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REPACKAGING HALTHANE 73-18

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MANUFACTURING ENGINEERING DIVISION

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MASTER

*Process Development*  
*Endeavor No. 101*



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## REPACKAGING HALTHANE 73-18

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#### INTRODUCTION

Like other urethanes, the Halthanes are susceptible to reaction with moisture. The curing agents absorb atmospheric moisture and form inferior products when later reacted with the prepolymer; thus, precautions must be taken to protect all components from moisture prior to use.

This endeavor was undertaken to determine if Halthane 73-18 adhesive could be repackaged into preweighed kits for WR usage without utilizing an inert atmosphere (1% RH or less) during the repackaging operation.

#### BASIC CONCEPT

This technique employs an oven to dry all cleaned containers (polypropylene cups and syringes), a desiccating cabinet held at 10% or less RH for their storage, and a pump/load cell arrangement for dispensing and weighing the material.

#### PROCESS

The bulk material to be repackaged is siphoned from the manufacturer's (BX) original container by use of a tubing

pump utilizing silicone tubing as the only contact surface. The air space above the material in the bulk container is supplied with dry air received from a drying column connected to the vendor's container by means of silicone tubing.

An air-actuated pinch valve is used to shut off the material flow. The silicone tubing passes through the pinch valve and as the desired quantity of material is dispensed, a four-way solenoid valve energizes a piston which squeezes the tubing shut and simultaneously shuts down the pump.

The actual weighing device is a load cell coupled to a pre-set digital readout. Using a constant rpm on the feed pump, a constant flow column (volume) of fluid is established and the weight thereof is controlled by means of a "fall adjust" potentiometer on the console. Material dispensing can be controlled within 1% of desired weight.

Exposure time to ambient conditions is minimized by use of desiccating cabinets held below 10% RH. Each cup or syringe to be filled is removed from the cabinet, filled, and returned to the cabinet.

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The lids on the cups are pulled down tightly on the cup by blister sealing a plastic film on the cup/lid assembly. This precaution prevents cup leakage during transportation and/or handling.

One cup of Comp. R, one syringe of Comp. T, two 5 gram ampules of desiccant, and an indicator card are then placed into vapor barrier bag consisting of an outside polyester film, a central layer of aluminum, and an inner polyethylene film. The polyethylene film serves as a heat sealing surface. Material stored in this type of package has shown the interior of the plastic bag to contain less than 10% relative humidity after three months' exposure to ambient conditions. Table I contains all analytical test data concerning

the endeavor. Data beyond the three month test do not presently exist.

Photographs of the equipment arrangement and the repackaged kit (shown in Fig. 1 and 2, respectively) are included to assist in understanding the process.

The delay in the completion of this project was caused by the cups and syringes being insufficiently dry. Analytical data showed the water content to exceed the specification limits by approximately 0.02% and the NCO content of the resin to be about 0.05% below specification limits. Preliminary tests conducted on material repackaged in drier containers show these material analyses to be well within the specification limits.

Table I. Analytical Data

Halthane 73-18  
(Comp. T = 73 Prepolymer, Comp. R = 18 Curing Agent)

| Comp. T             | Total NCO (%) | Viscosity (PaS) | Free MDI (%) |
|---------------------|---------------|-----------------|--------------|
| Specification       | 10.25 ± 0.40  | 9 ± 3           | 22 ± 2       |
| "As Received" Bulk  | 10.39         | 8.8             | 21.7         |
| Repackaged 3 Months | 10.40         | 8.7             | 22.5         |

| Comp. R             | Acid No. | Hydroxyl No. | Water Content (%) |
|---------------------|----------|--------------|-------------------|
| Specification       | 0.15*    | 250 ± 10     | 0.08*             |
| "As Received" Bulk  | 0.021    | 243          | 0.045             |
| Repackaged 3 Months | 0.043    | 255.5        | 0.050**           |

\*Maximum

\*\*The material was repackaged in an environment of approximately 20% RH, temperature 23.3 C.

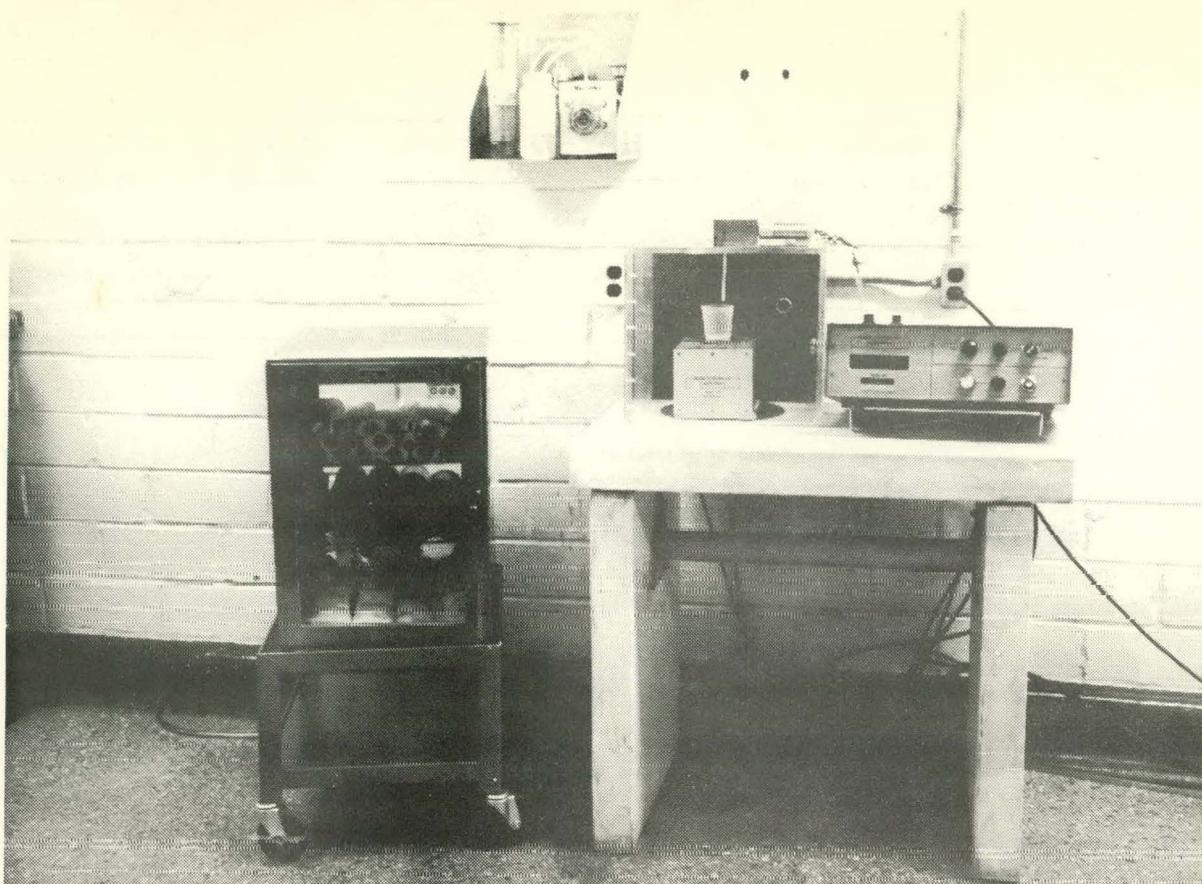


Fig. 1. Dispensing Equipment

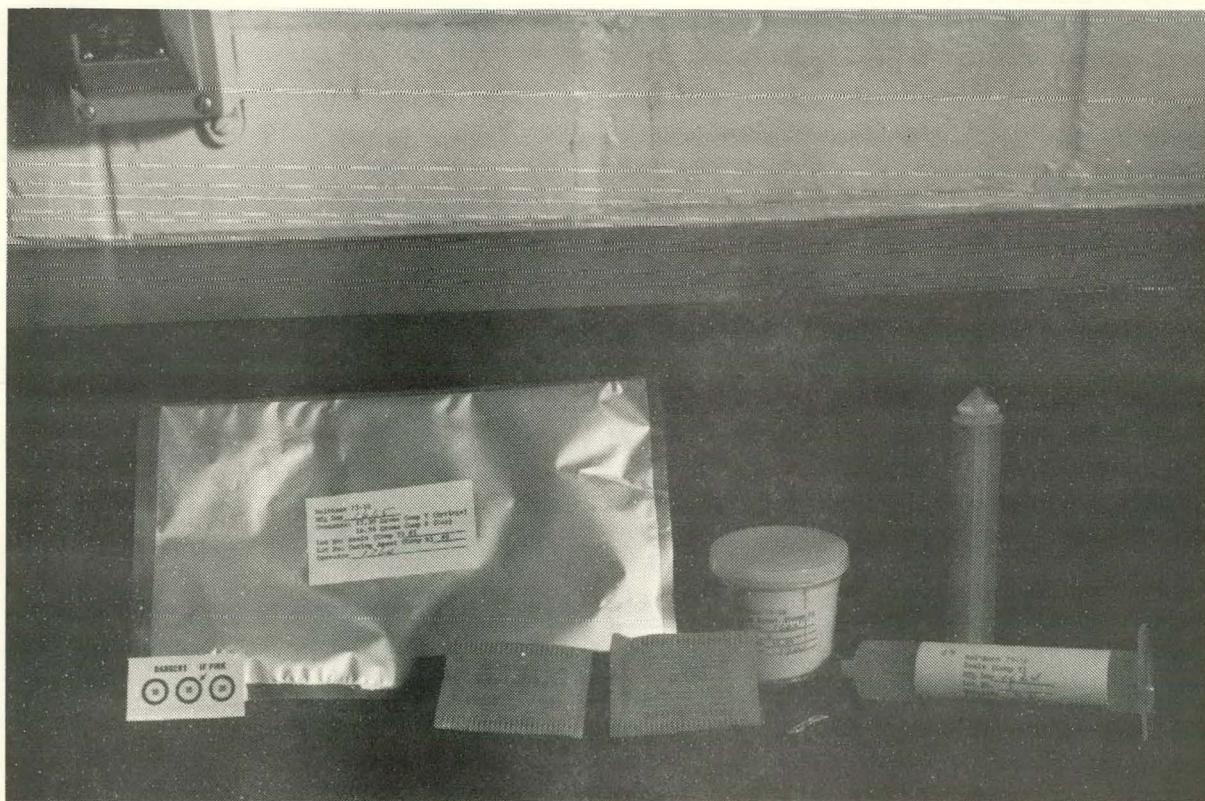


Fig. 2. Repackaged Kit

## CONCLUSION

It is concluded that repackaging of the bulk material can be accomplished without resorting to a "glove-box" type operation.

## FUTURE WORK

Relative humidity in the repacking area is not controlled. It is recognized that the 20% relative

humidity atmosphere in which this experiment was conducted is not the most severe atmospheric condition that will be encountered. Therefore, an attempt to repackage material, in which the relative humidity is maintained at approximately 50 to 60%, without significantly increasing the moisture content of the curing agent will be conducted. The final repackaging technique developed will also be used for the prepolymer.