

THE SOLUBILITY OF VINYL CHLORIDE IN POLY(VINYL CHLORIDE)

by

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ABSTRACT

The solubility of vinyl chloride monomer (VCM) in PVC powders has been studied as a function of VCM partial pressure (P_m) at temperatures from 30° to 110°C. Data at $P_m < 1$ atm. were obtained by electrobalance sorption techniques and at higher P_m by equilibrium vapor-pressure measurements. Above T_g , the solubility follows Henry's law for low P_m , with a solubility in mg VCM/g PVC of $S = 88 (P_m/P_m^0)$, independent of PVC molecular weight and resin type over the commercial range. As T is reduced below T_g , the solubility at a given P_m increases considerably; the solubility-pressure relationship becomes increasingly non-ideal and dependent upon time, resin type, and sample history. These results may be interpreted through the dual-mode sorption concept of Michaels, Vieth, and Barrie: Solubility includes normal dissolution, proportional to P_m , plus a "hole-filling" contribution which approaches saturation with increasing P_m . It appears that the dissolution contribution is temperature-independent. The "hole-filling" contribution disappears above T_g , but may provide a major part of the solubility at $T \ll T_g$. Changes in the available volume of "holes" through swelling and relaxation processes seem to account for the observed effects of time and sample history on the solubility of VCM in PVC.

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INTRODUCTION

Studies of the solution and transport of small molecules in polymers are important both in their direct bearing on practical problems and in their contribution to basic understanding of polymer structure and properties. Both of these aspects are illustrated by our current study of sorption of vinyl chloride monomer (VCM) by poly(vinyl chloride) (PVC). The immediate practical problem results from concern about potential health hazards from exposure to VCM (1). The removal of residual VCM from PVC is consequently a matter of current importance to the entire PVC industry. Presentation of some basic solubility and diffusion data pertinent to VCM removal processes is the principal purpose of this and a companion paper. (2) These data also are useful in predicting the extent and rate of VCM escape from PVC products into their environment.

From a more fundamental viewpoint, the VCM/PVC system is especially interesting in exhibiting most of the complexities and non-idealities of behavior reported in recent studies of other organic vapor/glassy polymer systems (3), and perhaps some new complications as well. For example, the solubility of VCM in PVC is highly dependent upon the polymer type and history, as well as upon time, temperature, and VCM partial pressure, (P_m). Solubility measurements, perhaps using other small molecules as well as VCM, offer a sensitive and useful way to study and follow changes in PVC structure.

While solubility and diffusion are closely interwoven topics, it seems useful to emphasize each one separately. In this paper, we concentrate on solubility (the amount of VCM sorbed or retained by PVC) under varied conditions. This is the type of data needed, for example, to estimate the equilibrium distribution of VCM between PVC and its vapor or liquid environment. In an accompanying paper (2), we consider the diffusion coefficient, which controls the rate of VCM uptake or removal and hence the time required for equilibration of VCM content between PVC and its environment.

EXPERIMENTAL

Materials: Polymer samples used in this study included a number of commercial and experimental PVC homopolymers made by suspension, emulsion, and mass polymerization techniques. All were used in the powder form obtained directly from the polymerization process by normal recovery and drying procedures. Particle structures were characterized by optical and electron microscopy and by nitrogen-adsorption surface-area measurements. Specific results are given in this paper for the samples listed in Table I.

TABLE I

PVC Samples Studied

Sample	Type	Intrinsic Viscosity	Surface area, m ² /g
A	Suspension	~ 1.10	2.3
B	"	~ 0.95	0.8
C	"	~ 0.70	< 0.1
D	Emulsion	1.58	9.8
E	"	1.53	19.4

Methods: Solubilities of VCM in PVC at P_m above 1 atmosphere were obtained by measuring the pressure at apparent equilibrium in stirred, thermostatted autoclaves containing weighed amounts of dry PVC powder and VCM. (1)

vapor density, vessel volume, and PVC weight and density, calculation of the amount of dissolved VCM is straightforward. For comparison, solubilities of VCM in water and in 50/50 ethanol/water were similarly determined.

More extensive and precise measurements of VCM solubility in PVC at P_m below 1 atmosphere were made gravimetrically with a recording electrobalance (Model RG, Cahn Division, Ventron Instruments Corp., Paramount, California). The balance was mounted in a glass vacuum-chamber with connections to a source of VCM vapor and to vacuum. PVC samples were suspended in a light aluminum pan near the bottom of a 40 cm. Kovar hangdown tube, which was immersed in a circulating liquid bath for temperature control. Sample weights were from 100 to 500 mg; use of the balance at 1 microgram sensitivity thus detected weight changes of 2 to 10 ppm. Our standard procedure was to evacuate the sample on the balance to constant weight, then admit VCM vapor to various pressures and record weight changes. Pressures were measured to ± 0.1 mm Hg with a strain-gage transducer and digital voltmeter. Because of the large vapor volume and small sample size, sorption produced negligible pressure change after an addition of VCM vapor. Saturated vapor pressures of VCM up to 60°C were interpolated from the data of Dana, et al. (4), and for higher T's were estimated from the Rydell-Planck-Miller correlation (5).

RESULTS

VCM Solubility in Water and in Ethanol/Water: As a point of reference, Figure 1 shows our data for VCM solubility in H_2O at 30° and 50°C and in 50/50 EtOH/ H_2O at 30°, plotted against P_m/P_m^0 , where P_m^0 is the saturated vapor pressure of VCM at the temperature of measurement. The data follow Henry's law, with a slope that appears independent of temperature, over most of the pressure range.

VCM Solubility in PVC at $P_m > 1$ atm: Figure 2 shows the results of our equilibrium vapor pressure measurements on the VCM/PVC system at temperatures from 30 to 60°C. The deviation from Henry's law behavior is substantial, the data qualitatively following the Flory-Huggins theory for a polymer-solvent system. Above $P_m/P_m^0 \approx 0.5$, the data for four temperatures follow the same curve within our experimental accuracy and indicate a saturation solubility at P_m^0 of about 300 mg VCM/g PVC. At lower pressures, however, S shows a distinct increase with decreasing temperature. Extrapolation of these data would suggest a disturbing finite solubility at $P_m = 0$. We also noted, in the low P_m/P_m^0 range, that the solubility appeared to depend on the direction of approach to equilibrium; measurements while decreasing pressure gave higher apparent solubility than those during pressure increases. Clearly there were some effects at low VCM pressures that demanded closer study.

VCM Solubility in PVC at low P_m , $T > T_g$: At 90°C, just above PVC's glass-transition temperature (T_g), of $\sim 85^\circ C$, our electrobalance solubility data extend only up to $P_m/P_m^0 \approx 0.05$, since $P_m^0 = 13500$ mm. Over this range, VCM-in-PVC solubility closely follows Henry's law; the solubility S , in mg VCM/g PVC, is given by $S = 88(P_m/P_m^0)$. This relation, as shown in Figure 2, provides a very reasonable asymptote to the data at higher P_m and lower T . Thus data from two different techniques seem quite consistent and complementary.

Figure 3 shows the details of electrobalance sorption/desorption runs at 90°C on four different PVC samples. In these runs, a 10-minute equilibration time was allowed at each P_m , and the samples were held 60 minutes at $P_m \approx 700$ mm between sorption and desorption runs. The linearity and constancy of slope is quite striking, showing that S under these conditions is independent of PVC molecular weight and particle structure over the ranges encountered in commercial products. The slight "hysteresis loop" shown by samples B and C may be attributed to a relatively slow approach to diffusion equilibrium in these lower surface-area polymers (2).

We may note here that the simple solubility behavior at $T > T_g$ provides the basis for a rapid and reliable analytical procedure for residual VCM in PVC resins. One simply needs to heat a PVC powder sample in a closed container to 90°C , then determine the pressure, or the VCM content of the vapor by gas chromatography, in order to determine the original VCM concentration in the PVC. Details and results will be published separately (6).

VCM Solubility in PVC at low P_m , $T < T_g$: Typical electrobalance sorption/desorption runs at 50 and 70°C are shown in Figure 4. In these experiments, equilibration time at each pressure point was 30 minutes, and the samples were held at $P_m = 700$ mm for ~ 18 hours between sorption and desorption runs. In sharp contrast to the 90° results, we now see marked deviations from Henry's law behavior and pronounced hysteresis: Solubilities on desorption are higher than on sorption, both lie above the 90°C Henry's law line, and the deviations become greater the farther below T_g is the temperature. We also see pronounced differences between PVC resins in sorption/desorption behavior at $T < T_g$, as illustrated in Figure 5; the pressure-time sequence for these runs was the same as for Figure 4. Diffusion equilibrium for the high-surface, emulsion polymer (Sample D) is reached in much less than 30 minutes at 50°C (2), hence the hysteresis loop cannot be attributed to diffusional non-equilibrium.

Sample History and Time Effects: In an effort to better understand the hysteresis effect and the deviations from Henry's law behavior, we are studying the effects of sample history. Some of our early results are illustrated in Figure 6, where each point is now the equilibrium solubility reached in several hours after bringing the sample from vacuum to the indicated P_m/P_m^0 at 30°C . The original sample had been stored at room temperature for about 10 years before use. The "annealed" sample was heated to 100°C and cooled to room temperature over about 18 hours. The "pre-swollen" sample was conditioned in VCM vapor for 4 days at 30° and $P_m = 700$ mm, its VCM content reaching 38 mg/g, then evacuated for 4 hours to remove all the sorbed VCM; solubility measurements were made the same day the conditioning VCM was removed, and another after holding under vacuum for one week at 30° . The results show a substantial reduction in VCM solubility upon annealing, and an increase upon preswelling; the effect of preswelling, however, had largely disappeared after a week.

To follow the apparent relaxation of the preswelling effect in more detail, a sample of polymer E was conditioned 80 hours at 30° , $P_m = 700$ mm and 38 mg/g VCM content, then evacuated for one hour; repeated measurements of VCM solubility were then made at $P_m/P_m^0 = .01$. Equilibration times of 15 min. were allowed, and the sample was kept under vacuum between measurements. Figure 7 shows how the VCM solubility changed with time after removal of the conditioning VCM. The data resemble relaxation curves obtained by thermal analysis (7) or by changes in diffusion coefficient (8) in other glassy polymers. It appears that solubility measurements in polymer/vapor systems offer another way to follow glassy-state relaxation processes in polymers.

DISCUSSION

Interpretation of Results: It seems that our results can most reasonably be explained in terms of the dual-mode sorption concept of Michaels, Vieth, and Barrie (9). These authors consider the solubility of a vapor in a glassy polymer to be the sum of a normal dissolution process, which follows Henry's law, and a "hole-filling" contribution, which approaches saturation with increasing partial pressure of the vapor. In the VCM/PVC system, this concept may be applied as shown schematically in Figure 8. The normal dissolution contribution follows a Flory-Huggins behavior, approaching Henry's law as a limit at low P_m/P_m^0 . Above T_g , no "hole-filling" contribution is seen; this may mean that the relaxation

time for segmental motion is shorter than the experimental observation time, so that "holes" do not persist long enough to be detected by solubility measurements. As T is lowered below T_g , an increasing volume of holes becomes frozen in, or the lifetime of holes is increased, and the hole-filling contribution to solubility becomes increasingly significant. Our data at $P_m > 1$ atm (Figure 2) indicate that the hole-filling contribution is seen only over a limited composition range at each temperature. We suggest that this is due to plasticization of PVC by VCM; the hole-filling contribution to solubility disappears when the composition and temperature of the system bring it from the glassy to the rubbery state.

Within the glassy region, the solubility varies with sample history and time. We may attribute this to variations in volume of "holes" and relate the solubility changes to the well-known effects of polymer history on free-volume in the glassy state (10). We suggest that for fine-particle polymers, where diffusion equilibrium is reached rapidly, short-time vapor-in-polymer solubility data offer a direct measure of the free-volume state of the polymer at a given time; i. e., the "excess solubility" (total solubility minus the normal dissolution contribution) may be directly proportional to the excess volume or excess enthalpy determined by other techniques. Our attempts to check this by density measurements have so far been inconclusive; thermal analysis studies are in progress.

Explanation of most of our experimental results follows directly from the above concepts if we also recognize that the sorption process itself introduces a swelling stress; slow relaxation of this stress seems likely to increase hole volume and consequently produces time-dependence of solubility. This effect would explain the hysteresis of sorption/desorption experiments. To account for the effect of preswelling, we need only suggest that VCM can diffuse out of PVC much faster than can the holes it produces; subsequent resorption then results in VCM solubility greater than that of the original samples.

We tentatively ascribe the differences in VCM solubility in different types of PVC (e. g., emulsion vs. suspension, Figure 5) to an effect of particle structure. Emulsion PVC's consist of discrete spherical particles $< \sim 1\mu$ in diameter; in suspension PVC's, primary micron-size particles are bonded together into more or less porous, well-knit agglomerates of roughly 100μ diameter. (11) We suggest that the more limited extent of continuous polymer networks in emulsion PVC, relative to suspension PVC, results in a lower resistance to swelling, and therefore, in a greater "excess solubility."

Further studies of particle structure effects, thermal and solvent history effects, and other intriguing aspects of the solubility of VCM and other small molecules in PVC are part of our planned continuing study.

ACKNOWLEDGEMENTS

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Figure 1. VCM SOLUBILITY in WATER and in 50/50 ETHANOL/WATER

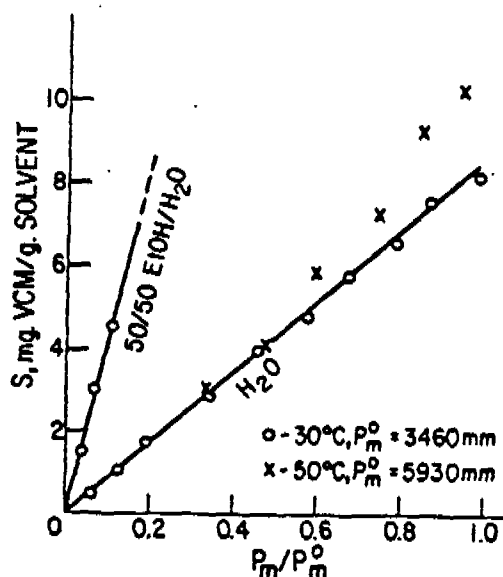


Figure 2. VCM SOLUBILITY in PVC at $P_m > 1$ ATM

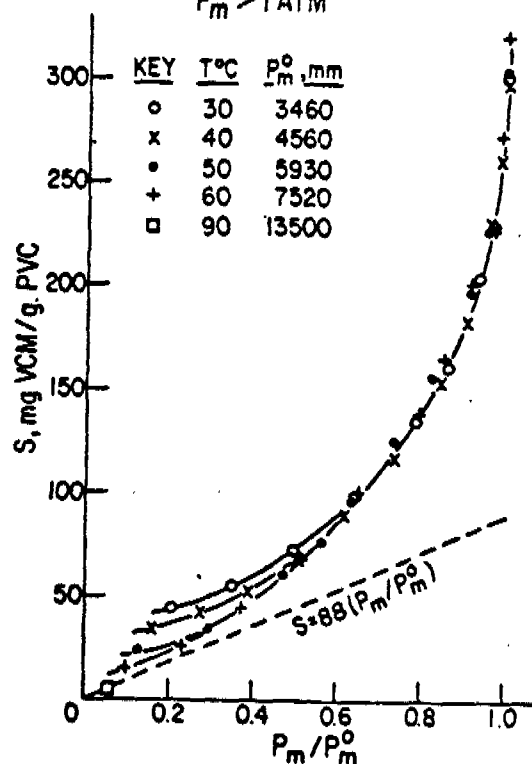


Figure 3 VCM SORPTION/DESORPTION at 90°C

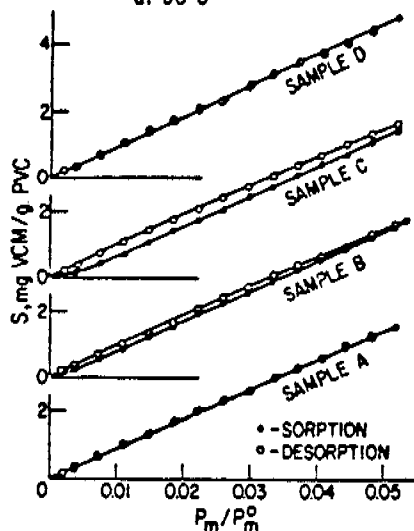


Figure 4 VCM SORPTION/DESORPTION on PVC, SAMPLE A

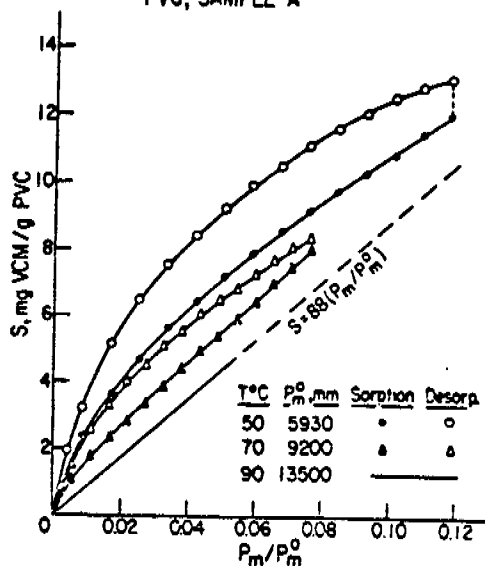


Figure 5 VCM SORPTION/DESORPTION, TWO SAMPLES at 50°C

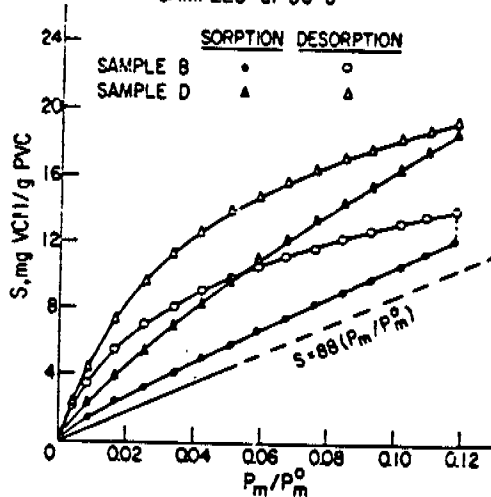


Figure 6 HISTORY EFFECTS on VCM SOLUBILITY, SAMPLE D, 30°C

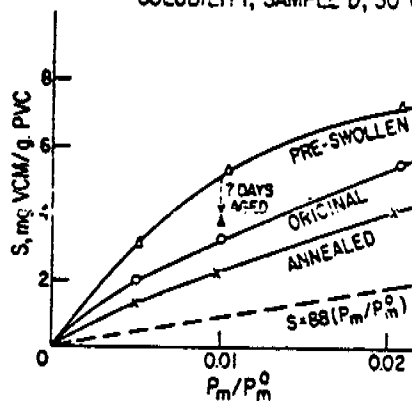


Figure 7 CHANGE of VCM SOLUBILITY with TIME, PRE-SWOLLEN SAMPLE E, 30°C

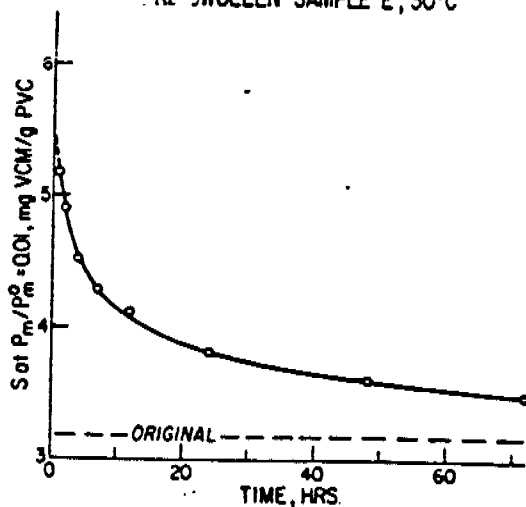
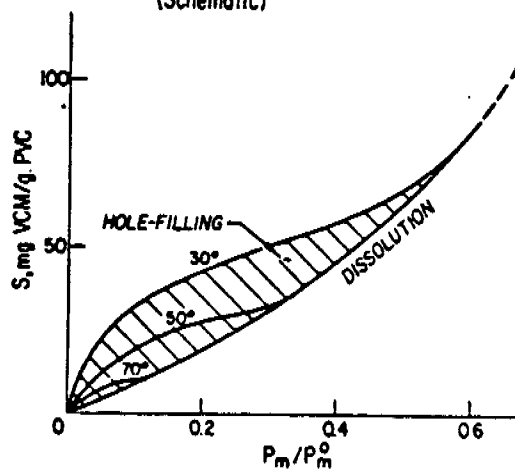


Figure 8 DUAL MODE SORPTION of VCM by PVC (Schematic)



ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: UNLOADING VINYL CHLORIDE	ASPI 1013
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GENERAL (continued)

Vinyl Chloride Monomer is generally received in 20,000 or 30,000 gallon tank cars and placed by the railroad at our 6 unloading stations. Each car being hooked up will be grounded by attaching the grounding clips at each station to each car. Each car will be hooked up by the unloader to the unloading boom by attaching the 1-1/2" stainless steel flex hose to the vapor valve at the tank car dome as well as the two stainless steel liquid unloading hoses to the 2" valves. This is accomplished by removing the plugs from the three valves at the dome of the car and installing the flanged elbows to the reducing bushing at each valve. Each respective flex hose is then attached to the flanged elbows being sure that the gasket is in the correct position before tightening the connection. Monomer, from the storage tanks converted to vapor, is then used to pressure the liquid out of the car to the storage tanks. After the liquid transfer, the vapor in the car is reduced to 3-8 psig (depending on the outside temperature). This is done by pulling down the pressure in the car by using the compressors to pull the vapor from the car, compress it, and send it to the storage tank.

After unloading, the unloader will disconnect the car from the unloading boom and turn the tank car placards to show empty. After making sure that the catwalks at the unloading station are clear of the car, the car is ready to be pulled by the railroad.

UNLOADING LIQUID MONOMER

Monomer from the storage tanks is fed to the vaporizer by opening the valve on the main feed line and both valves at the vaporizer automatic control valve. The two 2" valves on the discharge line of the vaporizer are then opened to feed vapor to the unloading racks. The 1-1/2" vapor valve on the unloading rack and the vapor on the car are then opened. Open both liquid valves on the tank car, slowly to avoid popping the excess flow valve, and check for leaks at all connections, * then open the liquid valve on the boom. Back in the compressor building, open both block valves at each of the four site glasses on the liquid lines to the storage tanks. Open the steam feed valve to the vaporizer located on East wall in compressor room. Maintain a level in the vaporizer site glass by controlling the monomer feed to the vaporizer with the by-pass valve. The level in the site glass should be at a point 3/4 of the way up.

* Leaks at connections can be detected by the appearance of snow-like material at major leaks or by the use of soap bubbles at small leaks.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT:	ASPI 1013
UNLOADING VINYL CHLORIDE	PAGE 3 OF 4 PAGES

UNLOADING LIQUID MONOMER (continued)

Check for monomer flow at the site glass on the monomer line to the storage tanks and after flow is evident open the by-pass valve and close the two site glass valves. Recheck the level at the vaporizer to see if it is holding.

Continue unloading with periodic checking for leaks at the tank car and for the end of the liquid flow at the site glasses. Experience will show what unloading times can be expected and when checks should be made. The unloader will check at least once during the liquid unloading for any leaks at the car and correct same. Evidence of the approach of the end of the liquid flow is when the level in the site glass drops as well as bubbles will appear in the site glass.

VAPOR PULL DOWN

Bubbles showing in the flow site glass are an indication that the liquid is nearly unloaded from the car. Unloading will continue until vapor only is flowing through the site glass and the flapper vibrates about 1/2 inch. After liquid is out of the car shut off the monomer flow to the vaporizer by closing both valves at vaporizer inlet. Close both valves on the discharge side of vaporizer as well as the steam flow to the vaporizer. Shut off the liquid lines by closing all site glass valves and site glass by-pass valves.

To set up for vapor pulling open the 4 red valves on the line from the compressors to the vapor line to the cars (the 4 valves are colored red). Open the two orange valves, depending which tanks the vapor is to go to on the discharge line of the compressor to the liquid line to the storage tanks. Start the compressors. After starting the compressors, shut the liquid valves on the unloading boom. Then recheck the compressor to see if they could be liquid-locked. If so, stop the locked compressor and close the corresponding red valve on the compressor inlet line and open the two valves on the compressor bleed off line which will allow liquid and some vapor to be forced from the compressor to the atmosphere.

ASSONET STANDARD PRACTICE INSTRUCTION

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UNLOADING VINYL CHLORIDE	PAGE 4 OF 4 PAGES

VAPOR PULL DOWN (continued)

After all the liquid is forced from the compressor and the check valve re-seats itself, close the two bleed-off valves and open the corresponding compressor inlet valve. Start the compressor to pull the vapor, once the inlet to the compressor becomes hot, it is safe to assume that the compressor will not shut down. After following the vapor pull down on the tank car vapor line gauges, experience will show what pull down times can be expected and how often checks should be made. Continue pulling vapor until the tank car vapor gauge reads 3-8 psig, according to outside temperature.

SECURING EQUIPMENT AFTER UNLOADING

After the vapor pressure in the car is pulled down to the correct pressure, shut the compressors off and close the 4 red valves on the compressor inlet as well as the two orange valves between the compressor and the liquid line to the storage tanks. The vapor valve on the unloading boom and the liquid and vapor valve at the tank car are then closed and all lines disconnected being sure that the flanged elbows are not left in the car dome and returned with the car to vendor. Disconnect the grounding clips from the car and turn the placards to read empty. On the way back from the tank farm close monomer valve from the main feed line to the vaporizer.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: TRANSFERRING POLYMERIZED PVC RESIN SLURRY	ASPI 1007
	EFFECTIVE DATE: 4/10/74
	SUPERSEDES: NONE
	PAGE 1 OF 4 PAGES

PURPOSE

To establish a standard procedure for transferring polymerized PVC resin slurry from the reactor in the reactor building to the dryer building blend tank, with a minimum VCM exposure.

RESPONSIBILITY

A joint effort between the dryer room personnel and the reactor building utility men.

CALL-IN PROCEDURE

1. When the reactor batch has been properly recovered and is ready for transfer to a blend tank, the reactor building utility men will contact the dryer room personnel by phone, extension 53, and give him the reactor number, batch number, and resin type.
2. Dryer room personnel will determine to which blend tank the batch will be transferred into. He will inform the reactor building utility man of the blend number, the number slurry line to use, and the blend tank number to which the batch will be dumped.
3. The utility men will record the blend tank number and blend number on the reactor loading sheet in the space provided.
4. The dryer room personnel will record the plant batch number on the blend tank card, which also includes the blend number, blend tank number and resin type.

SET-UP PROCEDURE (REACTOR BUILDING)

1. The utility men will set up the proper valves to the preset slurry transfer line. Particular notifications must be made to check the slurry cross-off line valve and the slurry line drain back valve. Both valves should be closed.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: TRANSFERRING POLYMERIZED PVC RESIN SLURRY	ASPI 1007
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	PAGE 2 OF 4 PAGES

SET-UP PROCEDURE (REACTOR BUILDING)

2. The slurry basket strainer should be emptied of any large reactor chips and reactor scraping into dumpster located outside of building.
3. Install the cover on the slurry basket strainer.
4. Position the slurry basket elephant trunk exhaust over the open portion of the slurry basket cover and open the exhaust duct damper.
5. Check the foot valve position of all empty reactors and shut valves if open.
6. When ready the utility men signal the dryer room with a horn. Signal as follows:

RA - one short
 RB - two short
 RC - three short

SET-UP PROCEDURE (DRYER ROOM)

1. The dryer room operator will hook up the flexible slurry transfer hose from the discharge of the present slurry line to the blend tank that the particular batch will be dumped into.
2. Open slurry line valve and close drain valve.
3. When ready, the dryer room will signal the utility men with the horn signal as described above.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: TRANSFERRING POLYMERIZED PVC RESIN SLURRY	ASPI 1007
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DUMPING PROCEDURE

1. When the signal is received from the dryer room, to proceed to dump, the utility men will open the reactor foot valve, with the dump line valve at the slurry basket closed.
2. The utility man will open the valve above the slurry transfer pump, open the pump seal water valve and start the pump.
3. By regulating the dump line valve, the utility man will proceed to transfer the batch by maintaining a constant level in the slurry basket, paying particular attention not to overflow the slurry basket strainer or interrupt the transfer in any manner that would cause the slurry line to plug. Minimum use of water should be added to the resin slurry during dumping. If a slurry spill occurs, it should be cleaned up immediately by shoveling up product into feed bags, or washing to drain. Feed bags must be removed from reactor building as soon as possible.
4. With assistance from other personnel at the reactor level, when the slurry level reaches the bottom agitator blades, the agitator should be shut off. With the reactor exhaust elephant trunk in the reactor opening, rinse down agitator shaft, baffle, reactor walls and dome with water.
5. When the batch is dumped and the reactor has been rinsed down, the utility man will then pump over water as follows: RA and RB - 5 slurry buckets, RC - 6 slurry buckets of city water from the hose located above the slurry basket to flush the slurry transfer line.
6. The utility man will then signal by RA - one, RB - two, RC - three, signals on the horn that he is finished dumping and flushing.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: TRANSFERRING POLYMERIZED PVC RESIN SLURRY	ASPI 1007
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DUMPING PROCEDURE (continued)

7. The utility men will secure the transfer equipment by closing the valves, and shutting off the pump. He will empty the slurry basket if needed, drain back the slurry transfer line, fill the slurry basket with water, and close the damper in the slurry basket elephant trunk exhaust system.
8. The dryer room operator will acknowledge the completion of the transfer by returning RA - one, RB - two, RC - three signals on the horn.

EMERGENCY SITUATION

1. If problems develop during resin slurry transfer, the signals for trouble will be 5 short signals on the horn common to all reactor buildings. If the utility man hears trouble signal from the dryer room, he will immediately stop transferring, turn on the deionized water line above the pump discharge shut off valve, stop the slurry transfer pump, and shut the valve on the discharge of the pump, and the dump line valve. Both areas will determine the trouble and make corrective action.

:jaa

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: CHARGING POLYMERIZATION REACTOR	ASPI 1007
	EFFECTIVE DATE: 4/10/74
	SUPERSEDES: NONE
	PAGE 1 OF 5 PAGES

PURPOSE

To establish a procedure for reactor charging that will assure uniformity in batch to batch resin properties and maintain a high level of quality. It is expected that the procedures given below will be performed safely so as to protect men from VCM exposure and equipment.

GENERAL

1. Charging reactors is a critical step in the production of high quality PVC resin and demands the close attention of each charge operator.
2. Charge a reactor only if the charge header has been thoroughly rinsed and the reactor itself has been thoroughly rinsed and cleaned.
3. Resin or foreign matter left in a reactor has a major effect on gels.
4. Following charging instructions and formula sheet instructions carefully. This will insure that all operators are charging in the same manner. Changes in charging technique from one operator to another can result in changes in resin properties, therefore, all instructions must be followed correctly.

CHARGING PROCEDURE

1. Prepare the "Reactor Loading Sheet" using the proper formulation sheet in the formula book. There are changes made time to time in formulations, therefore, the formula sheet must be consulted, do not rely on memory.
2. Check the reactor to see that all valves are in the proper position:
 - a. Foot valve closed
 - b. All header valves closed
 - c. Blower off valve to stack closed
 - d. Reactor charge valve open
 - e. Pressure gauge vent valve shut
 - f. Cold water by-pass shut and one hot water block valve closed.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: CHARGING POLYMERIZATION REACTORS	ASPI 1007
	EFFECTIVE DATE: 4/10/74
	SUPERSEDES: NONE
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CHARGING PROCEDURE (continued)

3. Check manhole gasket condition, if there is any doubt about its usability then use a new gasket.
4. Fill the Methocel measuring tank with the proper number of inches of Methocel.
5. Inspect reactor for cleanliness and foreign objects and install reactor exhaust hose in reactor manway with damper open.
6. Place temperature chart on instrument and set pen in recording position.
7. Raise instrument temperature set point to 95°F.
8. Start water charge to reactor. Water temperature should be 90° to 100°F.
9. Begin charging Methocel to the reactor by starting the pump when the water charge is at 10 counts. Be sure all methocel is charged to the reactor.
10. Add all remaining additives including catalyst to the reactor while the water is being charged. Open hot water block valve after the catalyst is charged.
11. Reactor manway cover must remain open and reactor exhaust hose in reactor manway until all the water is loaded. Check water level by measurement from top lip of the bottom flange section of manway to the water level. Measurement should be about 58 inches.
12. Start agitator after all the water has been charged to the reactor if the measurement is correct. Check with foreman if measurement is not about 58 inches.
13. Agitator must remain on until batch is dumped.
14. Remove exhaust hose, shut damper, and tightly secure the hatch. Be careful to center the gasket on the manhole properly.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: CHARGING POLYMERIZATION REACTORS	ASPI 1007
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CHARGING PROCEDURE (continued)

15. Immediately start evacuation if internal temperature is 90°-100°F. Adjust temperature to 90°-100°F if required before evacuating.
16. In RA and RC, evacuate the reactor to the exact mercury level on the Meriam gauge that corresponds to the internal temperature. (Use evacuation - temperature chart). The evacuation valve must be closed at the exact mercury level to avoid making an off-grade batch.

In RB, the evacuation is automatically controlled by a vacuum control instrument. The setpoint of the vacuum control instrument on the West wall must be at 72. The instrument on the East wall does not control vacuum, it only records the vacuum. (See additional comments on evacuation procedure posted near each vacuum recorder).
17. If the evacuation takes less than two (2) minutes or longer than six (6) minutes, do not proceed with charge to avoid making a bad batch. Do the following:
 - a. Close the evacuation valve.
 - b. Bleed air back into the reactor.
 - c. Inform foreman of evacuation problem.
 - d. Correct problem before proceeding.
 - e. Over-evacuation will result in a coarse batch.
18. If evacuation is correct, immediately raise set pointer on instrument to running temperature.
19. Immediately start VC charge.

NOTE:

At the beginning of each shift, in each building, the Meriam gauge must be checked out using a standard gauge for accuracy and proper functioning. In RB, check the set point on the vacuum control instrument. The set point must be at 115 mm Hg.

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REACTOR HEAT-UP

1. Check to determine if the hot water and the c around both control valves at the downstairs open. Make sure the by-passes are closed. that cold water valves are correct. If for a cannot get cooling water, it will continue to h safety disc.
2. Set the temperature control instrument at th erature as called for in the formula. Make has been wound up and that the temperature on automatic.
3. Fifteen minutes after you start the heat up : the reactor itself and the temperature contr sure that:
 - a. The reactor pressure gauge is
 - b. Double check to determine the agitator is running. The hot w automatic valve will not open v off.
 - c. That there are no leaks at the gasket.
 - d. The temperature pen shows a ! that the instrument is on auton
4. Note on the "Reactor Loading Sheet" that by recording as follows on the first line fc
 - a. Under "time", record the tim check was made.
 - b. Using the rest of the spaces a record the following:

"Temp. control OK, Agitator

If this is not the case, take the proper act problems on the sheet.

ASSONET STANDARD PRACTICE INSTRUCTION

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REACTOR HEAT-UP (continued)

- Continue to monitor heat up to prevent over-heating or wrong settings. Check to see that the knife valve on the hot water return works properly. (Hot water should return to the hot water storage tank if the jacket temperature is 125°F, or above. Under these conditions the knife valve should be open).

MONITORING REACTION

- During the run take and record readings hourly, keeping a close watch for abnormal pressure and temperature. All readings must be observed, do not assume readings have not changed.
- By observing reaction time, the reactor pressure drop, and the change in the reactor jacket temperature, anticipate by past experience, when recovery conditions will occur so that the batch will not be "recovered" too late. Batches will be recovered when they reach a certain pressure or pressure drop as called for on the formulation sheet.
- Recover the batch as described in "Monomer Recovery Instructions".
- Follow all special instruction and record all data.

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ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: PREPARATION FOR ENTERING POLYMERIZATION REACTORS	ASPI 1003			
	EFFECTIVE DATE: April 10, 1974			
	SUPERSEDES: None			
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Before anyone enters the polymerization reactors the following procedures must be followed:

1. Insert safety pin in stop button, lock out agitator using the disconnect switch at reactor. Have the charge valve and dump valve shut. The man entering the reactor will have the key with him at all times.
2. Have hot water valves off and cooling water by-pass cracked open to keep jacket cool. Shut dump valve at this time.
3. Open the valve to the vent stack (with steam valve closed). Open the main charge valve, (some water from the stack will probably drain out). After the water has drained out close only the main charge valve and leave the vent stack valve open. Whenever a man is in a reactor these valves must be in this position unless the man must have the charges valve open in the act of cleaning it. To review - all header valves must be shut, main charge valve must be shut, vent stack valve must be open, dump valve must be shut, when a man is in a reactor. This will eliminate the possibility of VC pressure building up in the header and is a double block and bleed.
4. Turn on elephant trunk vent system and check to make sure that it is sucking air. Also make sure any other elephant trunk vent system dampers that are not in use are in the closed position.
5. Place elephant trunk vent system in the reactor so that it extends to the bottom of the reactor (VC vapors are heavier than air and will settle to the bottom of the reactor).
6. Reactor must ventilate for a minimum of 20 minutes with a large 8" elephant trunk inserted to the bottom of the reactor. Remove 8" elephant trunk after 20 minutes minimum then attach 3" flexible hose with adaptor to 3" elephant trunk and insert to bottom of reactor.
7. Put on safety harness before entering a reactor. Harness must be worn.
8. Make sure another man is in the building, either the lead operator or an operator and that he knows someone will be in the reactor. This man must stay on the second floor and must check on the person in the reactor at least once every 5 minutes.

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ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: PREPARATION FOR ENTERING POLYMERIZATION REACTORS	ASPI 1003		
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	SUPERSEDES: None		
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9. Before entering a reactor, double check that all header valves are shut, the main charge valve must be shut, the stack vent valve must be open.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: REACTOR CLEANING (MANUAL)	ASPI 1006
	EFFECTIVE DATE: 4/10/74
	SUPERSEDES: NONE
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PURPOSE

To establish a procedure for manual cleaning of reactors with a minimum VCM exposure.

PREPARATION

1. Before entering a reactor, following standard procedures as noted in "ASPI " Preparation for Entering Polymerization Reactors.
2. Attach a 3 inch flexible hose with adapter to 8 inch reactor elephant exhaust hose with damper open and insert to the bottom of the reactor.

PROCEDURE

1. Reactor should be entered for cleaning after each reactor run.
2. Check safety disk. It should be replaced if buildup covers more than one-half of the disk.
3. Pressure gauge nozzle should be clear of any buildup. Pressure gauge pipe nipple should be clear, if not, remove pressure gauge assembly and rod with 1/2" rod.
4. Baffle and dump nozzles should be cleared of any buildup.
5. Charge valve nozzle should be clear of any buildup.
6. Agitator shaft and nuts should be cleared of any buildup.
7. Baffle tips should be cleared of any buildup to provide good temperature control.
8. Face of manhole and reactor covers should be cleaned of any buildup.
9. Dump valve nozzle should be clear of any buildup.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: REACTOR CLEANING (MANUAL)	ASPI 1006
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PROCEDURE (continued)

10. If the foot valve must be open for cleaning, all foot valves on the same line must be closed and slurry basket valve must be open.
11. After cleaning, remove all refuse to dumpster immediately, open foot valve and thoroughly and completely rinse down reactor.

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ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: REACTOR BUILDING HOUSEKEEPING STANDARDS	ASPI 1005
	EFFECTIVE DATE: 4/10/74
	SUPERSEDES: NONE
	PAGE 1 OF 2 PAGES

PURPOSE

To establish standards and define the policy of reactor building cleanliness and to assist all employees in recognizing and controlling health and safety hazards.

SCOPE

The standard set forth in this instruction are minimum. They do not relieve management at any level of their responsibility for setting higher standards if such are required or desired.

RESPONSIBILITY

All employees are charged with the responsibility for providing full compliance to the standards. Also, all members of management are charged with the responsibility to provide full support and compliance to the provisions of this policy.

DEFINITIONS

1. The lead operator will keep the stairway clean.
2. The operators will keep the second floor clean. This area will be washed down with water during the last half hour of each shift.
3. The utility man will keep the first floor and the dumpster area clean. The first floor will be washed down with water during the last half hour of each shift. Area around the dumpster will be picked up of all refuse prior to the end of each shift.
4. Cleanliness of the raw material weighing area is the responsibility of the operators.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: REACTOR BUILDING HOUSEKEEPING STANDARDS	ASPI 1005
	EFFECTIVE DATE: 4/10/74
	SUPERSEDES: NONE
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DEFINITIONS (continued)

5. All reactor scrapping will be emptied into the dumpster immediately upon removal from the reactor.
6. All slurry basket strainers will be emptied into a container then emptied into the dumpster immediately upon removal from the slurry basket.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: HOUSEKEEPING STANDARDS	ASPI	1001
	EFFECTIVE DATE: March 29, 1974	
	SUPERCEDES: None	
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PURPOSE

To establish specific minimum conditions of cleanliness and order in all departments and areas of the Assonet Plant, individually and collectively. To make these standards known and to fix specific responsibilities for adherence to them.

SCOPE

The schedules and conditions as set forth in the standards are, as stated in the purpose, minimum. They do not relieve management at any level of their responsibility for setting higher standards if such are required or desired.

RESPONSIBILITY

Specific responsibilities shall be fixed in the standards for given locations, conditions, and or circumstances. Generally, all members of management are charged with the responsibility for providing full support to, and for participation in, any and all such activity as is required to meet and adhere to the standards set.

DEFINITIONS

1. Housekeeping - The term "housekeeping" refers to the normal routine duties and procedures that are required on a day-to-day basis to keep a plant, an area, a department, or a piece of equipment in a neat, orderly safe and presentable condition.

Basically, "housekeeping" is a function of the people who "live" in a specific area and take pride in keeping their "house" in order.

2. Housekeeping Standards - A housekeeping standard is a written statement of the minimum conditions that are acceptable in a specific department or location at any given time.

A housekeeping standard shall be developed by the people involved, and thus it becomes a commitment to maintain the conditions stated. Each standard must be specific and the responsibility for its successful accomplishment, in its parts and as a whole, must be fixed.

The various housekeeping standards in combination make up a standard for the Assonet Plant.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: HOUSEKEEPING STANDARDS	ASPI	1001
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DEFINITIONS (CONTINUED)

3. Housekeeping Program - A formal program supported by written departmental standards. It is an integral part of the overall safety program, based on the premise that a clean plant is a safe and well run plant.
4. Developing Housekeeping Standards - Each department superintendent shall have the responsibility for the development, implementation, and administration of the housekeeping program in his department. As a general rule, he will delegate the development of the standards to others in his department, but the basic responsibility for determining that the standards are realistic and workable must be retained by him. The final product will actually become a personal commitment that the standards will be met and maintained.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: UNLOADING VINYL CHLORIDE	ASPI
	EFFECTIVE DATE:
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PURPOSE

To assist all employees in recognizing and controlling health and safety hazards. To establish and define the policy covering the unloading of vinyl chloride from railroad tank cars.

SCOPE

The regulations included in this Standard Practice Instruction are intended as mandatory minimums of protection and Supervision is not relieved of the responsibility of setting higher standards as may be required within the area of their jurisdiction.

RESPONSIBILITY

Generally, all members of management are charged with the responsibility to provide full support to provisions of this policy.

GENERAL

Vinyl Chloride Monomer is generally received in 20,000 or 30,000 gallon tank cars and placed by the railroad at our 6 unloading stations. Each car being hooked up will be grounded by attaching the grounding clips at each station to each car. Each car will be hooked up by the unloader to the unloading boom by attaching the 1-1/2" stainless steel flex hose to the vapor valve at the tank car dome as well as the two stainless steel liquid unloading hoses to the 2" valves. This is accomplished by removing the plugs from the three valves at the dome of the car and installing the flanged elbows to the reducing bushing at each valve. Each respective flex hose is then attached to the flanged elbows being sure that the gasket is in the correct position before tightening the connection. Monomer, from the storage tanks converted to vapor, is then used to pressure the liquid out of the car to the storage tanks. After the liquid transfer, the vapor in the car is reduced to 3-8 psig (depending on the outside temperature). This is done by pulling down the pressure in the car by using the compressors to pull the vapor from the car, compress it, and send it to the storage tank.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: UNLOADING VINYL CHLORIDE	ASPI
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GENERAL (continued)

After unloading, the unloader will disconnect the car from the unloading boom and turn the tank car placards to show empty. After making sure that the catwalks at the unloading station are clear of the car, the car is ready to be pulled by the railroad.

UNLOADING LIQUID MONOMER

Monomer from the storage tanks is fed to the vaporizer by opening the valve on the main feed line and both valves at the vaporizer automatic control valve. The two 2" valves on the discharge line of the vaporizer are then opened to feed vapor to the unloading racks. The 1-1/2" vapor valve on the unloading rack and the vapor valve on the car are then opened. Open both liquid valves on the tank car, slowly to avoid popping the excess flow valve, and check for leaks at all connections,* then open the liquid valve on the boom. Back in the compressor building, open both block valves at each of the four site glasses on the liquid lines to the storage tanks. Open the steam feed valve to the vaporizer located on East wall in compressor room. Maintain a level in the vaporizer site glass by controlling the monomer feed to the vaporizer with the by-pass valve. The level in the site glass should be at a point 3/4 of the way up. Check for monomer flow at the site glass on the monomer line to the storage tanks and after flow is evident open the by-pass valve and close the two site glass valves. Recheck the level at the vaporizer to see if it is holding.

Continue unloading with periodic checking for leaks at the tank car and for the end of the liquid flow at the site glasses. Experience will show what unloading times can be expected and when checks should be made. The unloader will check at least once during the liquid unloading for any leaks at the car and correct same. Evidence of the approach of the end of the liquid flow is when the level in the site glass drops as well as bubbles will appear in the site glass.

ASSONET STANDARD PRACTICE INSTRUCTION

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VAPOR PULL DOWN

Bubbles showing in the flow site glass are an indication that the liquid is nearly unloaded from the car. Unloading will continue until vapor only is flowing through the site glass and the flapper vibrates about 1/2 inch. After liquid is out of the car shut off the monomer flow to the vaporizer by closing both valves at vaporizer inlet. Close both valves on the discharge side of vaporizer as well as the steam flow to the vaporizer. Shut off the liquid lines by closing all site glass valves and site glass by-pass valves.

To set up for vapor pulling open the 4 red valves on the line from the compressors to the vapor line to the cars (the 4 valves are colored red). Open the two orange valves, depending which tanks the vapor is to go to on the discharge line of the compressor to the liquid line to the storage tanks. Start the compressors. After starting compressors, shut the liquid valves on the unloading boom. Then recheck to compressor to see if they could be liquid-locked. If so, stop the locked compressor and close the corresponding red valve on the compressor inlet line and open the two valves on the compressor bleed off line which will allow liquid and some vapor to be forced from the compressor to the atmosphere. After all the liquid is forced from the compressor and the check valve re-seats itself, close the two bleed-off valves and open the corresponding compressor inlet valve. Start the compressor to pull the vapor, once the inlet to the compressor becomes hot, it is safe to assume that the compressor will not shut down. After following the vapor pull down on the tank car vapor line gauges, experience will show what pull down times can be expected and how often checks should be made. Continue pulling vapor until the tank car vapor gauge reads 3-8 psig, according to outside temperature.

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SECURING EQUIPMENT AFTER UNLOADING

After the vapor pressure in the car is pulled down to the correct pressure, shut the compressors off and close the 4 red valves on the compressor inlet as well as the two orange valves between the compressor and the liquid line to the storage tanks. The vapor valve on the unloading boom and the liquid and vapor valve at the tank car are then closed and all lines disconnected being sure that the flanged elbows are not left in the car dome and returned with the car to vendor. Disconnect the grounding clips from the car and turn the placards to read empty. On the way back from the tank farm close the monomer valve from the main feed line to the vaporizer.

* Leaks at connections can be detected by the appearance of snow-like material at major leaks or by the use of soap bubbles at small leaks.

ASSONET STANDARD PRACTICE INSTRUCTION

SUBJECT: ENTERING POLYMERIZATION KETTLES	ASPI
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Before anyone enters the polymerization kettles the following procedures must be followed:

1. Lock out agitator using the disconnect switch. Have the charge valve and dump valve shut.
2. Have hot water valves off and cooling water by-pass cracked open to keep jacket cool. Shut dump valve at this time.
3. Open the valve to the vent stack (with steam valve closed). Open the main charge valve, (some water from the stack will probably drain out). After the water has drained out close only the main charge valve and leave the vent stack valve open. Whenever a man is in a reactor these valves must be in this position unless the man must have the charge valve open in the act of cleaning it. To review - all header valves must be shut, main charge valve must be shut, vent stack valve must be open, dump valve must be shut, when a man is in a kettle. This will eliminate the possibility of VC pressure building up in the header and is a double block and bleed.
4. Turn on elephant trunk vent system and check to make sure that it is sucking air. Also make sure any other elephant trunk vent system dampers that are not in use are in the closed position.
5. Place elephant trunk vent system in the kettle so that it extends to the bottom of the kettle (VC vapors are heavier than air and will settle to the bottom of the kettle).
6. Let kettle ventilate for 20 minutes or below 50 ppm by analyzer.
7. Put on either a pair of wrist straps or a harness before entering a kettle. Either one or the other must be worn.
8. Make sure another man in the building, either the lead operator or an operator, knows you are in the kettle. This man should stay on the second floor so that he can check on you at least once every 5 minutes.

The first part of the paper discusses the importance of the study and the objectives of the research. It highlights the need for a comprehensive understanding of the subject matter and the role of the researcher in this process. The second part of the paper presents the methodology used in the study, including the data collection methods and the analysis techniques. The third part of the paper discusses the results of the study and the conclusions drawn from the findings. The final part of the paper provides a summary of the key points and offers suggestions for future research.

The study was conducted in a systematic and rigorous manner, following the principles of scientific research. The data was collected from a large sample of participants, ensuring the representativeness of the findings. The analysis was performed using advanced statistical techniques, allowing for a detailed examination of the data. The results of the study are presented in a clear and concise manner, highlighting the key findings and their implications. The conclusions drawn from the study are based on a thorough analysis of the data and are supported by the findings. The paper concludes with a summary of the key points and offers suggestions for future research, highlighting the need for further exploration of the subject matter.

The study has several strengths, including the use of a large sample size and the application of advanced statistical techniques. However, there are also some limitations to the study, such as the potential for bias in the data collection process. Despite these limitations, the study provides valuable insights into the subject matter and contributes to the existing body of knowledge. The findings of the study are discussed in detail in the following sections, highlighting the key points and their implications.

The study was conducted in a systematic and rigorous manner, following the principles of scientific research. The data was collected from a large sample of participants, ensuring the representativeness of the findings. The analysis was performed using advanced statistical techniques, allowing for a detailed examination of the data. The results of the study are presented in a clear and concise manner, highlighting the key findings and their implications. The conclusions drawn from the study are based on a thorough analysis of the data and are supported by the findings. The paper concludes with a summary of the key points and offers suggestions for future research, highlighting the need for further exploration of the subject matter.

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9. Before entering a kettle, double check all header valves are shut, the main charge valve must be shut, the stack vent valve must be open, and the dump valve must be closed.
10. Check for VC vapors when entering the kettle. If VC vapors are over 50 ppm then or at any other time you should get out of the kettle immediately and recheck steps 1, 2, 3, 4, 5, and 6 before re-entering.
11. When a man is in a kettle he should have the vent tube in the kettle.
12. The charge valve and dump valve should only be open during the time they are actually being cleaned.
13. Additional procedures will be forthcoming on protective measures on VCM exposure.