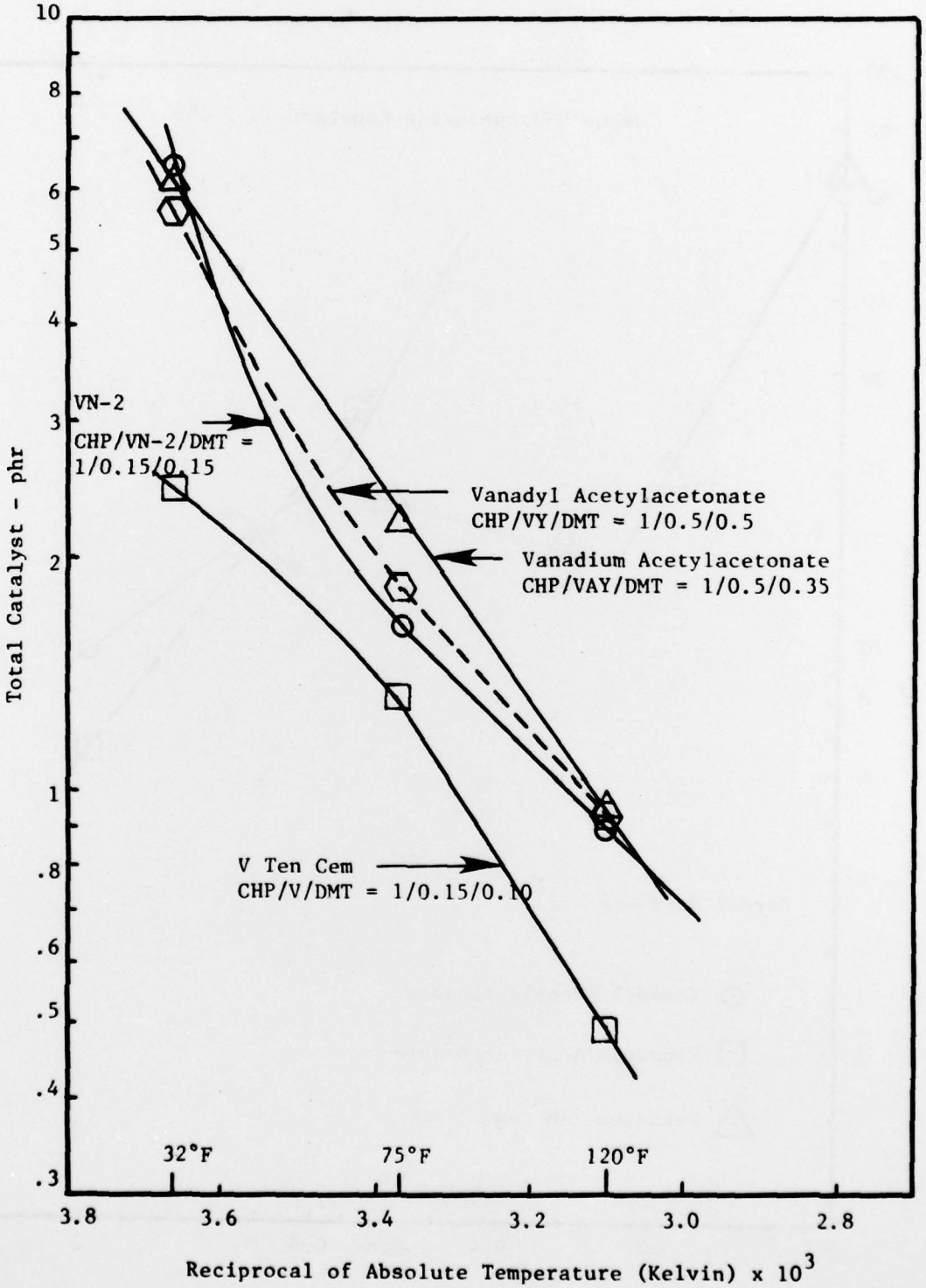

FIGURE 6

CATALYST COMPONENT STUDY
EFFECT OF VARIOUS VANADIUM COMPOUNDS

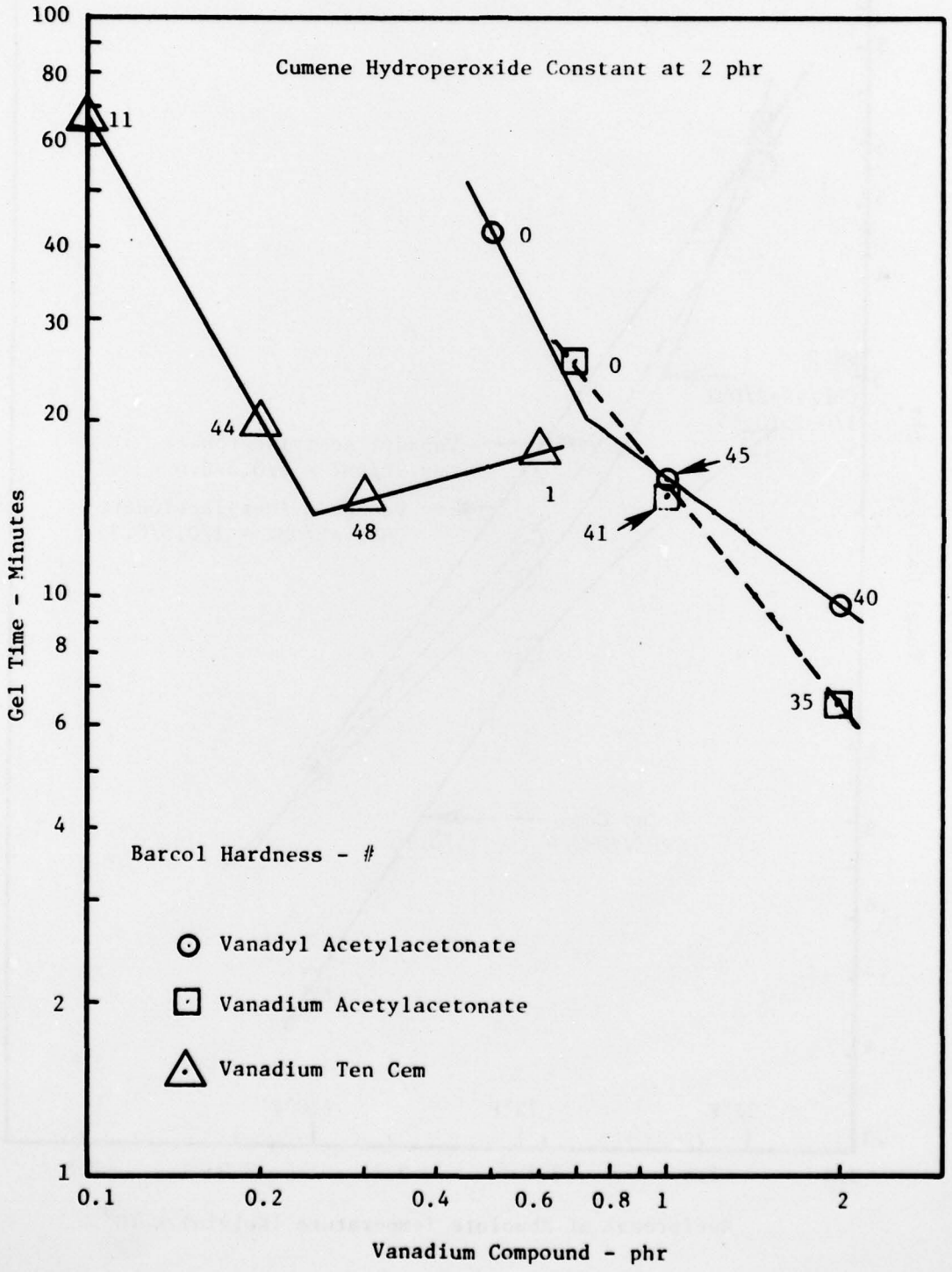

FIGURE 7

CATALYST COMPONENT STUDY
EFFECT OF VANADYL NAPHTHENATE

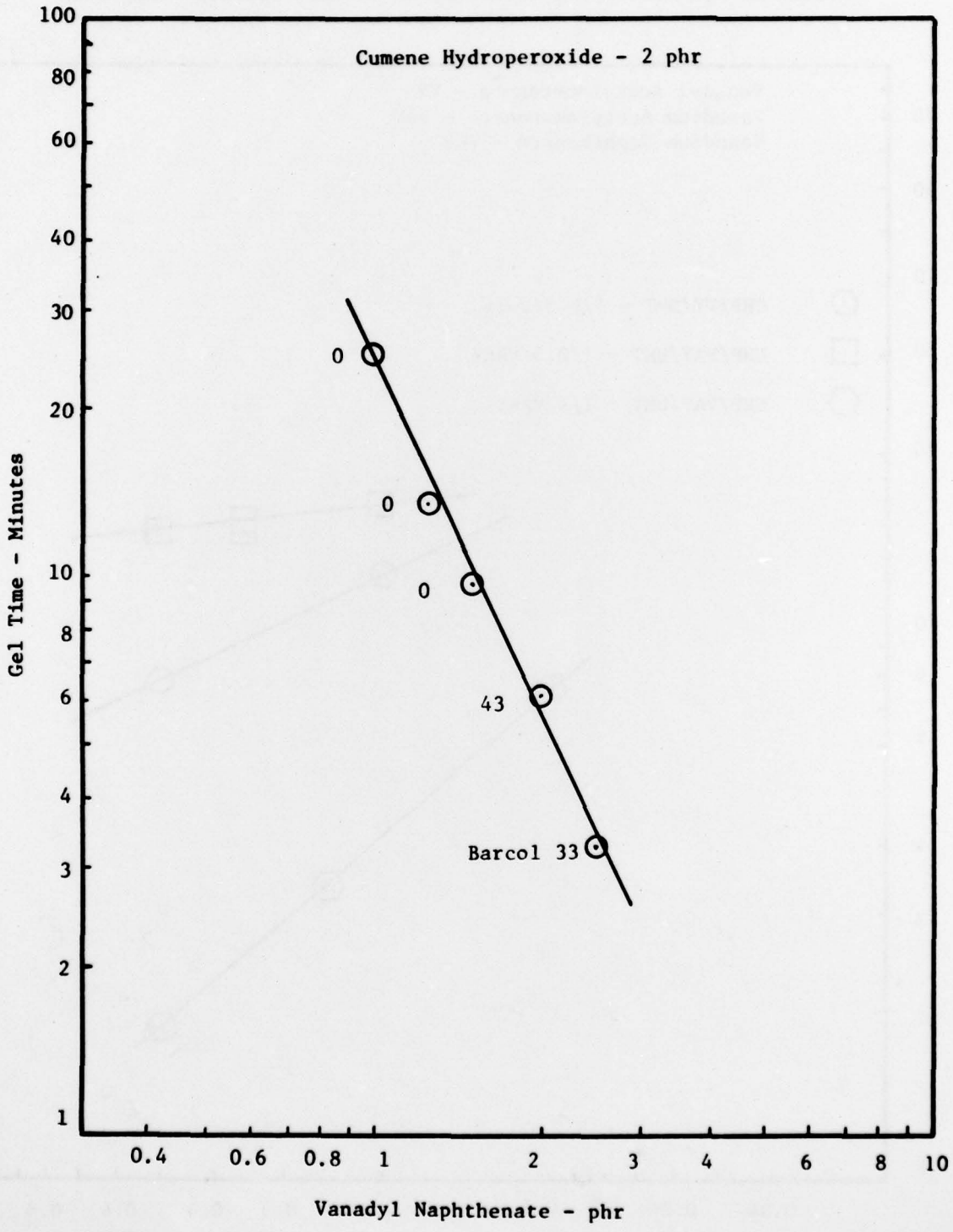

FIGURE 8

CATALYST COMPONENT STUDY
Effect of N,N-Dimethyl-p-toluidine on Various Vanadium Compounds

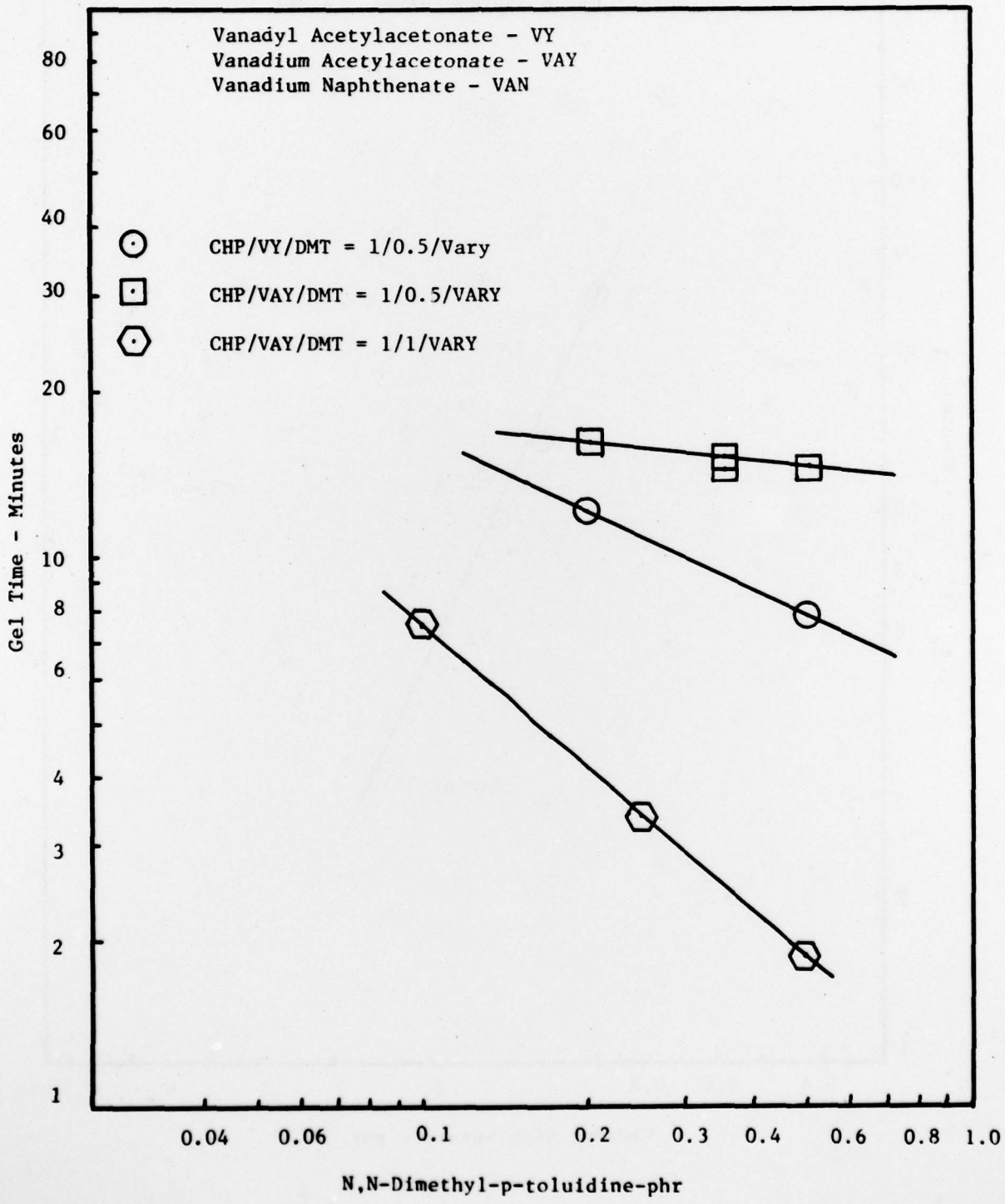

FIGURE 9

GEL TIME VS. CATALYST CONCENTRATION AT DIFFERENT TEMPERATURES

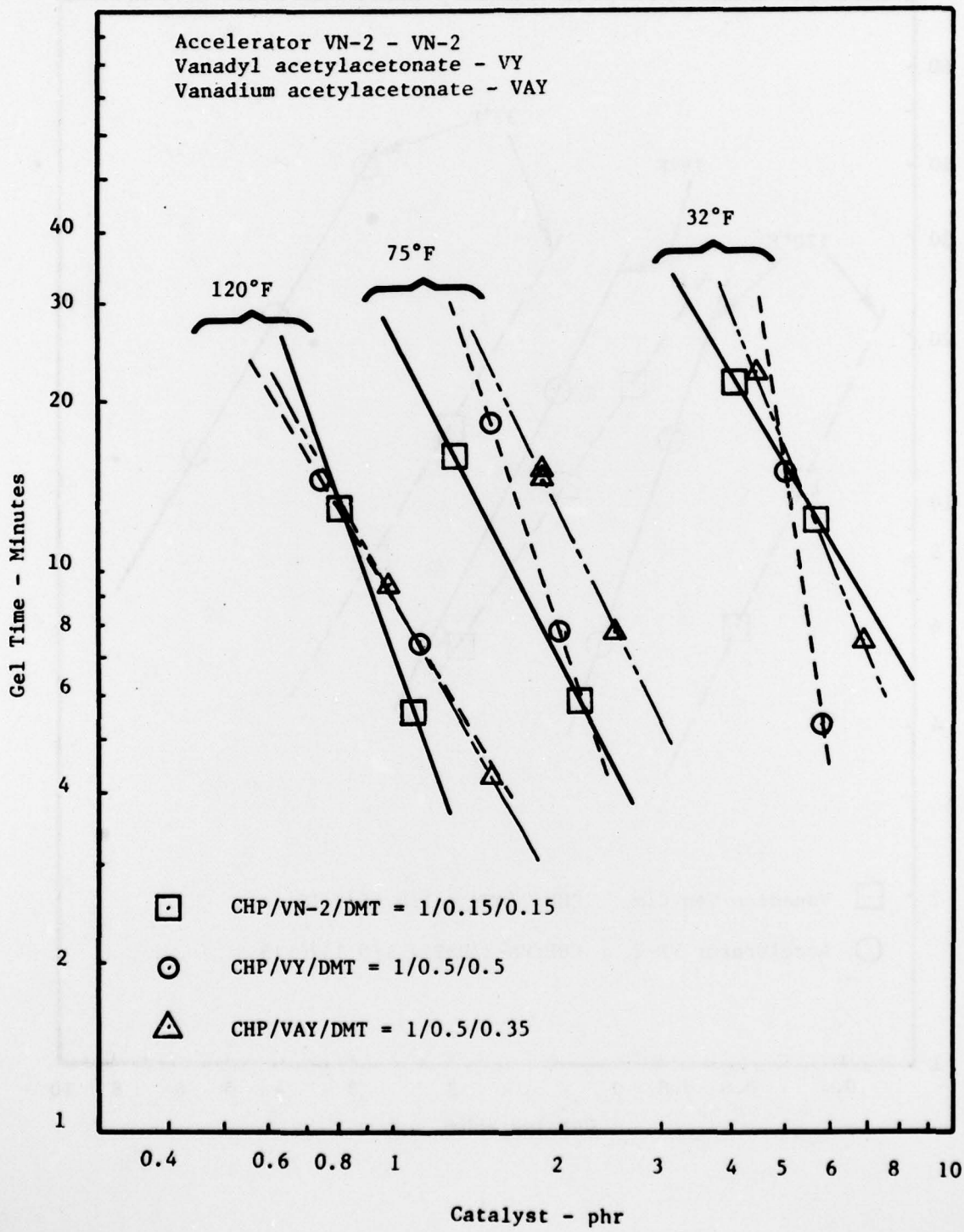

FIGURE 10

COMPARISON OF ACCELERATOR VN-2 AND
VANADIUM TEN CEM

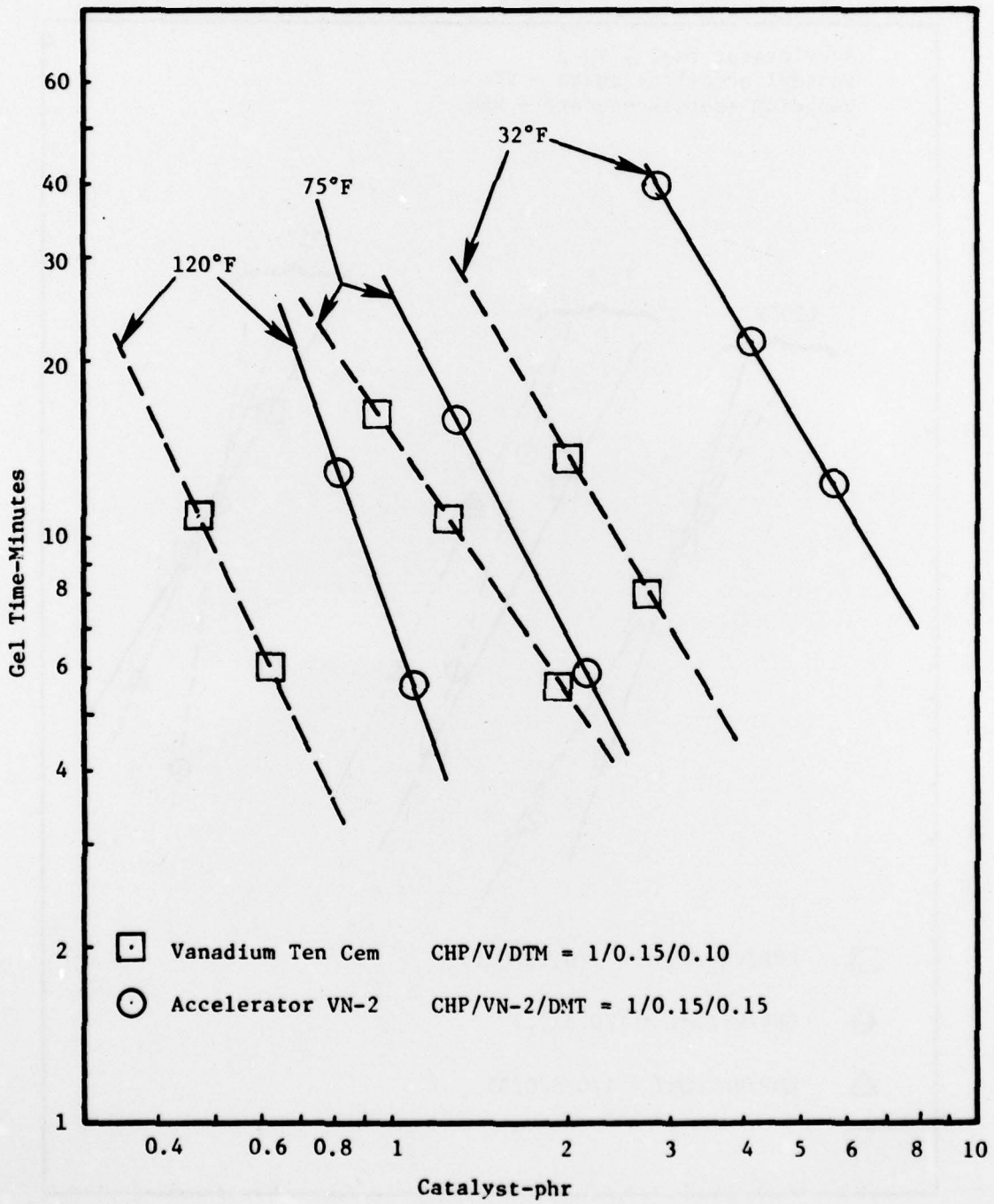

FIGURE 11

LAMINATE EXOTHERM TIME VS. GEL TIME

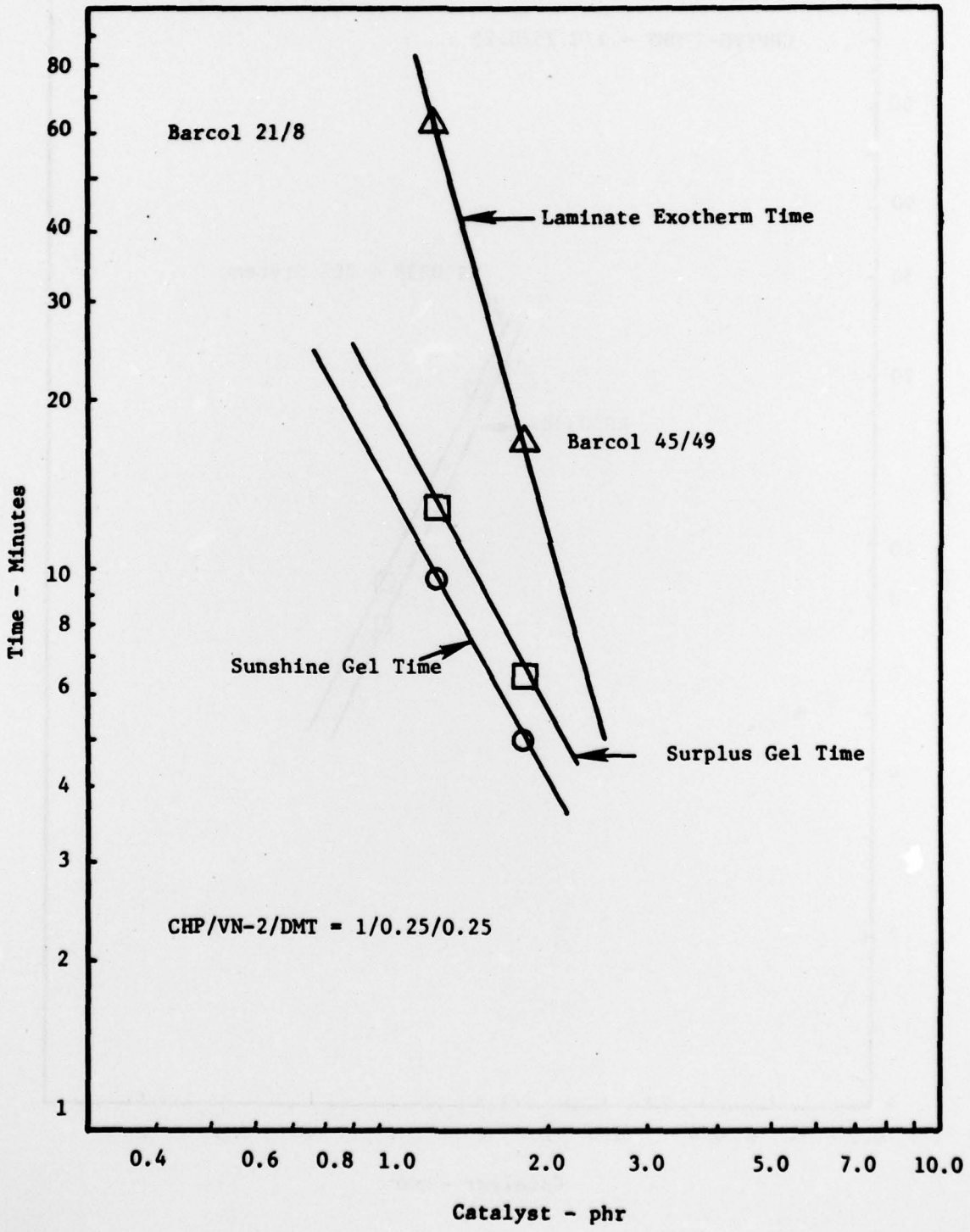

FIGURE 12

EFFECT OF CATALYST ON THE REACTIVITY OF
RS50338 and RS50338 + 20% Styrene