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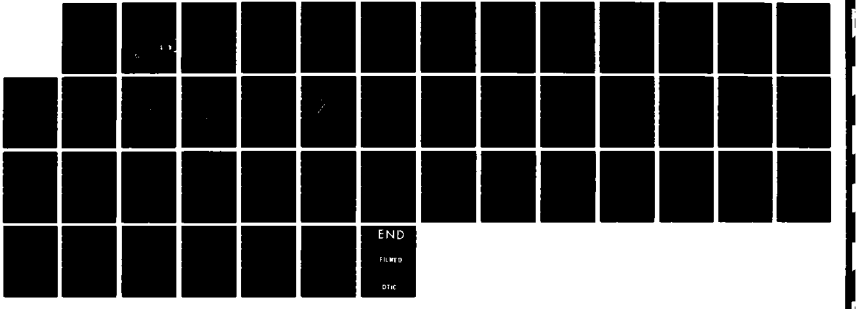

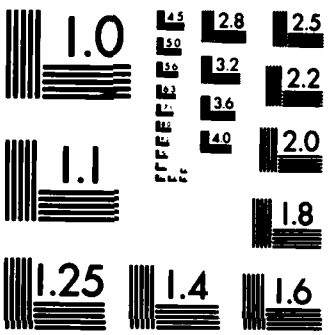

NSWC/TR 84-150

SCALE-UP OF VANADIUM TRINEODECANOATE

BY ROBERT E. FARNCOMB

RESEARCH AND TECHNOLOGY DEPARTMENT

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NAVAL SURFACE WEAPONS CENTER

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Vanadium trineodecanoate (VND) is one of the promoter components of the fiberglass-reinforced plastic required for the Marine Corps' flexible membrane surfacing for soils.

A synthesis procedure for VND, developed by the Naval Weapons Center, China Lake, was scaled up by a factor of 20 to produce 15-kg batches of the material. Since the VND-product solution is incompatible with the by-product hydrochloric acid, it is necessary to complete the reaction within 6 hours.

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after the addition of the neodecanoic acid. The minimum cost for VND solution prepared at the 15-kg batch level is $25/kg. It is calculated that a process based on a continuous flow reactor with a capacity of 10 kg/hr could produce the VND solution at a cost of $10/kg. In a much larger continuous reactor, the price for VND would drop to about $4/kg.

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FOREWORD

This report describes the development of procedures for the scaled-up synthesis of vanadium trineodecanoate (VND). This compound is one of the promoter components for the on-site preparation of flexible membrane surfacing for soil used by the Marine Corps. Fifteen kilogram batches have been successfully prepared and a continuous flow reactor is proposed for larger scale production. This work was supported by contracts, N6830579WR90217 and N6830582WRZ0143.

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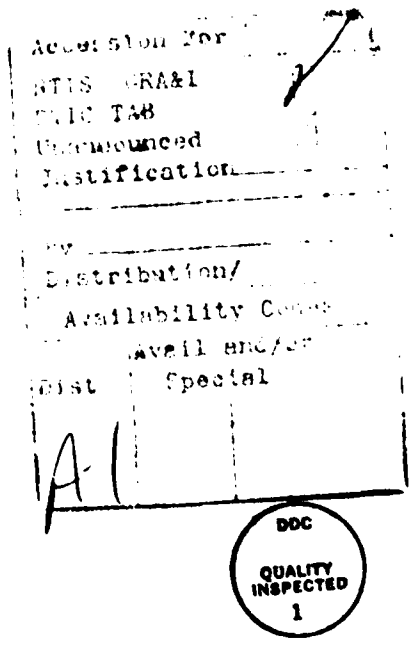

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INTRODUCTION

Vanadium trineodecanoate (VND) is one of the promoter components of the fiberglass-reinforced plastic composite material required for the Marine Corps flexible membrane surfacing for soils. This composite material was developed by the Naval Civil Engineering Laboratory, Port Hueneme, CA. It requires the following steps for application:

a. soil preparation
b. spreading of fiberglass matting
c. spraying of polyester resin, catalyst, and promoter solution (100:1.1:0.4) on the fiberglass, using three metering pumps
d. applying extra resin and fiberglass as needed

The catalyst for the system is cumene hydroperoxide and the promoter is a solution of N,N-dimethyl-p-toluidine and VND.

The flexible membrane surface is applied to soil surfaces to control erosion and to improve the possibilities for amphibious landings and vehicular and aircraft traffic (reference 1).

The Naval Weapons Center (NWC), China Lake, CA, developed a procedure for the preparation of VND (reference 2). The procedure has two steps, consisting of the reduction of pentavalent vanadium to trivalent vanadium and the formation of the metal salt. The chemical equations are shown below:

$$V_2O_5 + 6HCl + 2HCOOH \xrightarrow{80^\circ - 90^\circ} 2VCl_3 + 5H_2O + 2CO_2$$

$$VCl_3 + 3C_9H_{19}COOH \xrightarrow{100^\circ - 150^\circ} V(C_9H_{19}COO)_3 + 3HCl$$

In the first step, the vanadium pentoxide is dissolved in concentrated hydrochloric acid and reduced by formic acid to vanadium trichloride at 80° - 90°C. During this reduction the color of the reaction mixture changes from brown to deep blue. In the second step, VND is formed by reacting neodecanoic acid (NDA) with vanadium trichloride, in the presence of

triethylphosphate. The water and the byproduct hydrochloric acid are removed by azeotropic distillation with xylene. The reactants and their molar ratios, as well as the solvents used in the NWC procedure, are listed below:

Ingredient	Amount	Moles	Moles/Mole V_2O_5
Vanadium pentoxide	54 g	0.30	1.00
Hydrochloric acid (36%)	540 ml	5.31	17.88
Formic acid (90%)	42 g	0.82	2.67
Paraformaldehyde	6 g	0.20	0.67
Xylene	250 ml	-	-
Triethylphosphate	192 g	-	-
Neodecanoic acid	330 g	1.92	6.45

The reaction yields 750 g of a deep blue solution of the following composition:

Vanadium trineodecanoate	45%
Triethylphosphate	25%
Xylene	27%
Neodecanoic acid	3%
Water and chloride ion	<0.5%

A potential problem in the scaled-up preparation of VND is the by-product hydrochloric acid, which causes decomposition of VND. See Appendix A for further information on this problem.

SCALE-UP TESTS

SCALED-UP PREPARATION OF VND SOLUTIONS

Experiments to prepare VND solutions started with a repeat of the NWC procedure. Then a series of intermediate-size scaled-up reactions were run, primarily at the 3 and 15-kg levels. Three batches were run at the 150-kg level. Optimized procedures yielding an acceptable product at the 3-and 15-kg batch levels are described below and in more detail in Appendices B and C, respectively. The 150-kg batches did not yield an acceptable product. Table 1 gives a summary of the results of all VND batches run under this program.

Preparation of VND in 3-kg Batches

The equipment used to prepare 3-kg batches of VND solution consisted of a five-liter, three-necked glass flask in an electric heating mantle, equipped with stirrer, distillation head, and distillation set-up as shown in Figure 1. The reaction and distillation in the equipment were carried out with a nitrogen purge of 500 ml/min.

The five-liter glass reaction flask was charged with 1800 ml of 37% hydrochloric acid, 168 g of 90% formic acid, and 1000 ml xylene. After

starting the agitator, 216 g vanadium pentoxide and 24 g paraformaldehyde were added. Then the heating mantle was turned on to maintain about 90°C in the reaction flask for a period of three hours, during which the color of the reaction mixture changed from brown to deep blue. After this reaction period, 768 g triethylphosphate were added to the reaction mixture and the temperature controller setting for the heating mantle increased to 125°C. This started an azeotropic distillation of aqueous hydrochloric acid and xylene. The two distillate phases were separated, the xylene returned to the distillation flask, and the aqueous phase withdrawn automatically by a liquid level controller. After the distillation had produced 1300 ml of aqueous distillate phase, the temperature controller setting for the heating mantle was increased to 155°C, and 1452 g neodecanoic acid added to the reaction flask with a tubing pump. The azeotropic distillation was then continued until the temperature in the reaction flask reached 155°C, which yielded an additional 560 ml aqueous distillate phase. After the distillation residue (VND soution) had cooled to 20°C, 25 g Celite were added and the entire mixture poured through a coarse, 600 ml fritted glass Buchner funnel containing a bed of 20 g Celite. The resulting 3.09 kg of clear VND solution had a vanadium content of 3.79% (3.88% theoretical). The gel-time of this solution was 6.3 minutes and 12 minutes with a maximum reaction temperature of 210°C after the addition of 0.4 g promoter solution. See Appendix D for further information on measurement of gel-time and maximum temperature.

Preparation of VND in 15-kg Batches

The equipment used was a 22-liter flask in an electric heating mantle set up as shown in Figure 2. The flask was charged with 9000 ml of 35% hydrochloric acid, 840 g of 90% formic acid, 5000 ml xylene, and the agitator and nitrogen purge started; 1080 g vanadium pentoxide and 120 g paraformaldehyde were then added. The mixture was agitated for 3 hours at 81° - 92°C. During this time, the color changed from brown to deep blue, after which 3840 g triethylphosphate were added. The water and hydrochloric acid were removed by azeotropic distillation with xylene, the xylene returned to the flask, and the aqueous phase removed via a solenoid valve. The solenoid valve was controlled by an electronic conductance liquid level controller*. (See Table 2 for distillation cuts, temperatures of flask and vapors, etc.) A tubing pump was used to add 7200 g neodecanoic acid to the flask and the azeotropic distillation then continued until the flask temperature reached 155°C.

After the VND solution cooled to 20°C, 100 g Celite were added to the flask; the VND solution was then transferred, via a tubing pump, to a 3-liter coarse-fritted glass Buchner funnel which had a 100-gram bed of Celite. Using a second tubing pump, the VND solution was pumped to a 22-liter storage flask. The filtration was completed in less than an hour. The reaction flask, tubing, filter, and pumps were washed with one liter of xylene.

*DynaSense C-7186-00, Electronic Liquid Level Controller, Cole-Parmer Instrument Co., Chicago, Illinois 60648.

The 11.95 kg VND solution was sampled and analyzed as containing 3.80% vanadium, 0.35% chloride, and 0.04% water. The gel-time of this solution was 9.1 minutes, and 11 minutes for maximum temperature of 236°C after addition of 0.4 g promoter solution. The analysis of the 1.8 kg wash solution showed 2.52% vanadium. The gel-time of this solution was 19.5 minutes, and 24 minutes for the maximum temperature of 203°C. These solutions account for 82% of the starting vanadium; the remainder of the vanadium was in the precipitate - a mixture of coarse blue-black solids and blue slime.

A fire occurred in VND batch 67-27 while preparing anhydrous vanadium trichloride. Present in the 22-liter flask at the time of the incident were 5.5 liters xylene, 4224 g triethylphosphate, 2054 g vanadium trichloride, and up to 5 kg water, under nitrogen purge. At the time of the incident, all the excess hydrochloric acid had been removed and about half the water. The most probable cause of the incident is that the agitator stopped, allowing solid and liquid phases to separate. The unstirred material 'bumped'*, ejecting xylene and triethylhosphate out of the flask onto the hot heating mantle. The resulting fire ejected the rest of the flammable material.

Future VND batches will be made using equipment with beaded glass pipe fittings which are rated for 15 psig (Figure 5), thus preventing ejection of reactants if bumping occurs.

Preparation of VND in 150-kg Batches

Details of the 50-gallon glass-lined reactor with 2-inch glass heat exchanger used in this phase of the experiment can be seen in Figure 3. A nitrogen purge of one liter per minute was used during additions and 0.1 liter per minute during reaction and distillation.

Using a 3/8-inch ID Viton tubing pump, 8.4 kg of 90% formic acid, 102.6 kg of 35% hydrochloric acid, and a slurry of 10.8 kg vanadium pentoxide and 1.2 kg paraformaldehyde in 43.0 kg xylene were added to the reactor. The reactor was heated for 3 hours at 80° - 90°C while being agitated, then 38.4 kg triethylphosphate added. In 36 hours of xylene distillation, 108 kg of dilute hydrochloric acid were collected, and the xylene returned to the reactor. During the distillation, the reactor temperature was 100° - 110°C and vapor temperature was 97° - 106°C. The distillation was limited by condenser flooding. This flooding was controlled by a steam controller monitoring the vapor temperature.

The batch was left for 3 weeks** at room temperature. With the reactor temperature at 102°C; 66 kg neodecanoic acid and one liter xylene wash were

*'bumping' is localized superheating leading to unstable distillation.
**Interruption caused by circumstances beyond operator control- -jury duty.

then pumped into the reactor. The reactor was heated from 102°C to 149°C with 40 psig steam; the vapor temperature increased from 98° to 100°C. A total of 2.6 kg of 7.6% hydrochloric acid was collected. The remainder of the hydrochloric acid from the reaction of vanadium trichloride with neodecanoic acid was absorbed in the sodium hydroxide scrubber.

The VND solution was cooled to 25°C and filtered in a perforated bowl centrifuge in one hour. The centrifuge bowl was 12 inches in diameter, 6 inches high, and spun at 4000 rpm. The solids were collected in a 30-micron polypropylene filter bag with a 1-cm bed of Celite. The analysis of the VND solution showed values of 2.62% vanadium, 0.24% chloride, and 1.15% water. The gel-time using 0.4 g promoter from this solution was 27 minutes.

Three 150-kg batches were run in this 50-gallon reactor. The incompatibility of the by-product hydrochloric acid resulted in low vanadium concentractions in all three. The analyses of these VND solutions yielded values of 2.6%, 0.1%, and 0.1% vanadium, instead of the expected 4% vanadium.

Large quantities of blue solids remained in the reactor, which were soluble in 50°C hydrochloric acid.

Recommended VND Synthesis Procedure

Vanadium trineodecanoate (VND) is degraded when heated with hydrochloric acid at 90°C for 6 hours.

In a continuous reactor, the VND will be heated with the hydrochloric acid for less than 4 hours. The continuous flow reactor shown in Figure 4 has a capacity of 10 kg of VND solution per hour. The main components of this flow reactor are:

Three metering pumps to add:
1. Xylene
2. 85% vanadium trichloride in hydrochloric acid
3. Solution of neodecanoic acid, triethylphosphate, xylene

A 4-inch glass boiler at 100°C with 0.5 m^2 heat transfer area, with agitator

A 2-inch glass heat exchanger at 100°C.

A 2-inch glass heat exchanger at 150°C.

Three 2-inch glass heat exchangers to condense vapors

A 2-inch glass heat exchanger to cool product to room temperature.

For each kilogram of VND solution prepared, 22,000 calories of heat need to be transferred and about 60 to 100 liters of vapor condensed.

MATERIAL AND STORAGE REQUIREMENTS FOR VND SOLUTIONS

Construction materials for equipment used in the preparation of trineodecanoate solutions are limited by the nature of boiling xylene and hydrochloric acid. Glass, Teflon^{R}, and platinum are compatible with this mixture. Neoprene stoppers exposed to the hot vapors swelled and had surface discoloration, but were usable after more than 100 hours exposure. Viton^{R} tubing exposed to the hot vapors had surface scaling but was useable after 100 hours exposure.

Polyethylene and Viton^{R} tubing are compatible with the cold solutions encountered in making VND solutions.

VND solutions can be stored in glass and polypropylene bottles under a nitrogen atmosphere. The storage results are listed in Table 3. After one year storage, the gel-time increased 4% and 17% in glass and polypropylene, respectively.

VND solutions should not be in contact with 316 stainless steel or aluminum for more than a month.

Gel-time promoter solutions (50% VND, 50% DMT) stored in a nitrogen atmosphere in full glass bottles changed from 6.4 minutes to 8.3 minutes after one year storage. This is a 30% increase in gel-timne. Promoter solutions stored under air in almost-empty bottles were decomposed to a brown solution which had no gelling properties.

VND solutions can be stored in glass and polyethylene bottles under a nitrogen atmosphere; a gallon bottle holds 3.8 kg (8.4 pounds) of VND solution.

ENVIRONMENTAL CONSIDERATIONS OF VND PRODUCTION

During the distillation, a scrubber is needed to remove uncondensed hydrochloric acid. An efficient scrubber was 20% sodium hydroxide solution pumped through a plastic aspirator. In the larger batches, about 20% of the hydrochloric acid was absorbed in the scrubber. A more efficient heat exchanger will reduce the caustic consumption.

The vanadium concentration in the unusable product can be lowered to 0.1% by adding acid. Dilute hydrochloric or sulfuric acid decomposed the VND within 16 hours at room temperature. The vanadium concentration can be lowered by extraction with a liquid ion exchange system. Alamine 336, a (trademark) product of General Mills, is a liquid ion exchanger developed for vanadium extraction. It is a water insoluble tertiary amine dissolved in kerosene. Alamine^{R} extracts vanadium at pH 1.2-2 and releases it at pH 11-12 (reference 3); the vanadium can then be recovered using standard metallurgical techniques.

The vanadium concentration can also be lowered to 1 ppm (part per million) by treatment with sodium borohydride at pH 9-10 (reference 4).

Appendix E is the Material Safety Data Sheet for the polyester promoter, (OSHA standard form OMB No. 44-R1387).

VND MATERIAL SOURCES AND COSTS

Neodecanoic acid and triethylphosphate are made by one United States manufacturer (reference 5). Both compounds are used in large quantities and will be available for VND manufacture. The other raw materials have at least 5 United States manufacturers (reference 5).

The raw materials cost for VND solutions is $3.20 per kilogram. See Table 4 for an itemized list with suppliers. VND solutions will cost about $25 per pound when made in 15-kg batches. This cost is valid using the procedure in Appendix D and already-installed equipment.

CONCLUSIONS

1. VND solutions can be made in 15 to 30-kg batches.
2. VND solutions can be stored over a year in glass or polypropylene bottles under a nitrogen atmosphere.

RECOMMENDATIONS

The continuous reactor outlined in Figure 4 should be investigated because it has the potential of increasing production of VND and reducing the cost of the product.

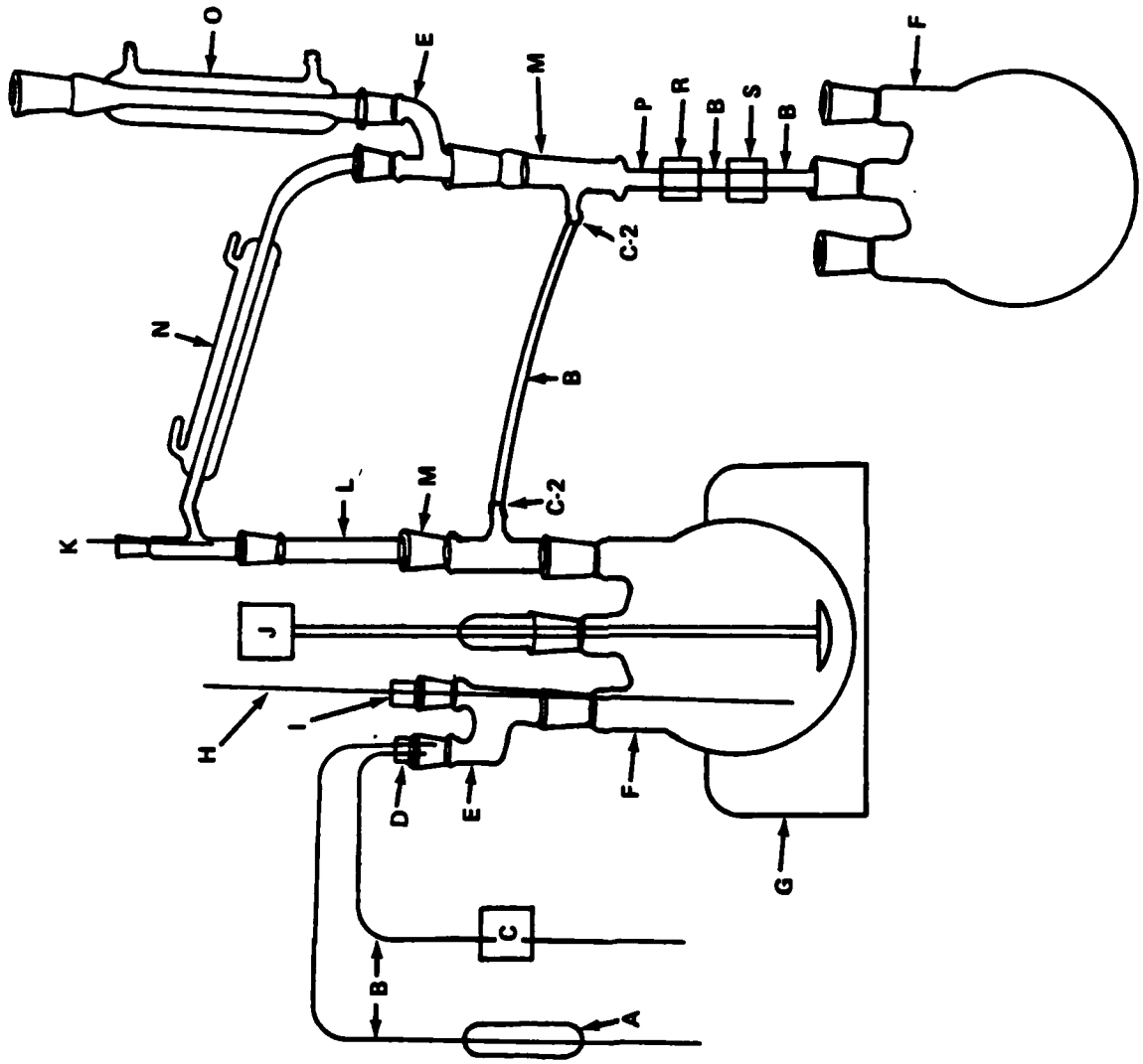

FIGURE 1. APPARATUS FOR MAKING VND IN 3 KG BATCHES
SEE TABLE 6 FOR LISTING

1. THERMOMETER VAPOR
2. AIR POWERED STIRRER
3. FLASK THERMOMETER EQUIPPED WITH THERMO-WATCH TEMPERATURE CONTROLLER
4. 22-LITER FLASK
5. HEATING MANTLE
6. XYLENE-WATER INTERFACE (WATER BOTTOM LAYER)
7. VITON TUBING
8. LEVEL CONTROLLER PROBES
9. TEFLON SOLENOID VALUV
10. WATER COLLECTION FLASK

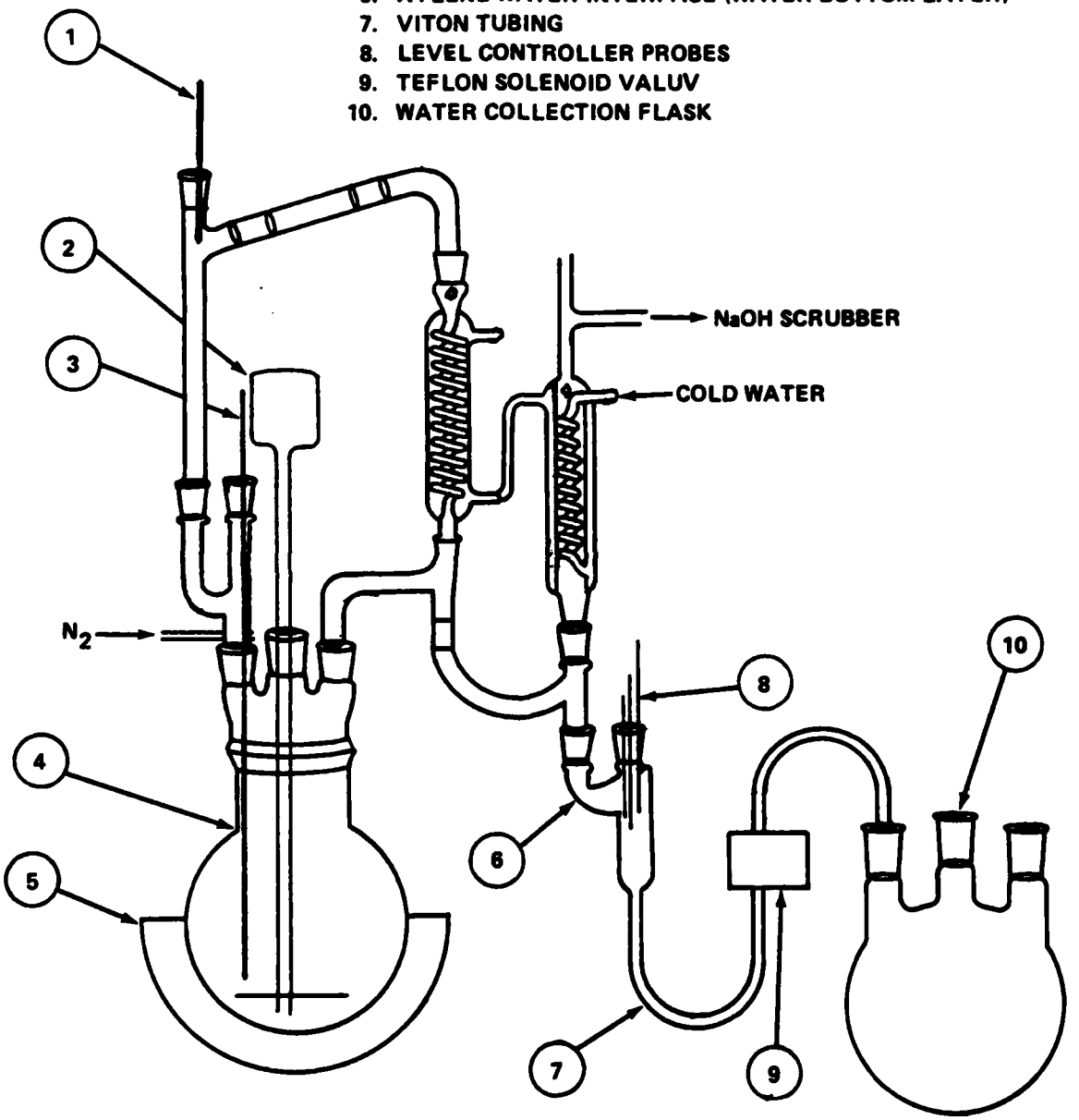

FIGURE 2. APPARATUS FOR 15-KG VND BATCHES

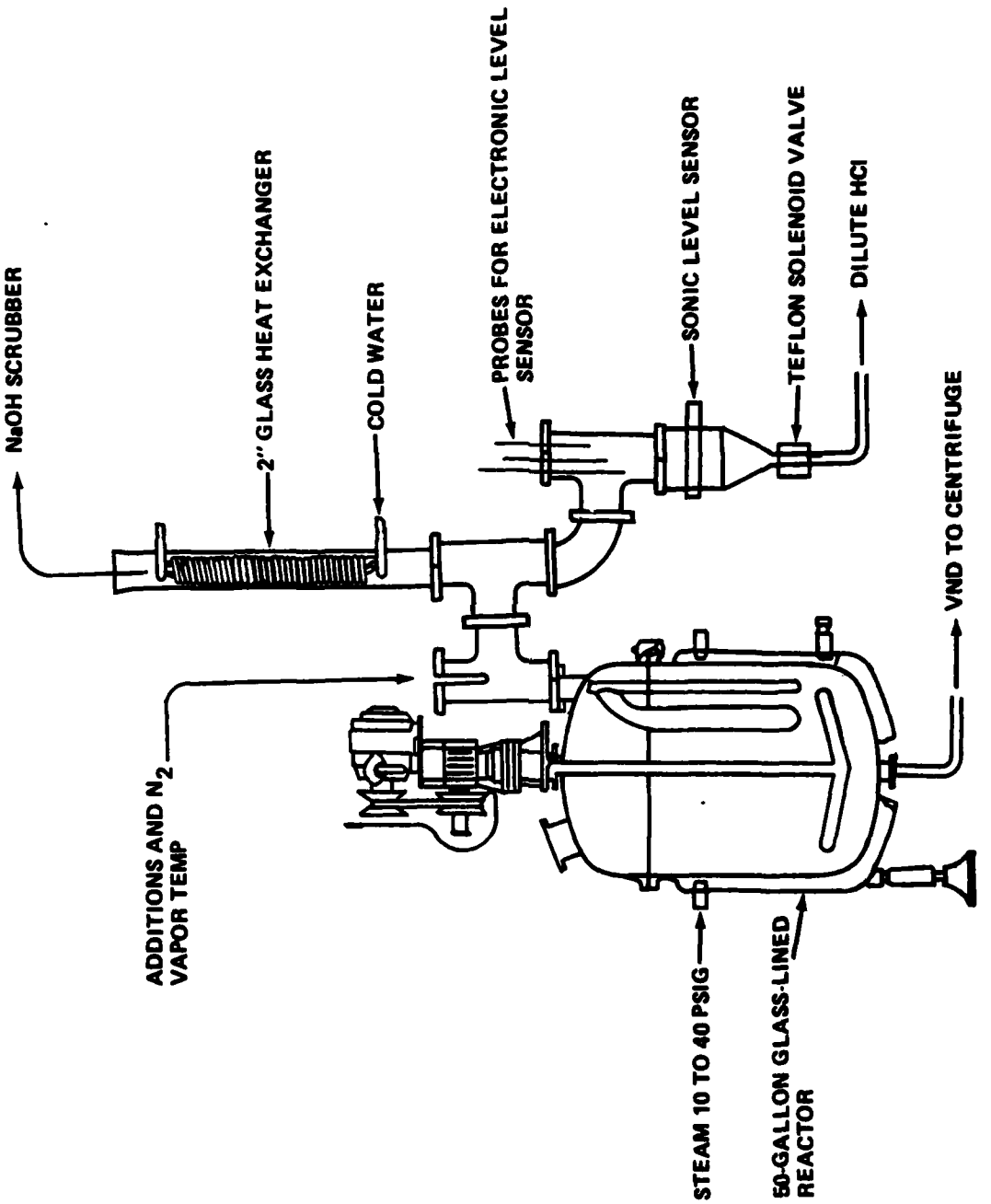

FIGURE 3. EQUIPMENT FOR 150-KG VND BATCHES

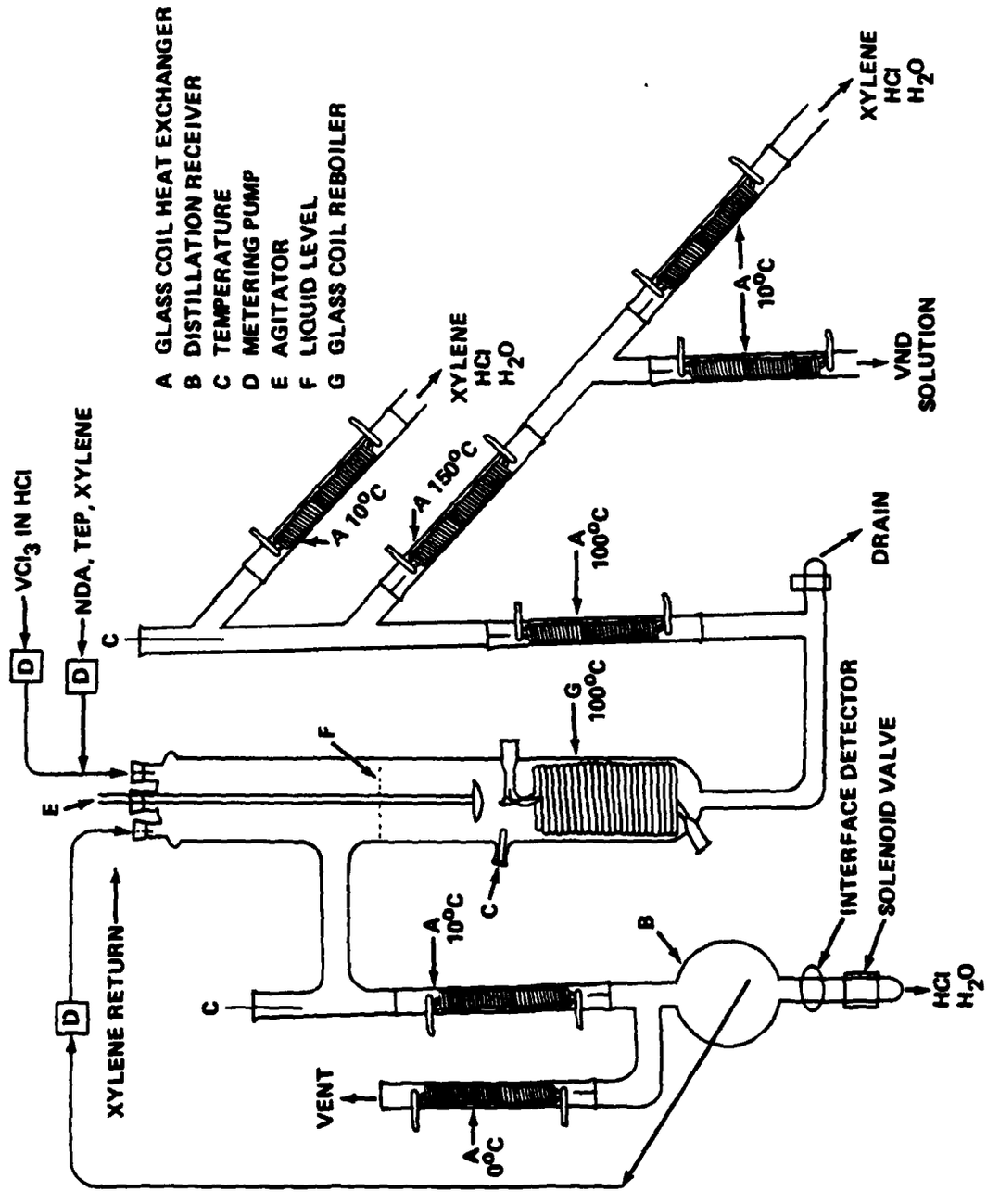

FIGURE 4. CONTINUOUS VND FLOW REACTOR

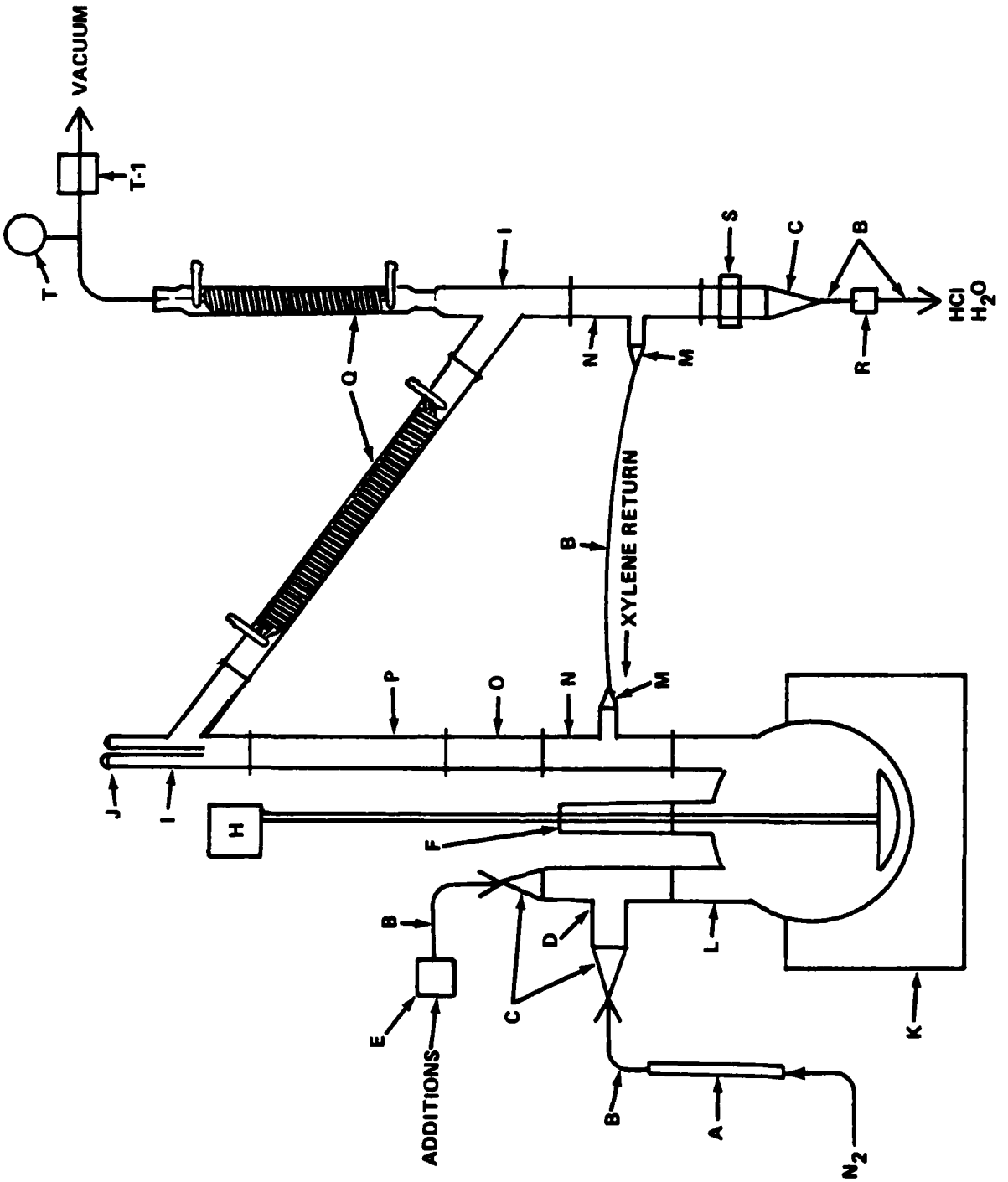

FIGURE 5. EQUIPMENT FOR MAKING VND SOLUTIONS IN 15-KG BATCH (22-LITER FLASK WITH 2-INCH BEADED GLASS FITTINGS)

USE TABLE 6 FOR LISTING

TABLE 1. SUMMARY OF VND BATCHES

Batch	Batch Size[1]	% V[2]	Theor. % V[3]	Hours VND Soln Heated After Addition of NDA	H_2O HCl Removal[4]	Gel Time With 0.4 g promoter min.	Maximum Temp. of Gel °C	Time of Maximum Temp. Min.	Comments
66-1	1	0.2	3	18	A	-	-	-	Large amounts of blue solids
90-2	1	1.9	3	15	A	10.5	190	13	
90-3	1	-	3	-					Black brown foam after temperature reached 185°C
95-4	1	0.5	3	12	A	40+	-		Pentrolite W-4088 Water-oil emulsion added - foamed
1-5	1	2.88	3	6	B	13.8	190	17	
2-6	1	2.6	3	6	B	15.4	145	20	
9-7	3	0.1	4.3	15	B	-	-	-	Brown solution
12-8	3	0.1	4.0	19	B	-	-	-	Brown solution
14-9	3	0.1	4.1	21	B	-	-	-	Brown solution-blue solid
16-10	3	0.1	4.0	-	B	-	-	-	High speed agitation, large N_2 purge used, black foam
17-11	3	0.27	4.0	24	B	-	-	-	Hot oil bath at 150°C used for heating
20-12	2	0.1	3.4	28	B	-	-	-	Brown solution, blue solids
28-13	2	1.1	5.0	20	B	-	-	-	Heated with hot oil bath, brown solution, course black solid

TABLE 1. SUMMARY OF VND BATCHES (CONT)

Batch	Batch Size[1]	% V[2]	Theor. % V[3]	Hours VND Soln Heated After Addition of NDA	H_2O HCl Removal[4]	Gel Time With 0.4 g promoter min.	Maximum Temp. of Gel °C	Time of Maximum Temp. Min.	Comments
29-14	2	-	4.0	12	B	-	-	-	Light yellow oil
30-15	1.5	2.57	3.3	5	B	-	210	21	160°C oil bath,
34-16	3	2.45	2.9	5	B	16.2	201	24	fast agitation, fast N_2 purge
34-17	3	1.1	2.9	7	B	-	-	-	Large amounts of solids
35-18	3	2.44	2.9	4	B			-	Add NDA after most of HCl removed
36-19	3	3.05	3.5	5	B	6.3	202	9	No solids in flask
38-20	3	2.28	3.1	3	B	30.3	175	52	
41-21	3	3.85	4.2	6	C	7.1	201	12	
48-22	4	3.50	3.7	3	C	10.0	205	13.4	
49-23	4	3.79	4.0	4	C	6.3	210	12.0	
51-24	20	3.84	4.3	6	C	8.1	210	9.7	22-liter flask
54-25	20	3.79	4.3	6	C	10.5	206	13.5	
64-26	20	3.80	4	4	D	9.1	236	11	0.35% Cl 0.04% H_2O
67-27	22	-	-	-	D	-	-		Fire occurred during batch
2-28	200	2.62	4	8	D and E	27	188	52	50 gallon reactor left 3 weeks, after about 50% HCl removed.

TABLE 1. SUMMARY OF VND BATCHES (CONT)

Batch	Batch Size[1]	% V[2]	Theor. % V[3]	Hours VND Soln Heated After Addition of NDA	H_2O HCl Removal[4]	Gel Time With 0.4 g promoter min.	Maximum Temp. of Gel °C	Time of Maximum Temp. Min.	Comments
4-29	200	0.1	4	8	D and E	-	-	-	The distillation cut before the NDA addition was 13% HCl; 1 hour after the addition of NDA, the precipitated solids stopped the agitator until the temp reached 120°C.
4-30	200	0.1	4	7	D and E	401	-	-	Agitator stopped 3 hours after addition of NDA because of solids.
11-31	1	3.75	4.1	-	D	10.1	210	12	VCl_3 in HCl added to NDA TFP, xylene at 140-150°C
13-32	4	3.80	4.0	-	D	9.2	212	12	Same comments as batch 11-31.
16-33	20	0.1	4.0	-	D	-	-	-	Same comments as batch 11-31.
29-34	25	3.57	4.0	3.5	D	13.8	196	18	Used procedure in Appendix C
30-35	25	2.37	4.0	7.5	D				Black foam, NDA added at 105.
34-36	20	0.1	4.0	4.0	D	-	-	-	Sodium salt of NDA added at 95°C
37-37	20	0.1	-	6	D	-	-	-	Brown foam, NDA add 115°C

TABLE 1. SUMMARY OF VND BATCHES (CONT)

Batch	Batch Size[1]	% V[2]	Theor. % V[3]	Hours VND Soln Heated After Addition of NDA	H_2O HCl Removal[4]	Gel Time With 0.4 g promoter min.	Maximum Temp. of Gel °C	Time of Maximum Temp. Min.	Comments
41-38	20	-	-	-	D	-	-	-	Heating mantle burn-out.
43-39	20	0.1	-	4	D	-	-	13	Blue solids NDA added 115-120°C
46-40	20	3.62	4	3	D	8	208	13	Operated under 10 inches of vacuum
48-41	20	1.06	4	6	D	12.6	218	18	
53-42	20	2.02	4	4	D	-			

[1]Batch size multiples of NWC procedure: 42 g 90% formic acid, 450 ml of hydrochloric acid, 54 g of vanadium pentoxide, 192 g triethylphosphate, 6 g of paraformaldehyde, 250 ml of xylene.

[2]Vanadium analysis by Galbraith Laboratories Inc., Knoxville, Tennessee 37921.

[3]Theoretical % vanadium calculated from total weight of solution and the amount of vanadium pentoxide added.

[4]Method for determining the dilute hydrochloric acid xylene interface:

A. Visual with manual valve
B. Reservoir filled with xylene and replaced with dilute hydrochloric acid.
C. Capacitance with Teflon solenoid valve, Thermo-o-Watch made by instruments for Research and Industry, Cheltenham, PA 19012.
D. Electronic with Teflon solenoid valve, Dyna-Sense Electronic Liquid Level Controller, Cat. No. C-7186-00 Cole-Parmer, Chicago, Illinois 60648.
E. Ultrasonic with Teflon solenoid valve, Envirotech Sensall Ultrasonic Non-Penetration Liquid Level Sensor, Model 621S with 400C Controler, ordered from Briggs and Cooper Controls, Wilmington, Delaware 19803.

TABLE 2. DISTILLATION CUTS IN 15-KG VND BATCH

Cut No.	Time for Cut hr	Temperature in		H_2O, HCl Distillate Grams	HCl in Distillate %
		Flask ^oC	Vapors ^oC		
1	12	91	87-88	6335	17.9
2	1/4	91	87	120	0.01
Added Neodecanoic Acid					
3	1 1/4	91-115	87-97	520	20.3
4	1	115	98	340	21.1
5	1 1/2	115-122	98	900	24.6
6	1	122-138	98	500	16.8
7	1 1/4	138-151	98	130	13.9
8	1 1/2	151-155	98	250	11.0

TABLE 3. STORAGE OF VND SOLUTIONS[a]

	Polypropylene Bottle	Glass Bottle	316SS in Glass	Aluminum in Glass Bottle
Start of test				
VND gel-time (min)[b]	9.1	9.1	9.1	9.1
Maximum temp of gel (°C)	236	236	236	236
Time of maximum temp (min)	11	11	11	11
Wt. of metal sample (grams)	-	-	8.216	0.9009
Area of metal smaple (cm^2)	-	-	8.718	6.397
After 30 days at 20-35°C				
VND gel-time (min)[b]	9.5	9.5	10.1	10.0
Maximum temp. (°C) during gelling	210	210	209	208
Time of maximum temp (min) during gelling	12	11	14	15
% increase in gel-time	4	4	11	10
After 400 days at 20-35°C				
VND gel-time (min)[b]	10.7	9.5	13	15
Maximum temp. (°C)	210	210	210	212
Time of maximum temp. (min)	13	12	18	21
% increase in gel-time	17	4	42	64
Change in weight of metal sample (grams)	[c]		+0.0043[d]	-0.0025[d]
Corrosion rate cm/400 days	-	-	-	0.00014

[a]VND solutions with 3.80% vanadium, 0.35% chloride, and 0.04% water. Stored in a nitrogen atmosphere..
[b]0.4 grams of promoter solution. The promoter solution is a 50% by weight mixture of vanadium trineodecanoate solution and 50% N,N-dimethyl-p-toluidine (DMT).
[c]The polypropylene bottle was deformed by xylene attack.
[d]After 400 days at room temperature, both metal samples were coated with a blue slime and had a tarnished surface even after cleaning.

TABLE 4. COST OF RAW MATERIALS FOR VND

Material	$/kg[a]	Unit of Issue	Kg/100-kg Batch	$/100-kg Batch
Nitrogen	-	Cylinder	-	10.00
Formic Acid	1.20[b]	500 lb drum	5.6	6.72
Hydrochloric acid	0.22[b]	30 gallon drum	68	14.96
Paraformaldehyde	1.40[b]	12 Kg drum	0.8	1.12
Vanadium Pentoxide	10.50[c]	100 lb drum	7.2	75.60
Xylene	0.60[b]	55 gallon drum	28	17.20
Triethyl-phosphate	3.00[d]	55 gallon drum	25.6	76.80
Neodecanoic Acid	1.99[e]	55 gallon drum	44.0	87.56
Celite	0.70[b]	100 lb bag	3	2.10
Other	-	-	-	27.94
				320.00
				or $3.20/kg

[a]Price December 1983
[b]Tilley Chemical Company
Baltimore, MD 21220 (301)574-4500
[c]Union Carbide Metals Division
Pittsburgh, PA 15205 (800)248-6272
[d]Eastman Kodak Company
Rochester, NY 14650 (716)254-1300
[e]Exxon Chemical Company
P.O. Box 3272
Houston, TX 77001 (800)231-6633

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3. D. W. Agers, J. L. Drobnick, C. J. Lweis, "The Recovery of Vanadium from Acidic Solutions by Liquid Ion Exchanger," General Mills Chemicals Inc., Minneapolis, Minnesota 55435.

4. M. M. Cook, J. A. Lander, "Sodium Borohydride Controls Heavy Metal Discharge," Pollution Engineering, December 1981.

5. R. G. Muller (Director), "1979 Directory of Chemical Producers in United States of America," SRI International, Menlo Park, California 94025, 1979.

APPENDIX A

COMPATIBILITY OF VND WITH HYDROCHLORIC ACID

When VND solutions decompose, the deep blue color changes to brown, solids form, and the gel-time changes from 7-10 min to over 40 min. VND solutions were heated at 90°C to 100°C with various amounts of 5 to 30% hydrochloric acid. In all tests, the solution decomposed in 6 to 8 hours of heating. Thus it is necessary to complete the removal of hydrochloric acid within 6 hours after the addition of NDA.

REMOVAL OF HYDROCHLORIC ACID

The excess and by-product hydrochloric acid is removed from vanadium trichloride and VND solutions by azeotropic distillation with xylene. Less than 20% of the distillate is the aqueous phase. At the start of this distillation there are two phases, the blue aqueous vanadium trichloride phas: and the top xylene triethylphosphate phase. Thus, good agitation is required to avoid bumping*. If agitation stops, restarting causes overpressurization of system and flooding of the condenser. At start of the distillation the VND solution is at 90°C. The distillation is continued until the aqueous phase of the distillate is about 3-5% hydrochloric acid. The neodecanoic acid is added, and the distillation is complete when the VND solution is heated to 150°C. If NDA is added when too much hydrochloric acid is present, black foamy solids form by acid attacking VND. If added when too little hydrochloric acid is present, the NDA precipitates the vanadium trichloride, forming blue solids. See Table 2 for typical distillation cut details. The addition of acid-stable emulsifying agents caused excessive foaming and is not recommended.

The removal of the dilute hydrochloric acid with the xylene azeotropic distillation was automated. The condensed xylene was returned to the reactor and the lower insoluble aqueous phase was removed through a Teflon® solenoid valve. Three types of xylene-water interface sensors were tested for controlling the solenoid valve. See Table 1 for batch tested.

*bumping is localized superheating leading to unstable distillation.

The Thermo-O-Watch capacitance level sensor controlled the interface in a 10mm glass tube. This unit works by detection of the difference in electrical capacitance in the xylene and the aqueous phase. This sensor worked well with 36 to 5% hydrochloric acid. It required frequent adjustments with lower concentrations.

The Dyna-sense electronic liquid level controller works by measurements of the conductance between platinum tipped glass probes. This sensor worked very well with all concentrations of hydrochloric acid. The probes were mounted in a glass tee with a neoprene stopper.

The Sensall ultrasonic liquid level sensor worked very well. This sensor was clamped on the outside of the 2 inch glass pipe. This unit works by detecting different transmittance of an ultrasonic signal in xylene and in the aqueous phase.

The ultrasonic sensor is the recommended controller. It works with all concentrations of hydrochloric acid and does not require entry into the piping.

APPENDIX B

DETAILED RECOMMENDED PROCEDURE FOR MAKING VND SOLUTIONS IN 3-KG BATCHES

$$V_2O_5 + 6HCl + 2\ HCOOH \xrightarrow[N_2]{80°\text{-}90°C} 2\ VCl_3 + 5H_2O + 2CO_2$$

$$VCl_3 + 3\ C_9H_{19}COOH \xrightarrow[\text{Xylene, } N_2\text{ Triethyl phosphate}]{100°\text{-}150°C} V(C_9H_{19}COO)_3 + 3HCl$$

By-product HCl not condensed

$$HCl + NaOH \xrightarrow[\text{Water}]{} NaCl + H_2O$$

Vandium trineodecanoate (VND) solution with 4% vanadium can be made in 3-kg batches with this procedure. The equipment illustrated in Figure 3 and listed in Table 5 consists of a 5-liter, three-neck flask with 29/42 standard taper joints. A spring clamp and copper wire keep each joint together.

ASSEMBLY OF EQUIPMENT

Reasonable substitution of equipment is acceptable.

A-1 Put 5-liter flask in heating mantle and clamp in place.
A-2 Assemble equipment as in Figure 1.
A-3 Install thermocouple in the flask's thermowell and connect to the Love controller - digital thermometer.
A-4 Plug the variable transformers into the controller. Plug heating mantle into the transformers.
A-5 Connect sonic level sensor and connect to controller. Plug solenoid valve into the controller.

A-6 Add the three sink-float standards to measure specific gravity (spgr) of the hydrochloric acid distillate. When the spgr is below the value the standard will sink.

SpGr	% HCl to Float Standard
1.02	3
1.03	6
1.05	10

Preparation of VND in 3-Kg Batches

B-1 Remove addition tee from 5-liter reaction flask and add:
252 grams of formic acid (90%)
2700 ml of hydrochloric acid (36%)
36 grams of paraformaldehyde

B-2 Turn on water to agitator bearing and turn on agitator
B-3 Slowly add 325 grams of vanadium pentoxide (V_2O_5) to the 5-liter flask.
B-4 Add 1200 ml xylene to the 5-liter flask.
B-5 Clamp addition-tee on the flask.
B-6 Turn on nitrogen purge at setting #35 (500 ml/min).
B-7 Turn on condenser water.
B-8 Turn on temperature controller and set for 85°C. Turn on and set transformer for 70 volts.
B-9 Agitate 5-liter flask for at least 3 hours at 80°-90°C.
B-10 Change temperature controller's set point to 110°C. (NOTE: agitation rate and set point should be changed to maintain rapid distillation without foaming. Reduce nitrogen purge to 100 ml/min (setting #10)).
B-11 Turn on liquid level sensor controller and collect dilute hydrochloric acid distillate.
B-12 Add 1152 grams triethylphosphate (TEP) to the 2-liter addition flask.
B-13 As dilute hydrochloric acid is distilled, slowly pump the TEP into the 5-liter reaction flask. Keep total volume under the original 4.5 liters.
B-14 Add 1992 grams prime-grade neodecanoic acid to a 5-liter addition flask.
B-15 When distillate is less than 3% hydrochloric acid:
-Add the neodecanoate acid with the tubing pump,
-Set temperature controller for 150°C,
-Set transformers to 100-110 volts.
B-16 Continue distillation until the temperature of the distillation flask is 150°C and the distillate contains no water.
B-17 Turn off transformers and cool product to room temperature.
B-18 Remove addition-tee and add 100 grams celite.

B-19 Assemble VND fitration system. The components are:
1. Three liter coarse glass Buchner funnel.
2. #12 Neoprene stopper.
3. Four liter filtering flask.
4. Connect and turn on water aspirator.

B-20 Mix 100 grams of celite with 100 ml of xylene and add to the 3-liter coarse-fritted glass Buchner funnel.

B-21 Filter by pouring the VND solution into the 3-liter Buchner funnel.

B-22 Add 200 ml xylene to the reaction flask, agitate and filter wash solution as in B-21 into a 3-liter bottle.

B-23 Weigh filtrates and send samples of VND solution and xylene wash for vanadium, water, and chloride analysis to:

Galbraith Laboratory
PO Box 4187
Knoxville, TN 37921
(615)546-1335

B-24 Calculate and add the amount of xylene wash and xylene needed to make a 4.0% vanadium solution.

B-25 Run gel time and time to maximum temperature.

B-26 Reaction flask can be cleaned with warm dilute hydrochloric acid collected in step B-11.

Table B-1. EQUIPMENT FOR MAKING VND IN 3-KG BATCHES

Item (See Figure 1)	Supplier (See Table 7)	Catalog #	Item Name
A	1	C-3202-20	Shielded flow meter size #2
B	1	C-6406-74	Teflon 3/8 inch FEP tubing
C	1	K-7553-10	Tubing pump 1 to 100 RPM
C-1	1	K-7018-20	Pump head
C-2	1	K-6412-48	Viton tubing
D	11	14-141H	Neoprene stopper size 6 1/2
E	2	5055-15	Claisen adapter
F	2	6947-64	5000-ml, three 29/42 T neck flask
G	2	12053-25	Electric heating mantle
G-1	2	12081	Powerstat variable electric transformer
H	24	6500-05	Thermometer well
H-1	11	13-280	Thermometer 308 mm immersion
H-2	7	1518-52-9	Type K thermocouple
H-3	7	149-786	Love controller
I	4	K150750-343	Adapter
J	1	K-4685-00	Heavy duty air driven stirrer
J-1	11	14-513-65B	Glass stirrer with Teflon paddle
J-2	11	14-513-60A	Jacketed bearing

Table B-1. EQUIPMENT FOR MAKING VND IN 3-KG BATCHES (CONT)

Item (See Figure 1)	Supplier (See Table 7)	Catalog #	Item Name
K	11	15-002C	Thermometer
L	2	5035-15	Straight adapter
M	2	5265-15	Gas inlet adapter
N		6608-22	Distilling head
O	2	5998-64	Condenser
P	2	7565-23	Full length joint size 14/35
P-1	2	5005-20	Reducing adapter
R	12	L6-1000	Thermo-O-Watch or sonic sensor
S	8	DV2-146NCA1	Teflon^{R} solenoid valve
Not Shown			
T	4	K-955000-3032	3-liter coarse fritted glass Buchner funnel
T-1	5	56388-280	#12 neoprene stopper
T-2	2	6982-25	4-liter filtering flask

APPENDIX C

DETAILED RECOMMENDED BATCH PROCEDURE FOR MAKING VND SOLUTIONS IN 15-KG BATCHES

$$V_2O_5 + 6HCl + 2\ HCOOH \xrightarrow[N_2]{80\text{-}90^\circ C} 2\ VCl_3 + 5H_2O + 2CO_2$$

$$VCl_3 + 3\ C_9H_{19}COOH \xrightarrow[\text{Xylene} + N_2]{100^\circ\text{-}150^\circ C} V(C_9H_{19}COOH)_3 + 3HCl$$

Vanadium trineodecanoate (VND) solution with 4% vanadium can be made in 15-Kg batches with this procedure. The equipment illustrated in Figure 4 and listed in Table 6 consists of the 22-liter flask, 2-inch beaded glass pipe fittings, and heat exchangers. Each joint of the beaded glass pipe has 3° of deflection and can withstand 15 psig.

ASSEMBLE THE EQUIPMENT

Reasonable substitution of equipment is acceptable. The 22-liter flask and heating mantle can be replaced with a 10-gallon steam heated glass lined agitated reactor.

A-1 Put 22-liter flask in the heating mantle and clamp in place.
A-2 Assemble equipment as in Figure 5 using beaded pipe couplings.
A-3 Install thermocouple in the flask's thermowell and connect to the Love controller - digital thermometer.
A-4 Plug the two variable transformers into the controller. Plug heating mantle into the transformers.
A-5 Connect sonic level sensor and connect to controller. Plug solenoid valve into the controller.
A-6 Add the three sink-float standards to measure specific gravity (spgr) of the hydrochloric acid distillate. When the spgr is below the value, the standard will sink.

SpGr	% HCl to Float Standard
1.02	3
1.03	6
1.05	10

Table C-1. EQUIPMENT SUPPLIERS FOR MAKING VND IN 15-KG BATCHES

Item From Figure 5	Supplier See Table 7	Catalog Item
A	1	C-3202,20, Shielded flowmeter, size #2
B	1	C-6406-74, Teflon FEP 3/8-inch OD tubing
C	2	Adapter, 2-inch beaded glass pipe to Ace #11 threads, Teflon bushing and FETFE o-ring.
D	3	72-2287, 2-inch glass beaded tee
E	1	K-6833-10 tubing clamp
F	4	K-784000, Modified heavy duty stirrer bearing modified with 2-inch beaded pipe
G	4	K-784010-0005, Heavy duty (19mm OD, glass shaft) with K-784030-0005 Teflon stirrer blade.
H	1	C-4685-00, Heavy duty air-driven stirrer.
H-1	5	56433-104, Vacuum tubing 3/4-inch ID, 4 inches long
H-2	1	C-6403-05, Hose clamp
I	3	72-6422, Sanitary Y-branch, 2-inch beaded pipe
J	2	8852-09, Thermowell with beaded pipe and 6 inches long.
J-1	6	870 Keithley digital thermometer with type K thermocouple.
K	4	K-726000-2200 Heating mantle for 22-liter flask.
K-2	5	62546-455 Variable transformer
L	4	K-606200 Modified 22-liter flask with three 2-inch beaded pipe fittings and thermowell.
L-1	7	150-876, Love controller for type K thermocouple-digital read out.
L-2	7	F4-250-600 KA33-B15, Type K thermocouple
M	2	Adapter, 1-inch beaded glass pipe to Ace #11 threads
N	3	72-2086, Beaded glass tee 2 x 1-inch
O	3	72-2062, Beaded glass pipe 2 inches, 6 inches long.
P	3	72-20122, Beaded glass pipe, 12 inches long.
Q	3	HE-2 QVF glass Coil condenser
Q-1	3	Flange system to connect QVF condenser to beaded glass pipe
R	8	DV2-146NCA1 Teflon solenoid valve.

TABLE C-1. EQUIPMENT SUPPLIERS FOR MAKING VND IN 15-KG BATCHES (CONT)

Item From Figure 5	Supplier See Table 7	Catalog Item
S	9	621S Sonic liquid level sensor for 2-inch glass pipe (OD 2-5/8 inches)
S-1	9	400 C Liquid controller unit
T	1	K-7380-62 vacuum gauge
T-1	1	K-6469-86 vacuum control needle valve
T-2	1	K-6378-00 water-powdered vacuum pump
Not Shown		
U	3	72-1521, Coupling for 2-inch beaded pipe
V	3	72-1519, Coupling for 1-inch beaded pipe
W	5	21572-104, Column clamp
X	4	K-955000-3023, 3-liter coarse fritted glass Buchner funnel
X-1	4	K-15001000-026 Adapter
X-2	5	56388-247 #10 Neoprene stopper
Y	10	Sink-Float standards, 1.02 SpGr 1.03 SpGr, 1.05 SpGr
Z	1	K-7589-19 Air powered tubing pump with K-6412-73 Viton tubing

Table C-2. SUPPLIERS OF EQUIPMENT FOR MAKING VND

1. Cole Parmer, 7425 North Oak Park Avenue, Chicago, Illinois (800)323-4343

2. Ace Glass, P.O. Box 688, Vineland, New Jersey 08360 (609)692-3333

3. Sentinel Process Systems (Corning), P.O. Box 67, Hatbro, Pennsylvania 19040 (215)675-5700

4. Kontes Glass, P.O. Box 739, Vineland, New Jersey 08360 (609)692-8500

5. VWR Scientific, 6601 Amberton Drive, Baltimore, Maryland 21227 (301)796-8500

6. Scientific Associates, 9512 Lee Highway, Fairfax, Virginia 22031 (703)385-0600

7. W. H. Cook and Co., P.O. Box 263, Finksburg, Maryland 21048 (301)833-8200

TABLE C-2. SUPPLIERS OF EQUIPMENT FOR MAKING VND (CONT)

8. Fluorocarbon, P.O. Box 3640, Anaheim, California 92803
(714)956-7330

9. Briggs and Cooper Controls, P.O. Box 7229, Wilmington, Delaware 19803
(302)478-3548

10. Cargille Laboratory, Inc., Cedar Grove, New Jersey 07009
(201)239-6633

11. Fisher Scientific, 7722 Fenton Street, Silver Spring, Maryland
(301)587-7000

12. Instrument for Research and Industry, 108 Franklin Avenue, Cheltenham, Pennsylvania 19012
(215)745-4440

A-7 Assemble VND filtration system (not shown in Figure 5). Starting with direction of flow the components are:
1. 3/8-inch OD feeling tubing
2. Viton tubing pump
3. 3-liter coarse Buchner funnel
4. #10 neoprene stopper
5. Adapter
6. 2nd Viton tubing pump
7. 3/8-inch OD tubing
8. Clean 22-liter flask for storing VND solution

Preparation of VND in 15-Kg Batches

B-1 Remove addition-tee from 22-liter flask and add:
840 grams formic acid (90%)
10,600 grams hydrochloric acid (36%)
120 grams paraformaldehyde
B-2 Turn on water to agitator bearing and turn on agitator
B-3 Slowly add 1080 grams of vanadium pentoxide to 22-liter flask. (Should take 30 to 45 minutes)
B-4 Clamp addition-tee on the flask
B-5 Turn on nitrogen purge at setting #50 (1000 ml/min)
B-6 Turn on condenser water
B-7 Turn on water-powered vacuum pump
B-8 Keep system at 10 inches of vacuum by adjusting vacuum control needle valve.
B-9 Turn on temperature controller and set for 85°C. Turn on and set transformer for 70 volts.
B-10 Agitate 22-liter flask for at least 3 hours at 80°-90°C.
B-11 Add 4 liters of xylene.

B-12 Change temperature controller's set point to 110°C. (NOTE: agitation rate and set point should be changed to maintain rapid distillation without foaming. Reduce nitrogen purge to 200 ml/min (setting #20).

B-13 Turn on liquid level sensor controller and collect dilute hydrochloric acid distillate.

B-14 Add 3800 grams triethylphosphate (TEP) to the 5-liter addition flask.

B-15 As dilute hydrochloric acid is distilled, slowly add the TEP into the 22-liter reaction flask, by opening tubing clamp. Keep total volume under the original 16 liters.

B-16 Add 6640 grams prime-grade neodecanoic acid to a 12-liter addition flask.

B-17 When distillate is less than 3% hydrochloric acid:
-Add the neodecanoate acid by opening tubing clamp.
-Set temperature controller for 150°C.
-Set transformers to 100-110 volts.

B-18 Continue distillation until the temperature of the distillation flask is 150°C and the distillate contains no water.

B-19 Turn off transformers and cool product to room temperature.

B-20 Remove addition-tee and add 100 grams of celite.

B-21 Mix 100 grams celite with 100 ml xylene and add to the 3-liter coarse fritted glass Buchner funnel.

B-22 The VND solution is filtered using apparatus assembled in step A-7 by:
-Inserting 3/8-inch tube into the VND flask
-Turning on 2nd tubing pump
-Turning on other tubing pump
-Continuing filtration until all VND has been filtered. Do NOT allow the filter to overflow!
-Turn off pumps.

B-23 Add 1000 ml xylene to the 22-liter reaction flask, agitate and filter wash solution as in B-22 into a bottle.

B-24 Weight filtrates and send samples of VND solution and xylene wash for vanadium, water, and chloride analysis to:

Galbraith Laboratories
P.O. Box 4187
Knoxville, TN 37921
(615)546-1335

B-25 Calculate and add the amount of xylene wash and xylene needed to make a 4.0% vanadium solution.

B-26 Send sample for vanadium analysis, and run gel time and time to maximum temperature.

B-27 Reaction flask can be cleaned with warm dilute hydrochloric acid collected in step B-13.

APPENDIX D

MEASUREMENT OF GEL-TIME AND TIME AT MAXIMUM TEMPERATURE:

Gel-time was measured by a Sunshine gel-time meter*, model #22, which measures gel-times to 0.1 minutes. This meter operates by a glass rod suspended from a torsion spring rotating in the sample. The glass rod is connected to the torsion spring by magnet coupling. When gel-point is reached, the torsion spring twists, making contacts that stop the timer and ring a buzzer. The temperature was measured using a 870 Keithley digital thermometer.

A promoter solution of 50% VND and 50% N,N-dimethyl-p-toluidine (DMT) was mixed in a glass sample bottle.

The gel-time measurement procedure follows:

1. Add 100 grams polyester resin** to a 150 ml disposable polypropylene beaker.
2. Add 0.4 grams VND-DMT promoter solution. Start timer on gel-meter and auxiliary timer and mix for 0.5 minutes with the glass rod (other quantities of promoter solution may be used).
3. Add 1.1 grams cumene hydroperoxide catalyst and mix for 0.5 minutes.
4. Clamp the sample under gel-meter and magnetically couple the glass rod to the torsion spring.
5. When gel-point is reached, the buzzer sounds and the time is noted.
6. A silicone grease-coated immersion thermocouple probe is inserted in the gel and the time noted at maximum temperature on the auxiliary timer.

*Sunshine Scientific Instrument, 1810 Grant Ave., Philadelphia, PA 19115

**Polyester Resin CRS 503381

APPENDIX E

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

Form Approved
OMB No. 44-R1387

MATERIAL SAFETY DATA SHEET

Required under USDL Safety and Health Regulations for Ship Repairing, Shipbuilding, and Shipbreaking (29 CFR 1915, 1916, 1917)

SECTION I

MANUFACTURER'S NAME	EMERGENCY TELEPHONE NO.
Naval Surface Weapons Center	
ADDRESS (Number, Street, City, State, and ZIP Code) White Oak, Maryland 20910	
CHEMICAL NAME AND SYNONYMS	TRADE NAME AND SYNONYMS
CHEMICAL FAMILY Polyester promoter	FORMULA

SECTION II - HAZARDOUS INGREDIENTS

PAINTS, PRESERVATIVES, & SOLVENTS	%	TLV (Units)	ALLOYS AND METALLIC COATINGS	%	TLV (Units)
PIGMENTS			BASE METAL		
CATALYST			ALLOYS		
VEHICLE			METALLIC COATINGS		
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX		
ADDITIVES			OTHERS		
OTHERS					

HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES	%	TLV (Units)
Vanadium trineodecanoate	45	
Triethyl phosphate	25	
Xylene	27	100ppm
Neodecanoic acid	3	

SECTION III - PHYSICAL DATA

BOILING POINT (°K) 150°C(302°F)		SPECIFIC GRAVITY (H_2O=1)	1.1
VAPOR PRESSURE (mm Hg.)		PERCENT, VOLATILE BY VOLUME (%)	30
VAPOR DENSITY (AIR=1) 4		EVAPORATION RATE (_____ =1)	
SOLUBILITY IN WATER Negligible			
APPEARANCE AND ODOR Blue solution			

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used)	FLAMMABLE LIMITS	Lel	Uel
greater than 80°F	Not available		

EXTINGUISHING MEDIA Use National Fire Protection Association (NFPA) Class B extinguishers (carbon dioxide, dry chemical or foam) designed to extinguish NFPA Class IC flammable liquid fires. If water is used, fog nozzles are preferable.

SPECIAL FIRE FIGHTING PROCEDURES

UNUSUAL FIRE AND EXPLOSION HAZARDS Keep containers tightly closed. Isolate from heat, electrical equipment, sparks and open flame. Closed containers may explode when exposed to extreme heat. Do not apply to hot surfaces.

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SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE

xylene..Toxic vapors in high concentration are anesthetic.

EFFECTS OF OVEREXPOSURE

Irritant to skin and upper respiratory system. Wear self-contained breathing apparatus. Vanadium causes skin irritation after prolonged or repeated contact and is poisonous if swallowed.

EMERGENCY AND FIRST AID PROCEDURES

Fumes: Remove from exposure. Restore breathing. Keep warm and quite. Notify a physician. Splash (eyes), Flush immediately with copious quantities of running water for at least 15 minutes. Take to a physician for definitive medical treatment if necessary. Splash (skin): Was affected areas with water. Remove contaminated clothing.

SECTION VI - REACTIVITY DATA

STABILITY			CONDITIONS TO AVOID
	UNSTABLE		
	STABLE	X	

INCOMPATABILITY *(Materials to avoid)*

Peroxides

HAZARDOUS DECOMPOSITION PRODUCTS

Vanadium compounds, and CO

HAZARDOUS POLYMERIZATION			CONDITIONS TO AVOID
	MAY OCCUR		
	WILL NOT OCCUR	X	

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Remove all sources of ignition (flames, hot surfaces, and electrical, static or frictional sparks). Avoid breathing vapor. Ventilate area. Absorb with filter aid or dry powder and shovel up.

WASTE DISPOSAL METHOD

Dispose in accordance with local, state, and federal regulations. Incinerate in approved facility. Do not incinerate closed containers.

SECTION VIII - SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION *(Specify type)*

Not normally needed

VENTILATION	LOCAL EXHAUST	SPECIAL
	Sufficient to avoid accumulationg of fumes	
	MECHANICAL *(General)*	OTHER

PROTECTIVE GLOVES	EYE PROTECTION
Impervious to oil	Chemical goggle

OTHER PROTECTIVE EQUIPMENT

Sufficient to avoid skin contact

SECTION IX - SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING

Product is flammable. Do not store in high-temperature areas or in areas of fire or open flame.

OTHER PRECAUTIONS

Do not take internally. Avoid prolonged contact with the skin. Keep containers closed when not in use.

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