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ANALYTICAL & PHYSICAL MEASUREMENTS SECTION
POLYMER PRODUCTS DEPARTMENT

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ANALYTICAL REPORT   February 23, 1990

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[signature: Harvey S. Gold]

DETERMINATION OF CHLORINE PERMEATION THROUGH TEST MEMBRANES
(Job No. 892-1076, 901-285;
LIMS Nos. 89066200 thru 89066202, 90069544, 900695445, 90071027)

Per your request, we initiated studies in mid-November which were designed to determine the relative rates of permeation of chlorine gas through "Sclar" (a "standard" polyethylene) and samples of polyethylene modified by AT&T Bell Labs to contain oxidation inhibitors. Subsequently, samples of native and modified (coated) "Mylar" film were added to the study.

This study was initiated as part of a joint effort involving Dupont and AT&T Bell Laboratories. The initial analytical protocol was developed with suggestions made by John Franey (AT&T Bell Laboratories) and was designed to yield results within the three-week time period designated for the stage-I investigation of the modified polyethylene material.

As you are aware, many iterations of the experimental set-up were required. At the time this study was initiated, none of the required equipment was on hand, nor had the Analytical Section ever made this type of measurement. Within the course of days, we identified the key pieces of required equipment: 1) a Mast Development Oxidant Monitor was lent to us by John Franey and rehabilitated, 2) a Vici Metronics gas permeation generator was purchased and delivered within 4 days, set-up and calibrated for operation, and 3) the "clam-shell" film holders were ordered, received, and modified on a priority basis. The system was completely plumbed, and intial check-out began. Over the course of days and weeks, numerous minor and major experimental modifications were made with the advice and (sometimes) the concurrence of
John Franey.

Attached is a block diagram of the final working system, both with and without a second "clam-shell" in place. Since the many earlier variants did not work properly, they are not documented in this report. As implemented, the apparatus (refer to Figure 1) exposes one side of the test film to

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Operating Directions for Permeability Test Apparatus

Plumb the Vici Metronics Model 340 "Dynacalibrator" gas permeation generator, the clam shell test chamber, and the oxidant monitor as per the attached diagrams and these instructions. Refer to the respective manufacturers' instruction manuals for the Metronics Dynacalibrator Model 340 and the Mast Oxidant Monitor Model 1724 to become familiar with the theory and operation of each of these devices.

In order to obtain greater sensitivity using the Mast Oxidant Monitor the auxiliary sample pump should be disabled and the input gas stream connected directly to the precision air sample pump.

Insert a film in the clam shell and adjust gas flows as follows.

1. Set regulator and gauge of tank A at 10 psi and a flow rate of 50-100 ml/min.

2. Position the dip tube of the A overflow (H1) to 13 inches of water and adjust needle valve A so that the exit chlorine carrier just bubbles in the NaOH scrubber solution.

3. Set H2 equal to H1, and adjust needle valve B so that the H2 tube just bubbles.

4. Adjust needle valve D for equal pressure at both entrances as indicated by the "Magnahelic" gauge (center zero type).

5. Set H3 to be equal to 90% of H1, and adjust needle valve C so there is a positive flow (5-10 ml/min) at the exit of the chlorine monitor.

Note: Interplay exist between valves B, C, and D, so iterative readjustment will be necessary to achieve the conditions stipulated above.

At the time of initial start up, as much as one day may be required to obtain equilibrium (due to adsorption of chlorine on the wall surfaces throughout the system) after plumbing is complete and permeation tubes have been installed. For the experiments described in this report, two chlorine permeation tubes were utilized simultaneously; each tube had a permeation rate of 2100 ng/min @30°C for 1 cm length.

When the second design is utilized, verification of continued proper operation of the system can be obtained at any time by switching to a test film (typically "Sclar") in the second clam shell. Once all valves and flow rates are properly adjusted, the first sample of "Sclar" should fail within 60 minutes, with subsequent failures occurring within 20 minutes.

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Attachments:
    Figure 1, Figure 2, Photographs of apparatus

Keywords:
    Permeability, polyethylene, "Mylar", chlorine

chlorine at 640 ppm, while sweeping the other side with dry, oxygen-free nitrogen. This stream of nitrogen is introduced directly into the detector of the Mast Oxidant monitor. Detailed operating directions are at the end of this Analytical Report.

The results obtained are documented in the table which appears below. Exposure of the first sample (ERD12404-9; LIMS 89066201) was terminated at 9:30 a.m. on Friday, December 8, 1989 so that results could be communicated to you for a preliminary business decision that day. A duplicate sample was then evaluated so that reproducibility could be determined after 77 hours exposure, which corresponded to 5 years of simulated exposure at the TLV of 1 ppm chlorine) on December 11, 1989. An apparent failure occurred at 173 hours.

To place the exposure time in proper perspective, it is necessary to calculate the exposure in terms of ppm-hours, and to then convert this to simulated exposures at 0.01 (a reasonable value for workplace chlorine level) and at 1.0 ppm (the TLV for chlorine, deemed to be far beyond the level which could be expected for constant exposure of man or material). Simulated exposures expressed in these units appear below.

TABLE I. OBSERVED PERMEABILITY BEHAVIOR OF VARIOUS FILM SAMPLES

LIMS #	Material	Actual Breakthrough or time to test end	PPM hours (at 640 ppm)	Simulated Exposure at Breakthrough 0.01 ppm	Simulated Exposure at Breakthrough 1.0 ppm
89066200	"Sclar"	16.6 min (average of multiple trials)	177	2.02 years	0.02 yrs = 7.4 days
89066201	Modified polyethylene ERD12404-9	41 hours*	26,240	299.5 years	2.995 years
		173 hours**	110,720	1,264 years	12.64 years
		190 hours***	121,600	1,388 years	13.88 years
89066202	Modified polyethylene ERD12404-9F	473 hours*	302,720	3,455 years	34.55 years
90069545	Modified "Mylar" ERD12404-22I	393 hours*	251,520	2,871 years	28.71 years

INTERCEPT (handwritten marginal notes beside rows 89066201, 89066202, 90069545)

*   test terminated at indicated hours total elapsed time
**   apparent indication of failure
*** chlorine generator failure at approximately 190 hours; no apparent failure of film at this exposure periode