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The North American Geosynthetics Society
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under the auspices of
The International Geotextile Society
(IGS)

GEOSYNTHETICS '93 CONFERENCE
VANCOUVER, BRITISH COLUMBIA

March 30-April 1, 1993

Vancouver Trade & Convention Centre

Waterfront Centre Hotel

Vancouver,
British Columbia,
Canada

GEO'93

CONFERENCE PROCEEDINGS

VOLUME 3

Geomembranes for the Containment of Sulphur: A Case History

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ABSTRACT

This paper presents a case history which examines the feasibility of several lining materials for the application of sulfur storage. The Shell Caroline Gas Development produces 4,100 metric tons of sulfur daily. A number of site linings at the project's sulfur handling facility were proposed, this case history deals with the liners proposed for the sulfur storage blocks.

Sulfur block liners may be exposed to 125°C molten sulfur. A material selection testing program explored the suitability of four geomembrane materials. Measurements were taken before and after sulfur exposure to measure material shrinkage and tensile properties. A second large scale test was carried out at the construction site where 3 m x 3 m samples were exposed to a 50 mm thick layer of molten sulfur. The large scale test samples were evaluated for tensile properties and shrinkage. A material recommendation was made based on the test results.

INTRODUCTION

The Shell Caroline Gas Development is the most significant sour gas development in the last twenty years. The development of the estimated 56 billion cubic meters of raw gas in this field is being undertaken by Shell Canada Limited along with their partners. The estimated production from this field is to be 2,550,000 cubic meters of natural gas (methane) per day, along with 4,100 metric tons of liquid sulfur and 7,200 cubic meters of natural gas liquids and condensates. In order to handle the sulfur produced by this development, the Shantz sulfur handling facility was built near Harmattan, about 80 kilometers north west of Calgary, Alberta. Molten sulfur is extracted from natural gas at the Shell gas plant in Caroline and then transferred by pipeline 41 kilometers to the Shantz sulfur facility. At the Shantz site molten sulfur is formed into pellets and transferred to rail cars for shipment.

In the event of a malfunction of the pellet manufacturing equipment, or in the event of an overabundance of sulfur on the world market, the sulfur produced by the Caroline gas field may need to be stored. A common storage technique used in Alberta is to pour liquid molten sulfur into an aluminum form and allow it to solidify into a block. Additional layers are placed on top of the previous layers by repositioning the forms. The yellow blocks of solid sulfur that are formed can reach twenty meters in height and are typically the size of a football field. Most Alberta sulfur blocks have been remelted and sold in recent years due to a strong demand for sulfur.

Decommissioning of sulfur blocks in the province of Alberta has revealed costly contamination problems. During sulfur re-melt, the sulfur melting equipment can recover all but the last 100 mm of sulfur from a well prepared block base. The remaining sulfur is chipped out which leaves a large quantity of sulfur contaminated with soil. Efforts to separate the soil from the sulfur have not yet proven successful. This results in quantities of contaminated sulfur which are not saleable and substantial quantities of contaminated soil that must be treated as hazardous waste. The ground below the sulfur block can also contain low pH contamination and remediation of between 150 mm to 300 mm beneath the base is common.

Alberta Environment, a department of the provincial Government of Alberta, required from Shell Canada a plan to prevent the migration of contaminants from the site. It was clearly stated that the long range goal was to restore the Shantz site to farm land at the completion of the life cycle of the facility. The Shell plan included a series of monitors around the site to detect the presence of H_2S gas, sulfur dust, the accumulation of sulfates in the surrounding area, and groundwater contaminants. All site run-off water was to be collected in a double lined retention pond, neutralized, and recycled. This water would be utilized in the cooling towers which would effectively eliminate the release of liquid run-off water from the site. In an effort to prevent soil contamination beneath the sulfur block base it was proposed that a geomembrane liner be placed at the interface of the soil and sulfur which would also result in reduced decommissioning clean-up cost. This paper describes the research that led to the construction of a geomembrane liner beneath the sulfur block facilities at the Shantz site.

SCOPE OF PROJECT

The sulfur block storage area is designed to store five months of production from the Shantz sulfur plant. Two sulfur block pads are built on weathered clay. Each of the two sulfur blocks is designed to be 35 meters wide and 324 meters long with a height of 15 meters. Alberta Environment proposed that a geomembrane liner be used under each sulfur block to prevent contamination of underlying soils.

The principle requirement of the sulfur storage block lining material is that it must withstand the molten sulfur pouring temperature of approximately 125°C. Each pour of sulfur is about 100 mm deep and takes about sixteen hours to cool sufficiently before the next pour is placed. It was felt that the insulating properties of the sulfur would protect the liner after the first 150 to 200 mm of solid sulfur was in place. The proposed liner might also have to remain exposed to the elements for many years before use as pouring a sulfur block at the Shantz site was considered a remote possibility under the current market conditions.

Since operating temperatures of 125°C are well beyond the temperature range of common geomembrane plastics (Modern Plastics Encyclopedia' 92) it was suggested that specific material selection testing be done.

There are a number of factors that come into play in the application of a containment liner. Material selection is a function of chemical resistance, heat resistance, weathering resistance (for an exposed liner), and mechanical properties. There were other factors that needed to be considered as well including: cost, installation requirements, cold temperature performance, and conformability to subgrade. A two part testing program was proposed that would attempt to determine a suitable lining material for the containment of molten sulfur. This two part testing program consisted of a small scale test to determine basic suitability of a number of materials, and a large scale test that would confirm the results of the small scale test under actual field conditions.

SMALL SCALE TESTING

The first step was to make a preliminary selection of a lining material that would meet the basic performance requirements of this project. The scope of this first phase was to canvass suppliers for likely materials and then perform some initial selection tests. The initial problems expected were difficulties with the heat of the sulfur and the possibility of chemical reactions with the lining materials and molten sulfur at elevated temperatures. Initial material selections were based on material cost and availability. A secondary selection was made from materials that had proven high temperature applications. Table 1 lists the materials selected for the small scale test.

Table 1. Materials for Small Scale Test.

1. High density polyethylene (HDPE) 80 mil, blown film.
2. Polyvinyl chloride (PVC) 20 mil, regular containment grade.
3. Ethylene interpolymer alloy material (EIA) "Solar" grade.
4. Silicone Coated Fibreglass (SCF) cloth.

The HDPE and PVC materials are standard containment materials that are widely available and low in cost. The EIA (Solar) material is a high temperature version of the standard ethylene interpolymer alloy style material. A 540 g/m^2 (16 oz/yd^2) silicone coated fibreglass cloth with a service temperature in excess of 260°C was selected to test the high end of the temperature spectrum.

The small scale test was a simple contact test between the molten sulfur and the lining material in question. After exposure, the material was tested for property changes and evaluated as to performance. Peak temperatures were measured under the liner to ensure that the liner was not damaged by sulfur pouring temperatures. Liner samples exposed to the molten sulfur were cut into rectangles approximately 216 mm by 260 mm. Reference marks were placed at equal distances along the side of each sample, three along the length and two along the width. Measurements were taken between opposite marks, before and after exposure to the molten sulfur to determine shrinkage. A suitably sized metal tray was filled with sand and a thermistor was placed in the center of the tray on top of the sand. The sample was centered over the thermistor. A metal container approximately 150mm x 150mm x 150mm was centered over the sample. Sand was placed around the outside of the metal container to prevent the sulfur from leaking out between the metal container and the sample.

Sulfur chips were heated to 150°C in a laboratory sulfur melting pot. The molten sulfur was then ladled into the metal container. The molten sulfur level was brought to approximately the top of the metal container. After substantial cooling of the sulfur, the samples were removed and examined for shrinkage/elongation, discoloration, warping, and bonding to the sulfur. Two tensile test samples were cut from where the material was in contact with the molten sulfur, and two control samples were cut from material outside the contact area. Samples from the control and exposed area were tensile tested according to ASTM D882. The tensile properties of the control samples were compared to the tensile properties of the sample that had been in contact with the molten sulfur.

<u>Small Scale Test: Results</u> Visual observations were made of each material after contact with the molten sulfur. The observations are summarized in Table 2.

<u>Small Scale Test: Discussion</u>. The purpose of the small scale testing was to determine if it was physically possible to utilize a geomembrane in contact with molten sulfur. Original expectations were that the EIA (Solar) and the silicone would show better performance than the HDPE and the PVC. The tensile strength trends in Table 3 show that the PVC and the HDPE exhibited performance equal to, or better than the EIA (Solar). The results from the silicone coated fibreglass cloth showed that strength lost after exposure was much higher than was expected in a high temperature fabric.

After these initial test results a cost estimate was prepared which clearly favoured the HDPE and the PVC. This cost comparison assisted us in limiting our initial material selection to HDPE and PVC. Table 4 shows the cost estimates.

Table 2. Small Scale Test
Visual Observations.

Material	Observations
HDPE	Showed warping during placement of the sulfur. In the initial test the warping of the HDPE lifted the metal form away from the sample and allowed the sulfur to spill out from the test area. A second test was performed with a substantial weight on the metal form to prevent lifting. The warping observed was permanent. The HDPE showed a slight surface discoloration; the contact area was lighter in color after the test and the color could not be removed. No significant shrinkage was observed.
PVC	Showed some warpage from the heat. An outline of the metal form was clearly set into the PVC sample after the test. The sample showed discoloration in the contact area but did not show measurable shrinkage. The PVC sample showed some bonding to the solid sulfur.
EIA (S)	Showed some warping from the heat. No discoloration or shrinkage was apparent in this sample. Some bonding of the sulfur to the sample was present.
SCF	Showed no warping, no discoloration, or shrinkage. Some bonding to the sulfur was present but the sample separated cleanly.

Table 3. Small Scale Test.
Tensile Strength Trends.

Material Type	Average Peak Tensile Strength	Average Peak Strain
HDPE	9% increase	no change
PVC	8% decrease	3% increase
EIA (Solar)	10% decrease	9% decrease
SCF	21% decrease	23% decrease

Table 4. Cost Estimates

Material Type	Installed Cost (Taxes Extra)
HDPE	CDN $ 206,000
PVC	CDN $ 114,000
EIA (Solar)	CDN $ 440,000
SCF	CDN $ 439,000

Using cost as a selection criteria limited the next considerations to HDPE and PVC. It was apparent from the testing that both the PVC and the HDPE did exhibit some physical changes, but it appeared that these changes were not of significant magnitude to affect the performance of the liner system.

A temperature of 125°C is above what may be considered the melting point of HDPE and PVC lining materials. Although processing temperatures for both these materials are normally in the range of 250°C or higher, a sustained temperature in the 125°C range could be expected to cause some significant changes. The small scale testing showed, however, that the temperature below the liner was much lower than the pouring temperature (approximately 80°C). Both the PVC and the HDPE appeared to be performing as insulators. Initial sulfur contact with the cool liner may have been providing an initial layer of solid sulfur that helped to mitigate the effects of temperature. The slow placement of sulfur in the small scale test may have contributed to a crust of sulfur forming.

HDPE Discussion. The HDPE initially warped so as to lift the sulfur mold off the material surface, spilling molten sulfur and requiring a repeat test. This warping was permanent as the sample remained warped after cooling. It was expected that the HDPE warpage was due to stress relaxation. No significant shrinkage was found. It was felt that the warpage observed in the HDPE could cause problems. Large scale warpage could cause mobilization of sections of the liner system or the raising of voids under large wrinkles in the liner.

The visual observations of the surface of the HDPE (Table 2) indicated that after contact with the molten sulfur the HDPE had taken on a lighter color. An attempt was made to wash off this color but it was a permanent change. HDPE has an extremely high chemical resistance and is normally not affected by inorganic compounds. At the elevated temperatures of the test, however, a chemical reaction on the surface of the material may have occurred. The physical strength trends (Table 3) showed a tendency toward higher tensile strengths after exposure.

One factor that must always be taken into consideration with a new HDPE application is environmental stress cracking resistance (ESCR). Evidence pointed to a physical change, albeit small, in the HDPE. Since ESCR is a surface phenomenon of polyethylene, and evidence was observed of possible chemical changes to the surface of the HDPE sample, it was possible that the risk of ESCR had changed. It was not possible, given this simple test, of knowing whether the observed changes would have improved or deteriorated the ESCR performance of the material. Additional testing would have needed to be done to determine if the ESCR of the HDPE material had changed. Determining any effects on the HDPE's ESCR performance was beyond the scope of this material selection testing. The recommendation for continuing investigation of HDPE as a lining material was that an ESCR test be performed to determine if the HDPE liner performance had changed.

PVC Discussion. Original expectations of the PVC's performance were that the high temperatures would produce an unacceptable softening of the material. In fact, the temperature plots on the PVC sample showed that the maximum temperature reached, as measured under the liner, was only 80°C, well below the softening point of PVC. Observations in Table 2 indicated that the PVC sample did show some permanent deformation; the outline of the metal sulfur mold made an impression in the PVC that did not relax after the test. The PVC did not appear damaged by this deformation and showed no signs of distress. It was felt that the mould served as a heat sink for the sulfur, directing the heat in a concentrated line on the PVC material. The small amount of deformation present, and the absence of shrinkage, indicated that the PVC would not exhibit any unexpected large scale deformations in contact with molten sulfur. The visual observations of the surface of the PVC (Table 2) indicated that after contact with the sulfur the PVC had taken on a lighter color. An attempt was made to wash off this color but it was a permanent change. As with the HDPE, this color change may have indicated a chemical change to the surface of the geomembrane. Flexible PVC is not however, susceptible to environmental stress cracking.

Flexible PVC geomembrane materials are mixtures of (rigid) PVC resin with a number of additives; the most important being the plasticizer. The plasticizer gives the material its flexibility. When plasticizers are extracted from PVC the material shows a characteristic increase in tensile strength and decrease in tensile elongation. If sufficient plasticizer is extracted then brittleness may occur. What was shown by the physical test data for the PVC testing is opposite to what would be expected from the scenario where plasticizer is extracted. The physical data showed a tendency for the PVC to decrease tensile strength and to increase strain at break. It is possible that the additional heat continued the dispersion of the plasticizer into the PVC matrix. In this case it appeared that the heat of the sulfur had acted to further plasticize the material and to make it more flexible.

Small Scale Test: Recommendations. At the conclusion of the small scale test two materials appeared largely suitable for the containment of molten sulfur. Both the PVC and the HDPE had stood up well to the heat of contact, and the observed changes were reasonable for the expected use of the liner. Before a final selection could be made however, a larger scale test would be required.

LARGE SCALE TEST

Large Scale Test: Material Selection. Given the results from the small scale testing it was possible to narrow the field of possible materials to two. HDPE and PVC appeared to withstand the molten sulfur and were within budgeted cost estimates. South-central Alberta, where the proposed sulfur block liners were to be installed has a temperature range of +30 to -35°C. In order to allow for thermal effects on an exposed liner, it must be installed with sufficient slack to allow for contraction at cold temperatures without inducing stress. Three problem areas arose with the HDPE liner as a result of the required compensation slack. The sulfur block areas had a grade of only 0.7 meters vertical to 100 meters horizontal. HDPE compensation wrinkles typically stand up between 100 and 200 mm and may have impeded drainage on this very flat pad. Pouring molten sulfur onto standing water was possible, but drainage was a concern. The second area of concern was with the possibility of void spaces under HDPE wrinkles as the sulfur block was poured. The small scale test had shown warpage in the HDPE after exposure to molten sulfur. It was possible that this warpage could exacerbate the compensation wrinkles already in place in the liner, or cause wrinkles to run together to form large additive wrinkles. The sulfur is poured in lifts of 100 mm at a time and impediments to the flow of the sulfur in the initial pours can make it difficult to set up a solid base. Since it is impossible to access the lined area once molten sulfur has started to flow, the possibility of forming void spaces of 300 to 400 mm high and of considerable length was a concern. It was felt that void spaces could induce unacceptable cracks in the block as it grew in size. The final area of concern was the construction schedule. The proposed time for the installation of the sulfur block was very late in the season. HDPE liners had been successfully installed in weather as cold as -20°C but maintaining a quality installation at these temperatures is very difficult.

The PVC material offered to solve a number of the problems that the HDPE had shown. Compensation wrinkles in PVC lie flat and do not normally impede drainage. PVC wrinkles also compress under low pressure, and are not expected to create void spaces. The warpage seen in the PVC during the small scale testing was of a different nature than the warpage of the HDPE. The PVC warpage was one of conformance to the base, while the HDPE warpage forced the mold up and out of position. The PVC was expected to lie flat and not interfere with sulfur pouring. PVC could also be installed much more quickly than HDPE allowing installation within the tight construction schedule. Two problem areas did exist for PVC: UV exposure and cold temperature flexibility. It is not common in Alberta for regular PVC lining materials to be exposed over the winter months. Although experience has shown that the UV stability of regular PVC is approximately 5 to 7 years at Alberta latitude, the cold temperature resistance of regular grades of PVC are marginal for an exposed liner. An exposed life expectancy of 7 years was acceptable to the client. Liner replacement or repair was possible after this time if sulfur had not been poured.

A PVC-style flexible liner would have a number of advantages over the stiffer HDPE liner. The UV resistance of a regular PVC liner was sufficient for the client's purpose, and the speed of construction and repairability was an advantage for this application. The limitation of the regular PVC material was that the cold temperatures expected at the site could possibly contribute to damage to the liner if the liner were impacted at cold temperatures. Since regular PVC materials have a typical cold temperature resistance of -26°C, available blends of PVC that might perform better were investigated.

The final selection for the large scale test were two styles of PVC material. The first, a regular PVC material blended for improved cold temperature resistance had a cold temperature resistance (ASTM D1760) of -29°C. The second material was an alloy of PVC and a modified rubber compound (PVC Alloy). This alloy material has a cold temperature resistance of -30°C and had the additional advantage of being a UV stabilized grade. Experience with this PVC Alloy grade had shown that installations as low as -35°C were possible. After reviewing our report on the small scale testing, the client accepted the proposal for a large scale test utilizing the improved cold temperature regular PVC and the PVC Alloy material.

Large Scale Test: Method. Would the sustained heat of a large quantity of sulfur create different reactions or physical changes in the PVC materials ? If a crust of solid sulfur had formed directly on the cool liner surface during the small scale test (as we suspected) would a larger amount of sulfur re-melt this crust and adversely affect the liner ? The large scale test was designed to answer these questions.

The large scale test exposed the two liner samples to molten sulfur at conditions expected during service at the site. Two samples were prepared to represent the materials. One sample each of regular PVC and PVC Alloy were prepared in panels approximately 3 meters square. These sample panels were marked for later shrinkage measurements. Each sample contained a fusion welded factory seam and a solvent seam placed approximately 600 mm apart and running across the full width of the sample. A 2.7 meter square wooden frame was prepared on the Shantz site for each sample. Thermistors were placed to measure temperatures; two below the lining samples and one suspended in the sulfur above the liner to measure actual pouring temperature. A 50 mm thickness of molten sulfur was placed on each sample and allowed to cool.

A basic comparison between an unexposed sample and the exposed liner sample was made. Tests of seams determined if exposure reduced seam properties to values below acceptable values. The tensile strength of the factory and solvent welded seams were compared to the exposed material strength. Destructive tests were performed to ASTM D882 (5 samples from each location tested).

Large Scale Test: Results. Observation of the samples removed from the large scale test showed that the sulfur had bonded to the PVC materials but was relatively easy to chip away from the liner (see Figure 1). The discoloration evident in the small scale test was present in this large scale test. The PVC materials had conformed to the subgrade during the application of the molten sulfur which "set" in a position that mirrored the irregularities of the subgrade.

The temperature under the lining samples reached a maximum of 103°C under the PVC liner and 116°C under the PVC Alloy. These maximum temperatures were reached about three and a half minutes after the start of the pour. The maximum observed temperature of the molten sulfur during pouring was approximately 125°C. The sulfur was allowed to cool for about two hours before stripping the liner samples for testing. Both samples were marked and measured for shrinkage. Both the PVC and the PVC Alloy showed negligible shrinkage (less than 1%).

Large Scale Test: Discussion. The results of the large scale test showed that additional heating of the regular PVC material continued the reaction shown during the small scale test. The 19% strength reduction of the PVC material (Table 5) indicated that additional heating from a large amount of molten sulfur created additional changes.

The seam shear results in Table 6 however, showed that the seam shear strengths after exposure were still within 10% of the initial material strength. Seam shear strengths were compared to the strength of unexposed, unseamed sheet samples.

Table 5. Large Scale Test.
Unseamed Sheet Tensile Test Results

Material Type	Average Tensile Strength (MPa)	% Change
PVC Unexposed	9.5	
PVC Exposed	7.7	19 % decrease
PVC Alloy Unexposed	4.9	
PVC Alloy Exposed	5.0	2 % increase

Table 6. Large Scale Test.
Seam Shear Tensile Test Results
Exposed Samples

Material Type	Average Tensile Strength (MPa)	% of Initial Material Strength	
PVC Factory Seam	9.0	9.0 vs 9.5	95%
PVC Field Seam	8.7	8.7 vs 9.5	92%
PVC Alloy Fact.	4.9	4.9 vs 4.9	100%
PVC Alloy Field	5.0	5.0 vs 4.9	102%

All seam samples exhibited film tear bond failures for all shear tests.

Table 7. Large Scale Test.
Seam Peel Tensile Test Results
Exposed Samples

Material Type	Average Peak Load (N/mm)	% of Unexposed Material Peak Load (N/mm vs N/mm)	
PVC Factory Seam	5.72	5.72 vs 7.35	78%
PVC Field Seam	5.04	5.04 vs 7.35	69%
PVC Alloy Fact.	4.29	4.29 vs 4.03	106%
PVC Alloy Field	3.25	3.25 vs 4.03	81%

All peel test samples showed a film tear bond in peel except the PVC Alloy field seam. One of the PVC Alloy field seams showed a film tear bond, the rest showed a failure in peel.

The PVC Alloy material showed negligible change in material tensile strength after exposure to the molten sulfur (Table 5). The PVC Alloy material also showed seam shear tensile strengths (Table 6) that were substantially equal to the unexposed material strength. The seam peel test results in Table 7 showed that the factory seam exposed to molten sulfur was equal in strength to the unexposed material strength. The PVC Alloy solvent weld peel strength after exposure of 81% of initial material strength (Table 7) was considered to be an excellent peel strength result for a solvent weld. A solvent weld peel strength of over 70% of initial material strength is considered a very good result.

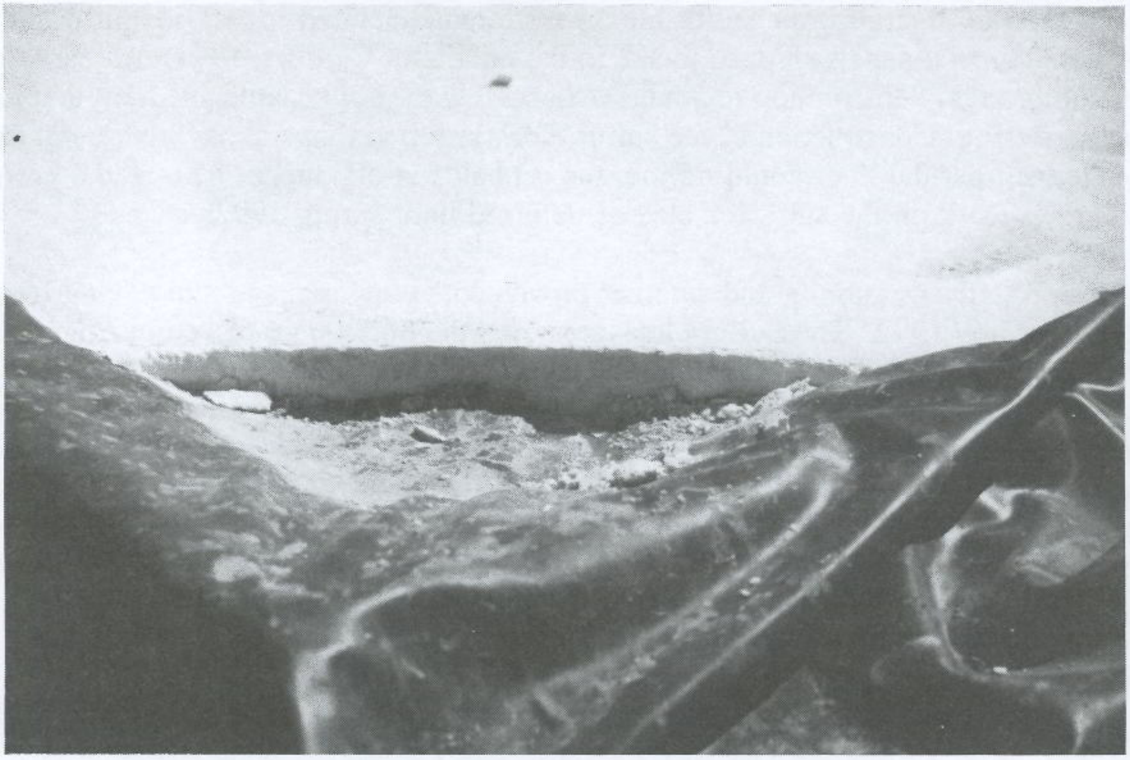

Figure 1. Solidified sulfur is removed from the sample at the completion of the large scale test.

The PVC Alloy material showed an overall excellent response to the large scale molten sulfur test. As a result of this large scale test the PVC Alloy material was selected. A cost comparison was prepared and the PVC Alloy material was estimated at CDN $ 149,000, which compared favourably to the PVC and HDPE material cost estimates prepared earlier. A report based on the test results was forwarded to the client recommending that the PVC Alloy would meet the project requirements.

Large Scale Test: Conclusion. On August 1st, 1991, Shell Canada Limited requested that a representative from Layfield Plastics attend a meeting with Alberta Environment. The environmental protection plan that Shell Canada Limited had proposed was reviewed at this meeting. A substantial part of the discussion pertained to the requirements of the sulfur block linings. The test results were discussed and a number of questions were answered. At the conclusion of this meeting Shell Canada Limited was informed that their permit for the Shantz site was approved.

The testing program had shown that a geomembrane liner of reasonable cost could meet the requirements of ground protection for a sulfur block. The geomembrane would allow for the complete removal of sulfur from the site without creating contaminated sulfur or soil.

CONSTRUCTION

The PVC Alloy liners for the sulfur blocks were manufactured in 18 identical 33 m x 36 m pieces with the first panels shipped to site in the middle of October, 1991. An early snow on October 17th brought construction to a halt. After a few days it became apparent that the snow would be persistent. Construction of the sulfur block required that site paving around the block be completed so that the liner could tie into the asphalt run-off ditches. Since the ground was now frozen, all work on the sulfur block was deferred until spring 1992.

An unexpectedly wet spring and summer prevented earthworks and site paving from being completed until July 1992. The Sulfur block areas and the liners were placed in August of 1992. A 400 mm high berm was prepared around the perimeter of each block to mark the liner location and to allow the aluminum sulfur forms to be placed. A strip of lining material was placed under each of these small berms during construction. The main liner panels were welded to these strips using solvent bonding. Repairs were accomplished using solvent welding techniques. Field welding was carried out in accordance with established Quality Control practices which included frequent qualification welds. All field welds and repairs were tested using the air lance test.

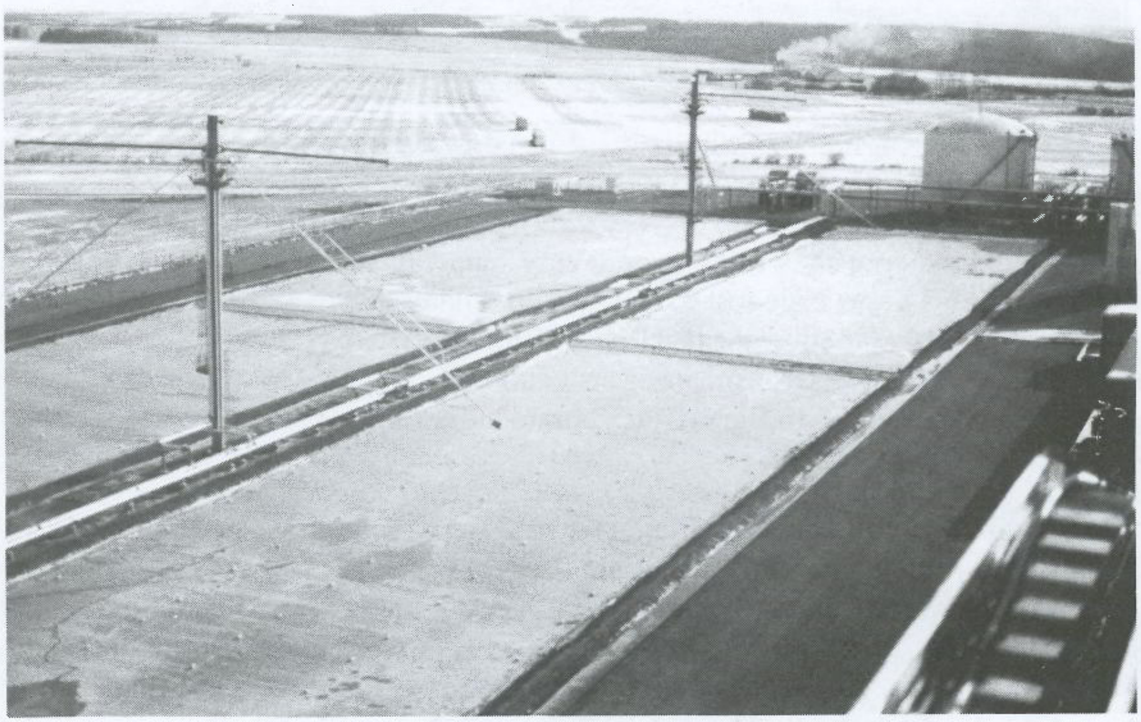

Figure 2. Sulfur Block Construction.
Four of the six cells are shown.

The sulfur block liners were permanently ballasted against wind uplift by using concrete weights. These concrete weights were actually concrete test cylinders collected from local engineering laboratories. The cylinders occasionally had small pieces of solid sulfur left over from compression testing. These ballast weights were available at low cost, and the attached traces of sulfur were not a concern on this particular project.

CONCLUSION

The Shantz sulfur facility is an example of the successful selection of a suitable geomembrane material in an application that had not been attempted previously. No one geomembrane meets all the lining needs of every application. The high temperatures expected in this project, as well as some unique site conditions required a logical approach to the determination of a suitable liner. This program of testing, that modelled site conditions, allowed the establishment of a high level of confidence in the material selected. This confidence allowed Shell Canada to receive construction permits for a previously untried application.

ACKNOWLEDGEMENTS

Layfield Plastics would like to thank Tom Moffat of Shell Canada Limited for his support. We would also like to thank Mike Kaut of Petrotech Lavalin and Tim Murphy of EBA Engineering for their assistance.

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