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August 11, 1983

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BAQM
REPLY TO:
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Mr. Ed Huck
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Department of Environmental
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Twin Towers Office Building
Tallahassee, Florida 32301

Re: South Pierce Prilled Sulphur Facility

Dear Ed:

Enclosed is the Burza report you requested and as referenced on page 9 of the most recent submission. Please call if you have any further questions.

Very truly yours,

PEEPLES, EARL, REYNOLDS &
BLANK, P. A.


William L. Earl
For the Firm

WLE/re

Enclosure

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**ANALYSIS OF ISSUES RELATING TO SOUTH
PIERCE PRILLED SULPHUR FACILITY**

Submitted By:

FREEPORT SULPHUR COMPANY

and

FREEPORT LAND COMPANY

August 8, 1983

CONTENTS

ANALYSIS OF ISSUES RELATING TO SOUTH PIERCE PRILLED SULPHUR FACILITY

I.	Lack of Reasonable Assurance That Moisture Content Will Be Sufficient At South Pierce.	2
	Moisture Content Decreases During Rail Transport According to Agrico Data.	3
	Agrico's Documented Moisture Losses.	4
	Wettability Studies Show That Sulphur Is Hydrophobic - It Repels Water.	5
II.	Agrico's Latest Emission Factor for Prilled Sulphur Contradicts Other Prior Submittals.	5
III.	Actual Observations of Commercial Prilled Sulphur Operations at High Moisture Levels Show that Particulate Emissions Are Substantial.	8
IV.	Lack of Information and Other Problems Related To Control Technology.	10
	Potential Secondary Emissions and Environmental Impacts Associated with the H ₂ S Scrubber Liquor.	11
	The Explosion Potential Associated With Agrico's Proposed Particulate Scrubber.	12
	The Newly Proposed Screw Auger Conveyor.	13
V.	The Type and Nature of the Expected Sulphur Particulate Emissions Is Not Clear and Unsubstantiated.	14
	Number of Emission Points.	15
	Agrico Has Not Substantiated Other Air Emissions From The Melter.	15

**ANALYSIS OF ISSUES RELATING TO SOUTH
PIERCE PRILLED SULPHUR FACILITY**

This analysis is presented with the accompanying materials to assist the Department in identifying important areas of consideration in determining the merits of Agrico's South Pierce prilled sulphur permit application. In general, the areas of concern are:

- 1) Lack of information necessary to evaluate any emission factor, such as moisture content and product history.
- 2) The unknown nature of the product and inability to control or ascertain its characteristics.
- 3) The difficulty in wetting sulphur and keeping it wet.
- 4) Questionable control technology.
- 5) A lack of essential information, and data that directly conflicts with prior submittals.

These issues, problems and information gaps demonstrate that there is no reasonable assurance that the proposed South Pierce facility will not emit air pollutants in quantities that exceed Department standards.

I. Lack of Reasonable Assurance That Moisture Content Will Be Sufficient At South Pierce.

The moisture content of prilled sulphur has been represented by Agrico as critical to control emissions. Yet little or no information has been provided about the moisture content or other specifications or characteristics of the prilled sulphur Agrico now proposes to annually unload and melt at its South Pierce facility. All that is known from the official record is that the prilled sulphur will arrive by truck or rail.

When the South Pierce facility was first proposed, at least some of the prilled sulphur was apparently to come from Agrico's proposed Big Bend facility in Tampa where Agrico asserted it could control the moisture content of the prilled sulphur destined for South Pierce. The proposed Big Bend facility, however, is not fully permitted and, in any event, the Big Bend facility could handle only half (300,000 tons per year) of South Pierce's proposed capacity (600,000 tons per year). Agrico therefore must obtain a substantial quantity, if not all, of the South Pierce prilled sulphur from another unknown source and has not demonstrated how it can control the moisture content, fines content, silt content, or other factors crucial to control dusting properties of the material. The unspecified prilled sulphur to be trucked or trained to South Pierce will originate somewhere outside of Florida and outside the Department's jurisdiction and control.

While Agrico maintains that moisture content is critically related to dust emissions, there are no provisions in the South Pierce application for stockpiling, rewetting, monitoring moisture content, or determining other characteristics, like fines and silt content, which Agrico admits are important to controlling the product's dusting capacity. In Agrico's July 5, 1983 letter to DER, Agrico represents that the prilled sulphur "will not be processed if it is too dry", but when the prilled sulphur arrives in South Pierce, Agrico will not know and will have no control over these essential characteristics. Agrico, therefore, has not provided reasonable assurance or essential information critical in determining emissions.

A. Moisture Content Decreases During Rail Transport According to Agrico Data.

The relevance of Agrico's lack of control over the moisture content of the prilled sulphur to a reliable determination of emissions from the South Pierce facility is substantiated by documented decreases in moisture during rail transit. Measurements of prilled sulphur moisture content both before and after rail transit were made available to DER as part of its "Faustina test" in Agrico's original Big Bend air permit application, and during the course of the hearing on that application. This data shows about a 25% decline in moisture

during rail transit:

When loaded:	2% moisture
When unloaded:	1.47% moisture

In the instance of South Pierce, the moisture content of the prilled sulphur when loaded onto trucks and trains is unknown. Since there is no information about this material, including its prior handling, its point of origin or the duration of its journey to South Pierce, it is impossible to predict the moisture content upon arrival at South Pierce. Even if Agrico's latest emission factor requiring 4% moisture or greater is to be believed, that factor cannot be applied because of lack of information about the moisture of the material.

B. Agrico's Documented Moisture Losses.

The data developed by Agrico and presented to DER in its Big Bend application as part of its "Faustina test" provides further documentation of the tendency of prilled sulphur to lose moisture. This data, reproduced on Figure 1, shows the decline in the moisture content of a pile of prilled sulphur over about six weeks. During this period, there were several heavy rains reported by Agrico, including over 1½ inches of rain on two different days. Overall, moisture content in the Agrico test prill pile dropped from 1.36% to .55%. See Figure 1.

This moisture loss over time, as reported by Agrico, is similar to that experienced in the laboratories of TRC

Figure 1

DRAINDOWN OF AGRICO PRILLED SULPHUR STORAGE PILE**DAILY % MOISTURE ANALYSISRAINFALL
(*AFTER SAMPLE)

<u>DATE</u>	<u>PILE SURFACE</u>	<u>PILE CORE</u>	<u>DATE</u>	<u>INCHES</u>
5/31	0.05	1.36	5/27	0.24
6/1	0.15	1.17	6/1*	0.44
6/2	0.88	1.38	6/2*	0.11
6/6	0.13	1.02		
6/7	0.03	1.05		
6/8	0.07	0.92		
6/10	0.02	1.11	6/12-13	0.69
6/14	0.79	0.96		
6/15	0.08	1.03	6/14	0.05
6/20	-	1.09	6/16	0.04
6/22	0.05	0.95	6/19-20	1.24
6/24	0.04	0.90		
6/27	0.10	0.95		
6/28	0.05	1.00	6/28	0.01
6/30	0.05	1.00	6/29	0.19
7/7	0.00	0.50	7/3	1.79
7/8	0.05	0.60		
7/11	0.05	0.90	7/10	1.69
7/12	0.47	1.00		
7/14	0.05	1.10	7/13	0.81
7/15	0.0	0.85		
7/18	0.0	0.55		

**Source - Agrico submittal, September 1977.

Environmental Consultants, Inc. ("TRC"). There, "Burza" prilled sulphur, another water formed or "wet" prill, was air dried and curves were developed that relate moisture loss to elapsed time. The results of this investigation have been previously reported to the Department. Recent conclusions drawn by Agrico's consultants in Texas markedly differ from and directly contradict Agrico's prior Faustina data and the experience of Freeport and Freeport's consultants. The importance of assuring that moisture levels are maintained is acknowledged by Agrico to be critical. Therefore, reasonable assurance is lacking.

C. Wettability Studies Show That Sulphur Is Hydrophobic - It Repels Water.

Because Agrico's pollution control technology is so highly dependent on wetting sulphur and keeping it wet, TRC has performed wettability tests on prilled sulphur. These tests utilized the methodology developed by the United States Bureau of Mines for determining the relative wettability of different coals. For accurate comparison, the same coal samples used by the Bureau of Mines were obtained by TRC for the tests. The results, presented in an accompanying report, reveal that sulphur is extremely unwettable.

II. Agrico's Latest Emission Factor for Prilled Sulphur Contradicts Other Prior Submittals.

Without reasonable assurance about the product's moisture content, and fines and silt content, no emission factor can be

rationally applied to the South Pierce facility. However, even were this information available, Agrico has so changed the emission factor it asserts should be applied, there can be no reasonable assurance which factor is correct. For example, Agrico's initial South Pierce emission factor presumed that the prilled sulphur would contain approximately 2% moisture. (Agrico's South Pierce Permit application Supplements 2 and 3, May 7, 1982). This factor first appeared in a February 28, 1979 report prepared for Agrico by Dr. Dale A. Lundgren. Although the .0068 pounds per ton factor has been changed by Agrico in subsequent submittals to DER, Agrico has repeatedly represented there would be "no dust" at 2% moisture, and these representations were a predicate to the issuance of Agrico's Big Bend permit, and the conditions of that permit.

This once-guaranteed 2% moisture content has apparently now been abandoned by Agrico. In another report by Dr. Lundgren dated February 1, 1983, a higher emission factor -- 0.01 lbs./ton -- is presented for 3% moisture content. Interestingly, this report emphasizes that the emission factor is solely dependent upon moisture, and that fines content is not important:

The tumble tests and multiple drop tests clearly show that only the product moisture content relates to product dust generation. Although measures of various sieve fractions of a product may be of interest and value for other purposes, it cannot directly correlate with product dust generation . . .

In its submittal dated June 3, 1983, Agrico directly contradicted these conclusions and presented another prilled sulphur emission factor of .002 pounds per ton per foot drop, this time at 4% moisture content. Unlike the earlier submittal, this latest submittal expressly admits the importance of fines content to determine emissions, contradicting the prior submittal:

[T]he dust emission factor is primarily dependent on moisture level at high moisture (perhaps 3% to 6% moisture) but is dependent on both moisture level and sieve size distribution (or % fines) at low moisture (~2% moisture). However, at both high and low moisture a definite conclusion can be reached. At high moisture level (~4%) all sulfur prill products tested had very low emission factors. At low moisture levels (<0.1%) the dust emission factor is always quite high.

If this latest Agrico statement is true, it underscores a lack of reasonable assurance because it is known that over time and during transportation, the moisture content of prilled sulphur declines. Further, during transportation and handling the percentage of fines and silt will increase. Agrico's latest emission factor, and the various other emission factors Agrico has used to date, cannot be reliably applied because Agrico has no control over the moisture content or handling history of the 600,000 tons of prilled sulphur it proposes to annually offload at South Pierce. Moreover, as shown below, actual observations of commercial prilled sulphur operations there have shown substantial dust, even at high moisture levels.

III. Actual Observations of Commercial Prilled
Sulphur Operations at High Moisture Levels
Show that Particulate Emissions Are Substantial.

The high moisture levels now recommended by Agrico in its latest emission factor are based largely on the contention that moisture binds together or "agglomerates" the smaller, potentially airborne particles. A simple demonstration of this asserted phenomenon, using a sieve, has even been presented by Agrico, has not been borne out by actual observations of prilled sulphur handling in unstaged, commercial settings. Some examples of these observations are summarized below.

1. Leith, Scotland -- April, 1983.

In April of 1983, Dr. D. J. Miller of Freeport witnessed, photographed, and made motion pictures of the unloading of prilled sulphur from the vessel "Atlantic Confidence" at Leith, Scotland. Dr. Miller's technical memorandum and photographs accompany this report. In a two-day period, amid episodes of rain, substantial emissions were observed and documented. The "Atlantic Confidence" contained only wet formed prilled sulphur, and samples were obtained for analysis. The subsequent analysis of the samples showed an average surface moisture content of 2.45%. The analytical report further characterizing the "Atlantic Confidence" samples as to moisture content and silt and fines content accompanies this report.

2. Vancouver, British Columbia -- January, 1983.

In January of 1983, Freeport representatives and several others had occasion to view a prilled sulphur pile at Vancouver Wharves in British Columbia. The day before was very rainy and, at times, the rainfall was torrential. Windblown dust emissions were observed from a vantage point approximately fifty yards downwind of the prilled sulphur pile. At the time, there was no activity at the facility; the pile was "at rest." Sulphur dust particles could be felt striking the observer's face. No sample could be obtained, but given the extreme weather on the previous day, it is believed that moisture levels were high.

3. Fort McMurray, Alberta -- October, 1982.

At Burza Resources in northern Alberta Province, Freeport representatives and consultants observed and photographed airborne sulphur dust covering the surface of a utility van parked downwind of a conveyor transfer point where newly-manufactured water formed prilled sulphur was being loaded into a railcar. The surface moisture content of this newly made prilled sulphur was later measured to average 3.6%. Observations and technical data relating to this occurrence have been previously reported to the Department.

The above observations are only some of the "real world" instances where significant emissions were observed during actual, unstaged commercial operations. In each instance, the

moisture content of the prilled sulphur was either actually measured or reasonably estimated to be high. Even at these high levels of moisture there are substantial dust emissions during commercial handling of prilled sulphur.

IV. Lack of Information and Other Problems
Related to Control Technology.

Apart from commercial realities and the inability to apply any emission factor to a product with unknown characteristics, there are numerous other problems and even dangers from other control technologies proposed by Agrico.

In addition to keeping the product wet, Agrico has proposed to use scrubbers to control particulate and hydrogen sulphide (H_2S) emissions. These proposed control technologies are unproven for this kind of facility and can create potential dangers and even secondary emissions. In Agrico's original May 7, 1982 permit application, Agrico proposed a caustic scrubber to remove H_2S gas emitted during the sulphur melting process. On May 13, 1983, Agrico submitted a new flow diagram (dated May 11, 1983) introducing a wet scrubbing system located at the conveyor discharge to the hopper, and substituting a screw auger for the vibrating feeder to the melter. Several of the problems and deficiencies related to these devices are discussed below.

A. Potential Secondary Emissions and
Environmental Impacts Associated
with the H₂S Scrubber Liquor.

Agrico's proposed caustic scrubbing process for H₂S gas is expected to generate a hydrosulphide blowdown sludge that will require careful management for safety and disposal purposes, and to prevent adverse environmental impacts such as the additional release of H₂S gas. DER has formally requested, but Agrico has not provided information concerning this scrubber liquor, other than to state that the liquor will be discharged to a "cooling pond." This pond will also receive a waste stream from the proposed unloading hopper dust collection system and the surge hopper wet scrubber system. (C.H. Fancy letter to Agrico, June 9, 1982; Agrico letter to C.H. Fancy, September 22, 1982, enclosing design engineer's letter, April 1, 1982.)

Absent this requested information on the scrubber liquor, reasonable assurance has not been provided. In an aqueous system, H₂S gas can be released from such a hydrosulphide sludge if the pH of the system falls below 7.0. The potential exists, then, for the release of additional H₂S gas from the scrubber blowdown sludge if it is discharged to an incompatible receiving pond or commingled with other incompatible wastes. More information is required to assess these secondary emissions. The characteristics of the waste streams generated by Agrico's air pollution control devices and the characteristics of the receiving pond or ponds have not been provided. This shortcoming

is particularly important as a release of only a small amount of additional H₂S from such a waste pond, when added to the already reported controlled emission of 8.4 tons of H₂S per year from the scrubber, could trigger PSD review. Chapter 17-2, Florida Administrative Code, Table 500-2.

B. The Explosion Potential Associated with Agrico's Proposed Particulate Scrubber.

It has long been recognized that dust collection systems, in general, are inappropriate to control sulphur dust. Any attempt to confine sulphur dust can lead to the accumulation of explosive concentrations of sulphur dust. On the question of whether wet scrubber technology is appropriate to control sulphur dust, the DER Southwest District permitting staff has expressed a position on this subject and testified under oath:

[M]y understanding is that sulfur dust is explosive when confined and therefore we felt those type of divisions [scrubbers or baghouses] could not be reasonably employed. [DOAH Case No. 78-315, transcript at 1456.]

In addition to this testimony, findings of fact on the explosion potential of enclosed sulphur dust were adopted by the Department. [DOAH Case No. 78-315, Recommended Order at p.10.]. Other materials submitted to the Department substantiate these findings and testimony.

Agrico's newly proposed enclosed collection system at the hopper could result in an explosive accumulation of sulphur dust.

Surely such a potential for an explosion compromises the reliability of the control system.

In addition to this explosion potential, Agrico has failed to provide the Department with adequate technical operating data for the scrubber. Without such information as equipment size, flow rates, and rated capacity, the efficiency of the system cannot be adequately assessed; therefore, reasonable assurance is lacking.

C. The Newly Proposed Screw Auger Conveyor.

Agrico has placed great emphasis on its intention not to stockpile any of the 600,000 tons of prilled sulphur at South Pierce. E.g., de la Parte letter, 7/5/83. In its recently amended application, Agrico substituted a screw auger conveyor in lieu of the vibrating feeder to convey the prilled sulphur to the melter. This newly proposed conveying system underscores the already serious question about the system's capability to handle product throughput as the sulphur arrives and is unloaded at South Pierce. If the system malfunctions, or if for any reason cannot handle the product at an adequate rate, the prilled sulphur must, of necessity, either be rejected and sent back, or allowed to sit during lengthy delays in idle trains and trucks or, alternatively, stockpiled. In a commercial setting, Agrico's choice of these alternatives will not be difficult.

V. The Type and Nature of the Expected Sulphur
Particulate Emissions Is Not Clear and
Unsubstantiated.

In Agrico's original application the process weight table was applied to the two emission points Agrico asserted would exist at the South Pierce facility at the unloading shed and at the conveyor/hopper drop. In DER's June 9th request for additional information, Agrico was asked to justify its assertion that there would be only two emission points since there are five drop/transfer points. This request also pointed out that the nature of the emissions would be "fugitive particulate." In responding to this request, Agrico included a revision to its permit application using its then latest emission factor for fugitive emissions in place of the process weight table (Agrico letter dated September 22, 1982). Now, the most recent Agrico revision provides for the wet scrubber placed at the conveyor/hopper drop and, therefore, there is now a "point source" vented from the hopper through the scrubber. (Agrico revisions, 5/13/83). Agrico has not, however, quantified or substantiated the emissions from this point source. Absent this information, there can be no reasonable assurance.

A. Number of Emission Points.

Agrico has represented that although there will be five drop/transfer points from which sulphur particulate emissions are generated, there will only be two emission points: at the drop from the truck/railcar unloader into the unloading hopper, and at the discharge from the covered conveyor into the surge hopper. The other three drop transfer points -- from the unloading hopper into a "covered" conveyor, from the surge hopper into the "enclosed" feeder, and from the feeder into the "enclosed" melters -- will not, according to Agrico, emit particulate because they are to be "enclosed." In other words, although particulate matter may be generated from the material, it will not escape into the air because those systems are closed. Leaving aside the already discussed explosion potential of these enclosures, it remains unknown where this material will go and whether emissions from transfer points were included in the emissions estimates from the two "given" emission points.

B. Agrico Has Not Substantiated Other Air Emissions From The Melter.

In DER's letter of June 9, 1982, Agrico was formally requested to provide information as to other criteria pollutants emitted from the sulphur melters, including detailed calculations about the emissions of such other pollutants. In its response of September 22, 1982, Agrico made bare assertions about SO₂ emissions from the melters without substantiation. No other

pollutants were discussed. Without more specific information about the possible characteristics of the prilled sulphur, including the possible presence of surfactants and emissions of other pollutants, reasonable assurance is lacking.

Conclusion

There are numerous deficiencies in the Agrico South Pierce submittals that relate directly to air pollutant emissions and demonstrate that there is no reasonable assurance. The most striking lack of information concerns the nature of the product itself, which makes the reliable application of any emission factor impossible. Agrico has also asserted a new emission factor, substantially different than its predecessors, which must be carefully examined and analyzed. Factual deficiencies about the characteristics of the scrubber systems compromise their reliability as a pollution control for this type of facility. Finally, the configuration of the facility and the lack of design and performance details for its major components, such as the conveyor system and screw auger, prevent any accurate analysis of potential emissions from the facility. Agrico has not yet provided reasonable assurance that this facility can meet Department standards.

AGRICO PRILLED SULPHUR EMISSION FACTORS (BY DATE)

<u>DATE</u>	<u>EMISSION FACTOR</u>	<u>COMMENT</u>
February, 1978	0.2 lbs./ton	transfer point
February 1, 1979	0.0068 lbs./ton	at 2% moisture
February 1, 1983	0.01 lbs./ton	at 3% moisture
June 3, 1983	0.01 lbs./ton	five-foot drop at 4% moisture

PRILLED SULPHUR MOISTURE LOSS DURING RAIL TRANSPORTATION

PERCENT MOISTURE AT ORIGIN OF RAIL SHIPMENT - 2.0%*

PERCENT MOISTURE AT DESTINATION OF RAIL SHIPMENT - 1.47%**

*Source - Canadian Superior Oil Ltd.

**Source - Agrico submittal, September 1977

TECHNICAL MEMORANDUM ON OBSERVATIONS
OF
OFFLOADING WET PRILLED SULPHUR FROM
THE VESSEL "ATLANTIC CONFIDENCE"
AT
LEITH, SCOTLAND
APRIL, 1983

by

DR. D. J. MILLER

Freeport-McMoRan, Inc.

TECHNICAL MEMORANDUM ON OBSERVATIONS
OF
OFFLOADING WET PRILLED SULPHUR FROM
THE VESSEL "ATLANTIC CONFIDENCE"
AT
LEITH, SCOTLAND

On April 7 and 8, 1983, the author witnessed the unloading of water-formed prilled sulphur ("wet prills") in Leith, Scotland. (See map, attached). The cargo was being unloaded from the vessel, "Atlantic Confidence," which had four days earlier unloaded part of its wet prilled cargo at Immingham, England. This memorandum will document my observations relative to this shipment.

Sulphur Loading

The "Atlantic Confidence" was loaded on February 28 - March 1, 1983, at the Vancouver Wharves in North Vancouver, British Columbia. I observed this loading and noted the generation of sulphur dust at the stockpile. The stockpile was being worked by two rubber-tired front-end loaders. The front-end loaders were used to move the water-formed prilled sulphur from the stockpile to a grating that feeds a conveyor belt. From there, the sulphur was then transported through a number of transfer points to a shiploader equipped with an "elephant trunk spout." Sulphur dust was observed at the point of discharge into the ship.

Initially, the stockpile was so large that very little front-end loader activity was required to feed the conveyor. As the

sulphur was removed, however, some increased activity was required to move the sulphur. Two major sources of airborne sulphur dust were evident in the immediate area of the stockpile. One source was the discharge of sulphur from the front-end loader bucket to the grating. As the water-formed prills were dropped, substantial visible releases of sulphur dust were evident. It had been raining intermittently during and for several days prior to the observations.

The second major source of airborne sulphur dust at the stockpile was the front-end loader traffic in the area. As the equipment moved from place to place, sulphur dust was raised by the tires. This resulted from the action of the tires upon the prills scattered about the area.

The sulphur loaded on the "Atlantic Confidence" in Vancouver was from a stockpile of PVC Commodities, Ltd. water-formed prills (PVC prills). This type of prill is known also as "wet prills," "wet-formed prills," etc. The entire ship was loaded from the PVC pile. Subsequent sampling and testing of the cargo confirmed that the cargo was 100% prills.

A portion of the "Atlantic Confidence" cargo was unloaded in Immingham, England prior to the delivery to Leith, Scotland. Leith (at Edinburgh) is approximately 220 miles from Immingham (See map attached).

Sulphur Unloading at Leith, Scotland

On April 7 and 8, 1983, the author observed at Leith the unloading of the PVC water-formed prill from the "Atlantic Confidence." Two large clamshell buckets were used to unload the prilled sulphur into trucks. The prilled sulphur was discharged directly into dump trucks for transport. Several sources of airborne sulphur dust were observed. As the clamshell bucket loaded with sulphur was dropped into the truck, the "hopper" effect was observed, causing substantial dust to ascend vertically into the air. Some displaced air, in addition to other forces acting on the sulphur, led to a discharge of substantial amounts of dust into the air.

In addition to the hopper effect, the horizontal wind acted on the falling sulphur prills and stripped fines or dust from the descending sulphur. The wind speed was variable during the unloading, with maximum velocities of approximately 10-15 miles per hour. The wind-stripping effect was evident whenever the wet prilled sulphur dropped through the wind. Along with the hopper effect, discussed above, wind stripping was a major source of airborne sulphur dust.

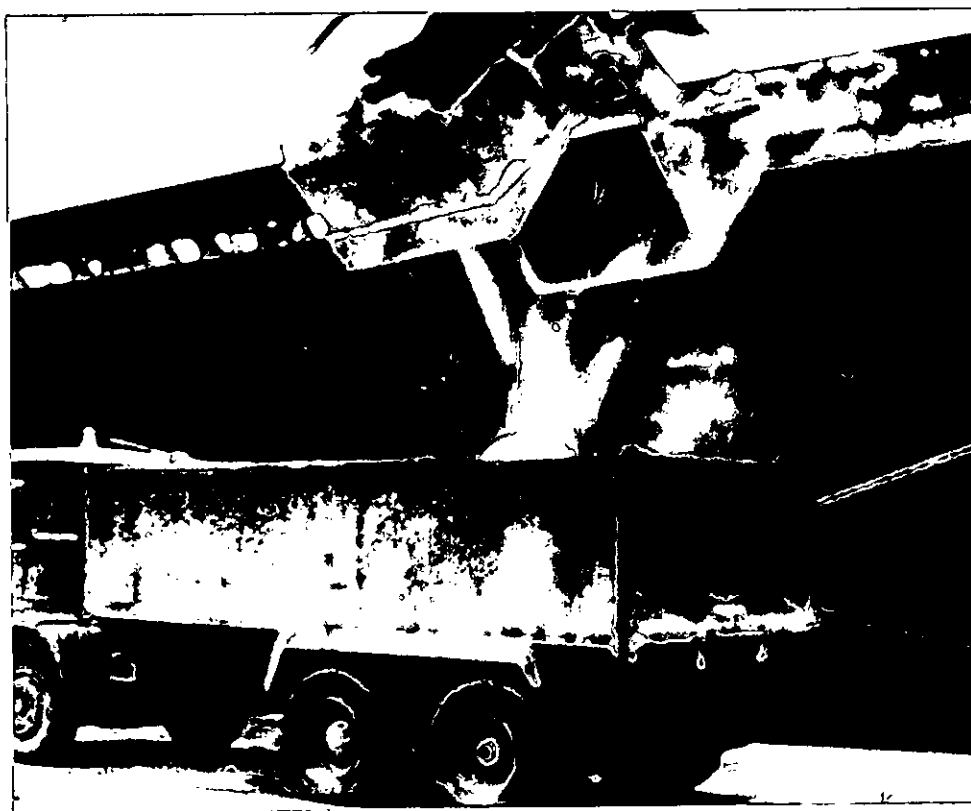
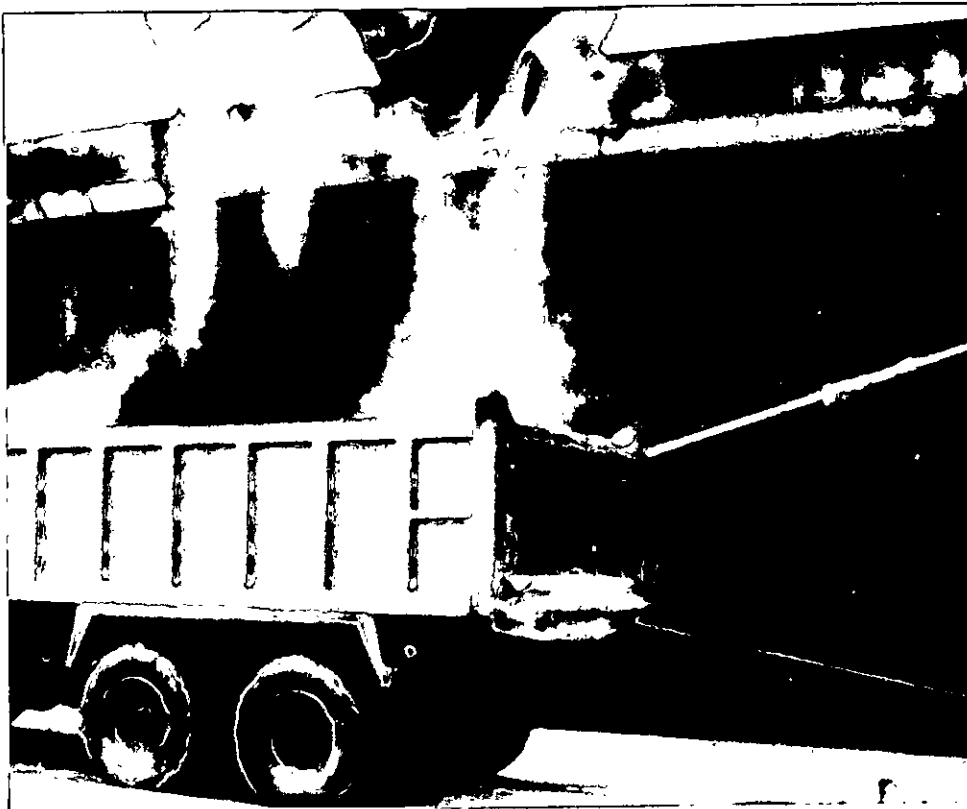
Another source of airborne sulphur dust was general truck traffic in the area of unloading and in the path of the sulphur trucks. This is related to housekeeping in the area, but under the circumstances at Leith, this spillage and emissions seemed unavoidable.

During portions of the unloading observed, intermittent rain was falling. This moisture did not appear to diminish the amount of sulphur dust being emitted into the air.

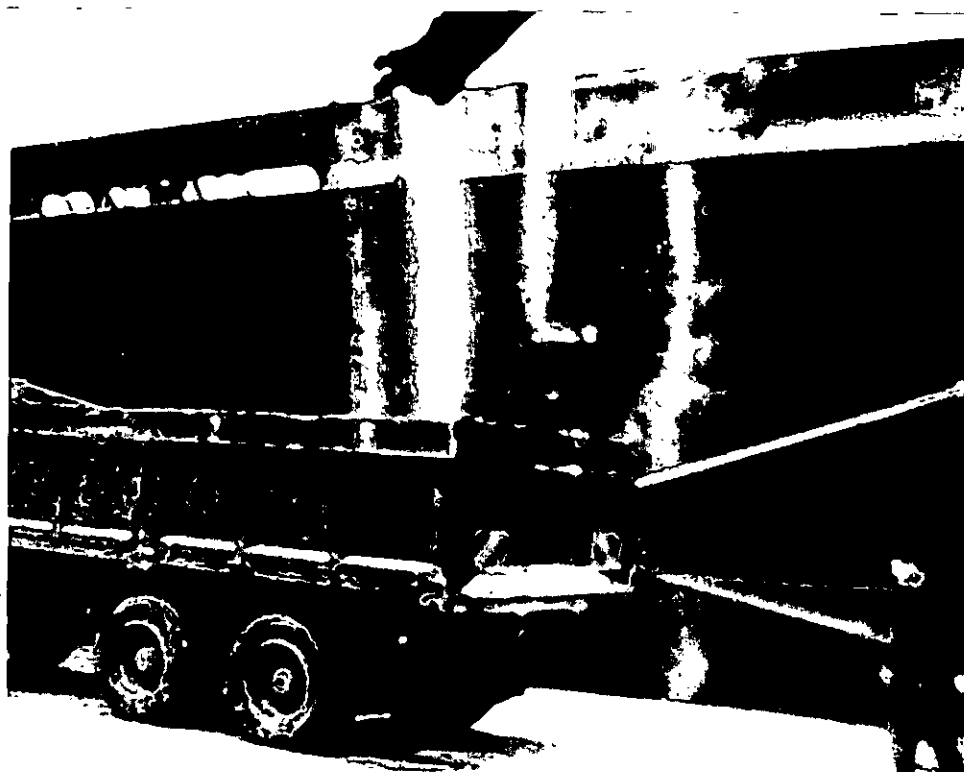
On April 13, 1983, I obtained samples of the prilled sulphur. Five 600 ml samples were taken from a truck that had just been loaded from the "Atlantic Confidence." The sulphur was so wet that the moisture was condensing in the containers. The sample jars were double sealed with tape and brought back to the United States for analysis to Mr. George V. Aseff of Law Engineering Testing Company.

Photographs of my observations accompany this memorandum.





OFFLOADING WET PRILLED SULPHUR FROM THE VESSEL
"ATLANTIC CONFIDENCE" AT
LEITH, SCOTLAND
APRIL, 1983



OFFLOADING WET PRILLED SULPHUR FROM THE VESSEL
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REPORT ON
COMPARATIVE WETTABILITY OF SULFUR
WITH SEVERAL TYPES OF COAL

by

J. E. Yocom
TRC Environmental Consultants, Inc.

The use of water sprays to wet bulk solids such as coal is a common practice to reduce dusting during mining, processing and transfer operations. Wetting of sulfur has been proposed as a means of reducing sulfur dust emissions in the handling of solid sulfur. Because of the recognized difficulties in wetting sulfur, there are questions on the effectiveness of this technique.

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The Bureau of Mines (USBM) has studied the relative wettability of a large number of coal types to determine the effectiveness of water containing surfactants in reducing dust generation from mining.* In this work they used several laboratory methods to determine relative wettability. They used the capillary rise test, a coal dust sinking test, and zeta potential measurements. Greatest reliance was placed on the capillary rise test, and the other two methods were used⁴ to supplement this test.

In this work the USBM found that coals exhibit a wide range of wettabilities. For coals relatively unwettable by water, the addition of wetting agents had only a minor effect in increasing wettability. In the in-mine tests, the addition of wetting agents decreased dust levels in the mine by only about 29 to 36 percent over those produced by water.

Described herein are laboratory tests conducted by TRC using the capillary rise test to confirm wettability data from the USBM work for selected coals and to compare these results with wettability data for sulfur. Briefly,

*J. A. Kost, G. A. Shirey, and C. T. Ford, "In-Mine Tests for Wetting Agent Effectiveness." Final Report to U. S. Bureau of Mines from Bituminous Coal Research, Inc. under Contract J0295041, December 1980.

this method consists of measuring the relative quantity of water absorbed by a 2 gram sample of minus 42 mesh coal (or sulfur) packed into an open 8 mm (inside diameter) capillary tube. The lower end of the tube is covered with a disc of glass fiber filter paper cemented to the tube. This filter keeps the material inside the tube and allows water to migrate up into the material. A series of pre-weighed tubes containing the materials under study is positioned in a rack with their lower ends in the same plane. A petri dish containing water is raised so that the water surface touches the filter papers. After 10 minutes in this position the water is lowered away from the tubes. The tubes are then weighed to determine the amount of water absorbed. Since the filter paper itself absorbs water, blanks were run (tubes with filters affixed but containing no material) and it was determined that, on the average, the filters alone absorbed 0.044 gram. This amount was always subtracted from the weight gain noted during each test.

Samples were obtained of three of the coals tested in the USBM study. They are:

<u>Coal Type</u>	<u>Particle Size</u>	<u>Quantity, g</u>
Hazard Lot 4381	-8 mesh	284.1
Pond Creek Seam Lot 4462	-8 mesh	226.9
Pond Creek Seam Lot 4462, 2278-24	-42 mesh	221.4
Cedar Grove Lot 4430	-8 mesh	125.1
Cedar Grove Lot 4430, 2278-24	-42 mesh	147.0

Initially, a 42 mesh sieve was unavailable in time to start the tests, so initial trials of the Hazard coal and sulfur (Fletcher-type prill from Burza) were conducted with material ground to minus 40 mesh. The Pond Creek and Cedar Grove coals were already available in minus 42 mesh form. Later,

when a 42-mesh sieve became available, the Hazard coal and sulfur were re-run ground to the minus 42 mesh fineness.

Table 1 is a copy of the first page of Table 10 from the USBM report summarizing the wettability results for the three coal types available. Note the relatively high wettability of the Hazard sample and the non-wettability of the Cedar Grove sample.

Table 2 summarizes the data collected in TRC's laboratory on July 29. All tests were carried out in triplicate with distilled, deionized water. TRC draws the following conclusions from this table:

- Sulfur is completely unwettable. In fact, the consistent negative values for water absorption imply that sulfur inhibits the complete wetting of the filter paper.
- There does not seem to be any significant difference in the results based on grinding sulfur and the Hazard coal sample to minus 40 or minus 42 mesh.
- The wettability of the Hazard coal sample (average of 10.6% for 6 tests) agrees extremely well with the results from the USBM tests (10.3% for Lot #4381).
- The measured wettability of the other two coals agree reasonably well with this USBM data.

From this work it is apparent that sulfur is less wettable than even the most unwettable coals. Thus, one would expect that it would be extremely difficult to achieve and maintain a uniform degree of wetting of solid sulfur in stationary piles and moving through a series of transfer operations.

TABLE 1: CAPILLARY RISE TEST RESULTS COMPARING BCR, USBM SAMPLES,
AND SURFACTANT SOLUTIONS WITH DEIONIZED WATER, HARD WATER, AND MINE WATER***

Seam	Lot Number	Surfactant*	Percent Weight Gain			
			Deionized Water	Hard Water	Mine Water #4388	Mine Water #4427
Hazard	4381	Water	10.3	12.2	-	-
	4537**	Water	14.5	15.5	-	-
Cedar Grove	4430	Water	0.0	0.0	-	-
		Aerosol MA-80	1.2	0.1	1.4	2.1
		Polytergent B300	0.6	1.6	-	-
		Tween 20	0.5	0.3	-	-
		Surfynol TG	1.3	1.6	0.8	2.1
	4538**	Water	0.0	0.0	-	-
		Aerosol MA-80	1.8	1.3	1.9	3.4
		Polytergent B300	0.0	1.1	-	-
		Tween 20	0.0	0.6	-	-
		Surfynol TG	2.1	1.0	1.1	2.1
Pond Creek	4462	Water	0.8	5.2	-	-
		Aerosol MA-80	8.8	9.7	-	-
		Polytergent B300	4.9	6.9	-	-
		Tween 20	4.8	7.4	-	-
		Surfynol TG	8.2	8.8	-	-
	4539**	Water	0.4	1.1	-	-
		Aerosol MA-80	1.9	2.4	-	-
		Polytergent B300	1.7	0.0	-	-
		Tween 20	0.1	0.7	-	-
		Surfynol TG	2.3	0.0	-	-

*Surfactant Concentration 0.1 percent.

**USBM sample.

***This is the first page of Table 10 in the report: "In-Mine Tests for Wetting Agent Effectiveness,"
Final Report to U.S. Bureau of Mines from Bituminous Coal Research, Inc., Contract J029504, December 1980.

TABLE 2

SUMMARY OF WETTABILITY DATA FOR
THREE COALS AND SULFUR

July 29, 1983

Test No.	Sample Description	Particle Size Mesh	Sample Size, g.	Weight Water Taken up to Liquid, g.	% Wet Weight of Sample due to Water
1	Sulfur	-40	1.97	-0.0053	Negative
2	Sulfur	-40	2.04	-0.0100	Negative
3	Sulfur	-40	2.03	-0.0033	Negative
4	Coal-Hazard	-40	2.03	0.2471	10.9
5	Coal-Hazard	-40	2.05	0.2191	9.7
6	Coal-Hazard	-40	2.08	0.2599	11.1
7	Coal-Cedar Grove	-42	2.06	-0.0080	Negative
8	Coal-Cedar Grove	-42	2.04	0.0064	0.31
9	Coal-Cedar Grove	-42	2.06	-0.0017	Negative
10	Coal-Pond Creek	-42	2.00	0.0081	0.40
11	Coal-Pond Creek	-42	2.01	0.0133	0.66
12	Coal-Pond Creek	-42	2.01	0.0035	0.17
13	Coal-Hazard	-42	2.05	0.2731	11.8
14	Coal-Hazard	-42	2.05	0.2235	9.8
15	Coal-Hazard	-42	2.02	0.2327	10.3
16	Sulfur	-42	2.04	-0.0101	Negative
17	Sulfur	-42	2.05	-0.0062	Negative
18	Sulfur	-42	2.02	-0.0072	Negative

AGRICO - SOUTH PIERCE - SULFUR PERMIT. 8/12/83

NAME	ORGANIZATION	PHONE
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Ed de la Parte	Agrico	813-327-2775
Martha Harrell Hall	DER/Legal	904-488-9730
Steve Smallwood	BAQM	904-488-1344

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August 8, 1983

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REPLY TO:

Miami

Mr. Steve Smallwood
Bureau Chief
Air Quality Management
Department of Environmental Regulation
2600 Blair Stone Road
Twin Towers Office Building
Tallahassee, Florida 32301

Re: Agrico Chemical Company, Proposed South
Pierce Sulphur Facility; Permit Application
No. AC53-55780; Sulphur Rulemaking Inquiry

Dear Steve:

The enclosed information and technical analysis is submitted on behalf of Freeport Sulphur Company and Freeport Land Company. Freeport believes the enclosed technical data requires a finding that Agrico has still failed to provide reasonable assurance that the South Pierce facility can meet Department standards. DER's decision on this matter is particularly important because a notice of intention to issue this permit would impair, if not destroy, the ongoing sulphur rulemaking inquiry and negate the meaningfulness of any rule which might emerge.

Freeport believes the new information and other materials in the record still do not support a finding of reasonable assurance and submits the attached analysis including the following technical data:

1. TRC analysis of comparative wettability of sulphur with several types of coal;
2. Agrico's Field Data on prill moisture loss in storage pile contradicting the recent moisture information submitted by Agrico;
3. Agrico's Field Data on prill moisture loss during railroad transport of prilled sulphur;

Mr. Steve Smallwood
August 8, 1983
Page 2

4. Memorandum of technical observations of prilled sulphur being offloaded at Leith, Scotland;

5. Law Engineering technical memorandum and analysis of prilled sulphur being unloaded at Leith, Scotland (moisture, fines, and silt content);

6. Freeport's technical analysis of current South Pierce permit application;

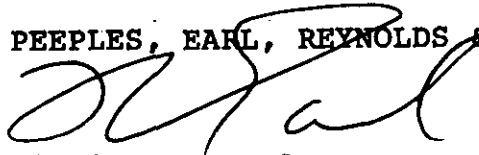
Freeport believes the subject solid sulphur application still fails to provide reasonable assurance because much of the information submitted by Agrico directly contradicts prior submissions and assertions by Agrico with regard to necessary moisture levels, moisture retention properties of prilled sulphur, applicable emission factors, and sources of emissions at the proposed facility. Also, Agrico has failed to provide reasonable assurance that they in fact can maintain necessary moisture levels in prilled sulphur arriving at the facility, and that the newest Agrico emission factor is applicable to enclosed emission points at the South Pierce facility.

The importance of the ongoing rulemaking inquiry requires that no precedents be set by the issuance of a solid sulphur permit before DER's technical investigation is complete. This is particularly important in this case where reasonable assurance does not exist. Please call if you have any questions.

We very much appreciate the hard work of you and your staff on the inquiry.

Very truly yours,

PEEPLES, EARL, REYNOLDS & BLANK, P. A.



William L. Earl
For the Firm

WLE/re
Enclosure

CHARACTERIZATION OF SULFUR PRILL
UNLOADED ONTO TRUCK
FROM CARGO SHIP ATLANTIC CONFIDENCE
(LEIGH, SCOTLAND)
IN ACCORDANCE WITH SUDIC METHODS
LETCO JOB NUMBER G-8657

SUMMARY OF RESULTS

The sampling and testing of a type of Fletcher sulfur prill was conducted in accordance with the standardized procedures published in the Sulphur Development Institute of Canada (SUDIC) 1978 Edition.

The purpose of the work was to characterize a Fletcher type sulfur prill sampled from a truck in the dock area after unloading from the vessel Atlantic Confidence (Leith, Scotland) on April 8, 1983. The characterization tests included:

SUDIC METHOD

TEST PROCEDURE

S1-77	Sampling and mixing.
S3-77	Particle Size Distribution by Washing the Fines prior to Sieving and Calculation of Fines on Dry Weight Basis.
S10-77	Surface Water Content of Sulfur Form by Constant Weight Determinations by Drying at $75 \pm 5^{\circ}\text{C}$ (4.1). Trapped Internal Water Content by Constant Weight Determination at $75 \pm 5^{\circ}\text{C}$ followed by Karl Fisher Titration Procedure (4.2).

The analytical results as summarized below reflect the averages of three determinations excepting for the percent silt (particle sizes less than 200 mesh).

<u>METHOD</u>	<u>TEST FOR</u>	<u>RESULT</u>
S10-77 (4.1)	Surface Water	2.5 Percent
S10-77 (4.2)	Trapped Water	0.08 Percent
S3-77	Particle Size- Fines Less than 50 Mesh	8.5 percent
	Particle Size- Fines Less than 200 Mesh (silt)	5.9 percent

SAMPLING OF A FLETCHER-TYPE SULFUR PRILL
LEITH, SCOTLAND

Dr. Jim Miller reported that all of the sampling of the Fletcher-type prill was done by him. Representative sampling of the sulfur prill was done on April 8, 1983 from the truck after unloading from the vessel Atlantic Confidence (Leith, Scotland).

As sampled from the truck, the prill was taken and stored in five thick wall, wide mouth plastic bottles with screw tops. On April 14 and 15, 1983 at TRC Laboratories in East Hartford, Connecticut, G. V. Aseff reviewed each prill sample, summarized the label information and assigned test numbers to the original sequence sample numbers.

Data concerning the sampling of the sulfur prill are summarized below.

VISUAL OBSERVATIONS OF PRILL SAMPLES BEFORE MIXING

The observations of the five sulfur samples in the containers from the truck showed that the samples appeared to be 100 percent prill form with a relatively large range of particle sizes. The amount of fines appeared to be significant.

When the containers were opened for mixing and quartering, the samples appeared to be 100 percent prill. No slate portions were observed. Individual particles of prill appeared to be somewhat abraded yielding a markedly smooth surface as contrasted to the as-manufactured surface finish of similar forms personally witnessed by the undersigned.

MIXING AND QUARTERING

The five bottles of truck sulfur prill were combined, mixed and quartered as per SUDIC Method S-1 to yield test samples weighing from 350 to 500g. The test samples after quartering were identified as follows:

<u>LOCATION</u>	<u>TEST SAMPLE NUMBERS</u>
Truck	L-T (A) L-T (B) L-T (C)

PREPARATIONS AND TESTS PER SUDIC METHODS

The approximate 350 to 500g samples of mixed and quartered as-received samples were initially weighed and placed in an

oven at $75^{\circ}\text{C} \pm 5^{\circ}\text{C}$ to dry to constant weight to determine surface water as per SUDIC S-10 (4.1).

At constant weight, as recorded for each sample, a small portion was taken (25 to 50g) for Karl Fischer method for trapped water as per SUDIC S-10 (4.2).

The balance of each sample was then washed down to remove fines less than 50 mesh, air dried and then dried at $75^{\circ} \pm 5^{\circ}\text{C}$ to constant weight. Samples were individually sieved per SUDIC S3-77 for particle size determinations. The weight of fines less than 50 mesh was added to the net weight of sieved material.

SUDIC S-10, TITRATIONS - KARL FISCHER

The water equivalent of the Karl Fischer reagent was determined by the use of 15 μL (15 mg) of high purity water and finding the equivalent volume of reagent to reach the end point. Results are shown below:

<u>RUN NO.</u>	<u>AMOUNT OF WATER (15 mg)</u>	<u>EQUIV. KF REAGENT (ml)</u>
1	15 mg	2.10
2	15 mg	2.15
3	15 mg	2.15
AVG	15 mg	2.13

$$\begin{aligned} 2.13 \text{ mlKF} &= 15 \text{ mg H}_2\text{O} \\ 1.00 \text{ mlKF} &= 7.04 \text{ mg H}_2\text{O} \\ \text{KF FACTOR} &= \frac{7.04 \text{ mg H}_2\text{O}}{\text{ml KF}} \end{aligned}$$

The correction factor using the Karl Fischer Reagent for the amount of water in the "anhydrous" methyl alcohol as used in SUDIC S-10 (4.2) was determined. Results are shown below.

<u>RUN NO.</u>	<u>AMT. "ANHY." METHYL ALC. (ml)</u>	<u>AMT. KF REAGENT (ml)</u>
1	25	0.40
2	25	0.50
AVG		0.45

Since 0.45 KF reagent was equivalent to 25 ml "anhydrous" methyl alcohol, then the correction factor of 0.09 KF was calculated as equivalent to 5 ml methyl alcohol; the volume of extract used in each determination for trapped water per SUDIC S-10.

CHARACTERIZATION TESTS

All characterization tests on the prill were done in accordance with applicable SUDIC methods as previously stated.

SURFACE WATER

Mixed and quartered test samples were placed in an oven at 75°C ± 5°C after initial weighing, to dry to constant weight.

The difference between the initial weight and constant weight divided by the constant weight and times 100 gave the percent surface water as per SUDIC S-10 (4.1). Measured values obtained for the percent surface water for sulfur prill are as follows:

<u>SULFUR TEST SAMPLE</u>	<u>PERCENT SURFACE WATER</u>
L-T (A)	2.48
L-T (B)	2.46
L-T (C)	<u>2.41</u>
AVG	2.45 (2.5)

TRAPPED WATER

Trapped water in the sulfur prill was determined by SUDIC S-10 (4.2) method on a 25 to 50g portion taken from the larger lot as mentioned above. The 25 to 50g portion at constant weight was covered with "anhydrous" methyl alcohol and later ground. A 5 ml volume of the extract was titrated with Karl Fischer reagent to an end point. Trapped water content was calculated as per SUDIC S-10 (4.2) as follows:

<u>SULFUR TEST SAMPLE</u>	<u>PERCENT TRAPPED WATER</u>
L-T (A)	0.08
L-T (B)	0.07
L-T (C)	<u>0.08</u>
AVG	0.08

PARTICLE SIZE DISTRIBUTION

The previously mentioned test samples at constant weight were thoroughly washed to remove fines less than 50 mesh, air



July 27, 1983

Page Seven

dried and oven dried to constant weight for a second time. The difference in weights was representative of the fines less than 50 mesh. One of the three samples was passed through a 100 and 200 mesh screen in the stack to collect particles in these size ranges.

After wash down of fines less than 50 mesh and drying to constant weight, sulfur prill test samples were sieved in accordance with SUDIC Method S-3.

Results of the sieve analyses are shown below and detailed in Figures 1P through 3P.

<u>SULFUR TEST SAMPLE</u>	<u>PERCENT FINES < 50 MESH</u>	<u>PERCENT SILT < 200 MESH</u>
L-T (A)	7.9	--
L-T (B)	9.4	5.9
L-T (C)	<u>8.3</u>	<u>--</u>
AVG	8.5	--

SUMMARY

In summary, the approximate 11.5 pounds of Fletcher-type sulfur prill were sampled by Dr. Jim Miller from the truck after unloading from the vessel Atlantic Confidence at Leith, Scotland on April 8, 1983 and brought to TRC, Hartford, Connecticut.

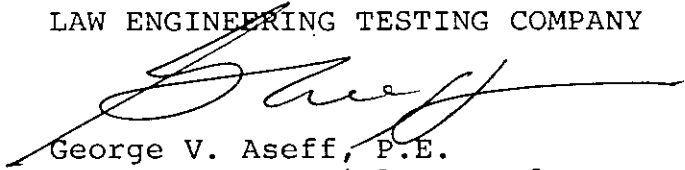
July 27, 1983
Page Eight

The sulfur prill was sampled and characterized by G. V. Aseff, Law Engineering Testing Company on April 14 and 15, 1983, in accordance with SUDIC Methods S1-77, S3-77 and S10-77. Custody of all samples has remained with G. V. Aseff through the entire characterization program. All of the laboratory sampling and testing associated with this prill was conducted either by G. V. Aseff or under his direct supervision.

Representations of the sieved size test data of the sulfur prill were attached as Figures 1P through 3P.

BY:

LAW ENGINEERING TESTING COMPANY


George V. Aseff, P.E.
Corporate Materials Consultant

REVIEWED BY:


Donald E. Dixon, P.E.
Corporate Materials Consultant

GVA:DED/ljh



**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

395 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 1P OF 3P
Fletcher Prill, Truck, Leith, Scotland
JOB NAME _____BY G. V. Aseff DATE 4/26/83CHECKED BY [Signature] DATE 4/28/83**L-T (A) TRUCK FROM VESSEL ATLANTIC
PARTICLE SIZE DISTRIBUTION CONFIDENCE**Sulphur Form: PRILL, FLETCHER TYPE

Producer: _____

Gross Sample Size: 414.4g

Date Formed: _____

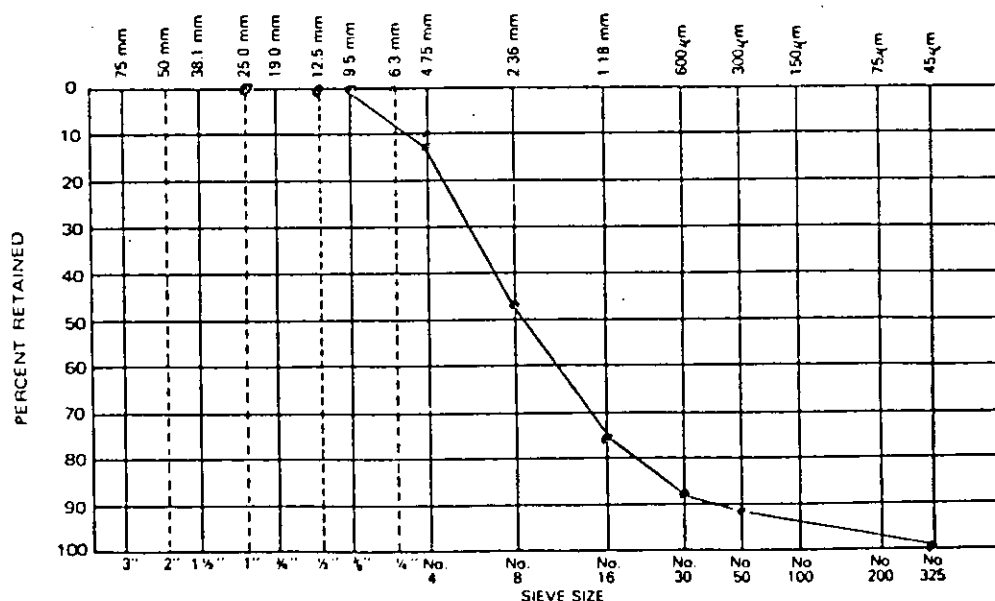
Date Received: 4/7/83Date Tested: 4/15/83Sieve Analysis: Wet ✓

Dry _____

Fines Content (% Minus No. 50): 7.89Coefficient of Uniformity: 6.67

Comments: _____

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	10.33
No. 8 (2.36 mm)	36.70
No. 16 (1.18 mm)	28.40
No. 30 (600 μ m)	12.31
No. 50 (300 μ m)	4.37
Minus No. 50	7.89

*To nearest
0.1%

**LAW ENGINEERING TESTING COMPANY**

Geotechnical, environmental & construction materials consultants

395 PLASTERS AVENUE N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 2P OF 3P
Fletcher Prill, Truck, Leith, Scotland
JOB NAME _____BY G. V. Ageff DATE 4/26/83CHECKED BY [Signature] DATE 4/28/83L-T (B)**PARTICLE SIZE DISTRIBUTION**Sulphur Form: PRILL, FLETCHER TYPE

Producer: _____

Sieve Size

Percent
Retained*Gross Sample Size: 412.5 g

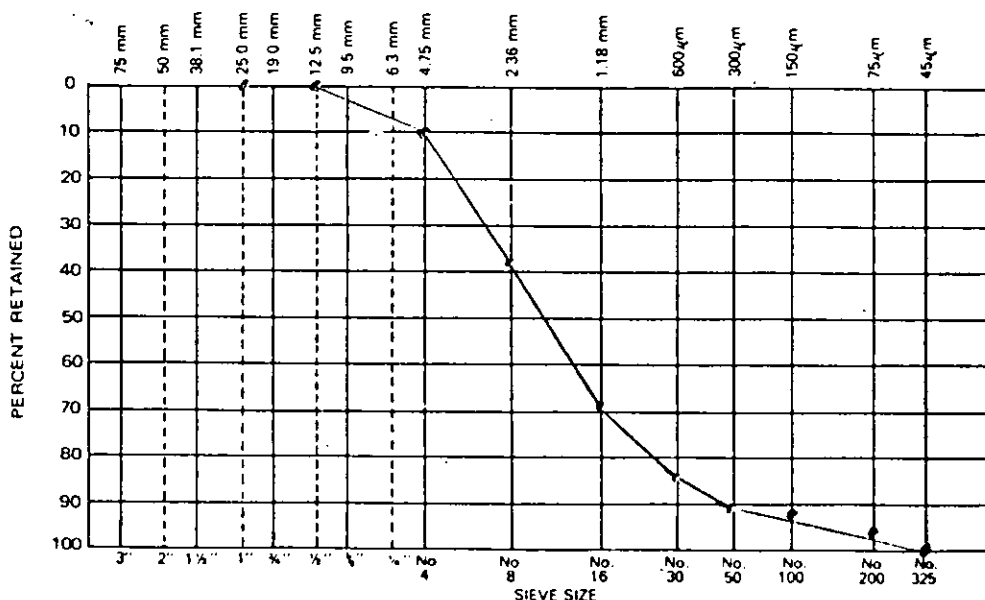
Date Formed: _____

Date Received: 4/7/83Date Tested: 4/15/83Sieve Analysis: Wet ✓

Dry _____

Fines Content (% Minus No. 50): 9.4Coefficient of Uniformity: 6.74Comments: WASHED FINES (%)< 50 > 100 1.31< 100 > 200 1.70< 200 5.94

1"	(25.0 mm)	
3/4"	(19.0 mm)	
1/2"	(12.5 mm)	
3/8"	(9.5 mm)	
No. 4	(4.75 mm)	<u>9.68</u>
No. 8	(2.36 mm)	<u>29.65</u>
No. 16	(1.18 mm)	<u>30.47</u>
No. 30	(600 μ m)	<u>15.61</u>
No. 50	(300 μ m)	<u>6.21</u>
Minus No. 50		<u>9.39</u>

* To nearest
0.1%

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

396 PLASTERS AVENUE N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 3P OF 3P
Fletcher Prill, Truck, Leith, Scotland
JOB NAME _____BY G. V. Asoff DATE 4/26/83CHECKED BY [Signature] DATE 4/28/83L-T(C)**PARTICLE SIZE DISTRIBUTION**Sulphur Form: PRILL, FLETCHER TYPE

Producer: _____

Sieve Size

Percent
Retained*Gross Sample Size: 436.0 g

Date Formed: _____

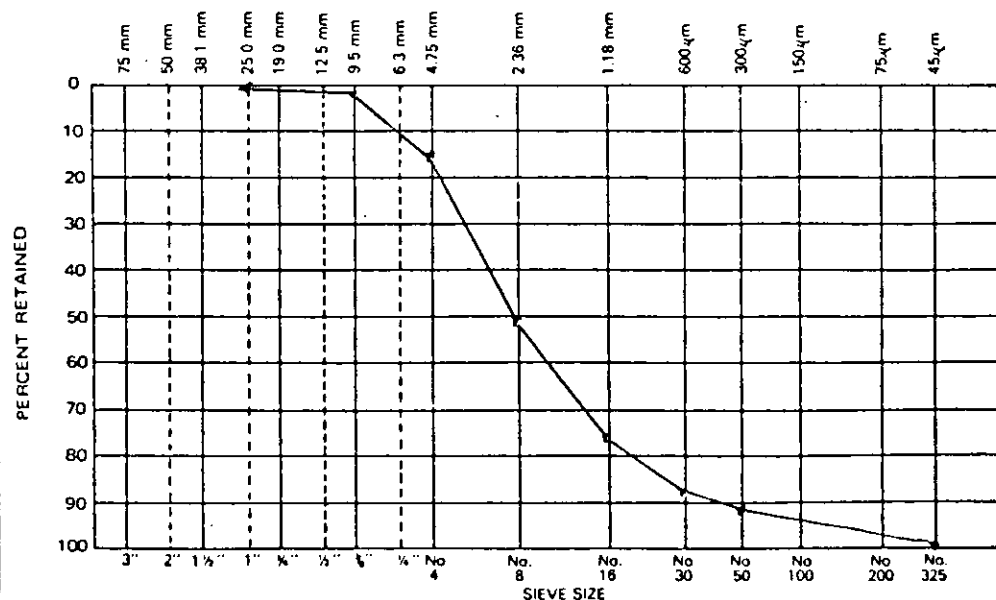
Date Received: 4/7/83Date Tested: 4/15/83Sieve Analysis: Wet ✓

Dry _____

Fines Content (% Minus No. 50): 8.3Coefficient of Uniformity: 7.47

Comments: _____

1"	(25.0 mm)	
3/4"	(19.0 mm)	
1/2"	(12.5 mm)	
3/8"	(9.5 mm)	0.21
No. 4	(4.75 mm)	15.34
No. 8	(2.36 mm)	35.48
No. 16	(1.18 mm)	26.26
No. 30	(600 μ m)	10.92
No. 50	(300 μ m)	3.44
Minus No. 50		8.34

*To nearest
0.1%

PEEPLES, EARL, REYNOLDS & BLANK

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(904) 222-2156

July 27, 1983

REPLY TO:
Miami

Steve Smallwood
Bureau Chief
Air Quality Management
Department of Environmental Regulation
2600 Blair Stone Road
Twin Towers Office Building
Tallahassee, Florida 32301

DER
JUL 29 1983
BAQM

Re: **Agrico South Pierce Air Permit.**
Permit No. 54-S5780; Sulfur Rulemaking Inquiry.

Dear Steve:

This letter is written on behalf of Freeport Sulphur Company and Freeport Land Company. Freeport has now had an opportunity to conduct a preliminary review of all the materials submitted regarding Agrico's South Pierce permit application. The Department's decision on this permit application is of critical importance because a decision to issue this permit prior to conclusion of the ongoing sulphur rulemaking would destroy the integrity and purpose of the rulemaking. We urge that you carefully consider any such decision.

A detailed technical analysis of the issues raised is being prepared. Our initial review, however, reveals several factual gaps in the material that make a thorough evaluation difficult, if not impossible. These gaps in necessary information or assurances include the following:

1. What specific kind of prills are being shipped? Where are the prills being shipped from? What is their handling history?

2. What will the moisture content of the prills be upon arrival of the sulphur at South Pierce and after handling there? How will moisture levels in the prill be monitored and maintained at the South Pierce facility? Since no onsite storage is indicated in the application, what will happen if a shipment of prill is received with inadequate moisture levels?

Steve Smallwood
Bureau Chief
Department of Environmental Regulation
July 27, 1983
Page -2-

3. Where will the dust generated in the screw conveyor go?
4. Is the conveyor system completely enclosed, or will it be only partially enclosed as per Big Bend?
5. How long is the conveyor system and what are its specifications? The sketches submitted with the permit application do not provide this information.
6. Has there been a quantification of particulate emissions generated within the conveyor belt? How much and where will it be emitted? If vented at one end or the other, what emission factor will be applied?
7. Since the surge hopper is now venting through a scrubber, isn't this a particulate emission point rather than a fugitive source? If so, what emission factor applies?
8. Will all prill come to South Pierce by rail or will trucks also be used? If trucks will be used, how will they be unloaded?
9. Will there be any provision for backup storage for the sulphur prills if the facility shuts down for any reason?
10. Will the prills be treated at any point with a wetting agent from production to melting? If so, what wetting agent will be used?
11. Is there an energy budget or other documentation of the availability of adequate waste steam to melt the prill in the amount and at the rate stated in the permit?
12. Is the wastewater from the railcar unloader spray system, the surge hopper scrubber and the melter H₂S scrubber going to an existing cooling pond? What will be the volume and chemical characteristics of the water from each waste stream and what are the characteristics of the pond into which the wastewater will be discharged?

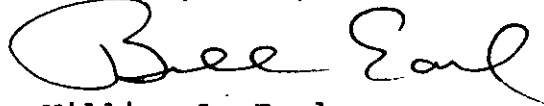
We have not been able to determine the answers to these questions from the record of this permit, and our technical

Steve Smallwood
Bureau Chief
Department of Environmental Regulation
July 27, 1983
Page -3-

people believe such answers are essential if the impacts of this facility are to be fully quantified. Once again, we believe this permit is of crucial importance because its issuance would destroy the very purpose of the ongoing sulphur rulemaking inquiry. As noted, Freeport will be submitting technical data on this permit application in the next week or so. Please call if you or members of your staff have any questions.

Very truly yours,

PEEPLS, EARL, REYNOLDS & BLANK, P.A.



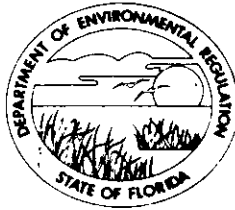
William L. Earl
For the Firm

WLE:btr

cc: Mr. Clair Fancy
Mr. Bill Thomas

STATE OF FLORIDA
DEPARTMENT OF ENVIRONMENTAL REGULATION

TWIN TOWERS OFFICE BUILDING
2600 BLAIR STONE ROAD
TALLAHASSEE, FLORIDA 32301-8241



BOB GRAHAM
GOVERNOR

VICTORIA J. TSCHINKEL
SECRETARY

June 13, 1983

Mr. Don R. Morrow
General Manager
Agrico Mining Company
Post Office Box 1110
Mulberry, Florida 33860

Dear Don:


The additional information which you submitted on May 13, 1983, is appreciated. As we have previously discussed, this new information does not constitute a reapplication and will not trigger a separate 90-day time period for processing, but will be considered along with other materials submitted by Agrico as a part of the original permit application.

Please call me if you have any questions.

Sincerely,

Steve Smallwood
Steve Smallwood, P.E.
Chief
Bureau of Air Quality
Management

SS/Wtb



UNIVERSITY OF FLORIDA
DEPARTMENT OF
ENVIRONMENTAL ENGINEERING SCIENCES
A.P. BLACK HALL
GAINESVILLE, FLORIDA 32611
PHONE (904) 392-0834

June 3, 1983

Mr. Steve Smallwood, P.E.
Chief, Bureau of Air Quality Management
Department of Environmental Regulation
State of Florida
2600 Blairstone Road
Tallahassee, Florida 32301

Subject: Response to questions raised regarding Agrico's
South Pierce Permit Application

Dear Steve:

I have prepared the following response to your March 17, 1983 letter which outlined several questions and items of information which relate to the Agrico Chemical Co.'s South Pierce permit application. I will reply to these items in order.

Your first topic titled "REQUESTED INFORMATION" lists nine items which I shall first state and then respond to:

1) A description of the drop test procedure and a response to an apparatus analysis performed by a Mr. Douglas Anderson of Texasgulf was requested.

A description of the test procedure was provided in the report Addition #1, dated February 28, 1983.

An analysis of the dust measurement apparatus and calculation of particle size collected was made by a Mr. Douglas Anderson of Texasgulf Chemical Company. In performing his analysis, Mr. Anderson was obviously unaware of the rather universal use (especially within EPA) of particle aerodynamic diameter or of its definition. This would have avoided the unnecessary calculations showing that particles of different density settle at different velocities. If one is analyzing work of others he should be

familiar with the standard terminology. Mr. Anderson states in his analysis that the air entering the chamber "occupies a negligible area when it is descending" but when it is ascending it is "distributed uniformly across the cross sectional area". Only by this assumption was he able to calculate a 41 μm maximum particle size to be collected. If he would have assumed air flowing down one side of the chamber and up the other side (or half area) the calculated particle size collected would have been 58 μm (which is essentially what I previously stated). Anyone who has operated Hi-Vol samplers in the field has, on occasion found grains of sand ($\sim 1000 \mu\text{m}$) on the filter surface. This does not demonstrate that 1000 μm size particles are normally collected by the Hi-Vol sampler (he referred to finding a 94 to 131 μm particle on a filter).

In responding to what size particle a Hi-Vol sampler does collect I find it informative to refer to the article by J.G. Watson, J.C. Chow, J.J. Shaw and T.G. Pace, published in the Journal of the Air Pollution Control Association, Vol. 33, No. 2, Feb. 1983. On page 114, data is presented which shows the high volume air sampler to have a 50% collection efficiency for a 25 μm particle at a 24 km/hr wind speed and for a 32 μm particle at an 8 km/hr wind speed. These data are from actual wind tunnel calibration tests done by others under contract to EPA. Further Hi-Vol sampling effectiveness data is presented on page 387 of the article by J.B. Wedding, A.R. McFarland and J.E. Cermak titled "Large Particle Collection Characteristics of Ambient Aerosol Samplers", and published in Environmental Science & Technology, Vol. 11, No. 4, April 1977.

The reference cited by Anderson as 40 CFR 50, Appendix B, should also be reviewed. A general statement is made that "air is drawn into a covered housing and through a filter by means of a high flow rate blower at a flow rate that allows suspended particles having diameters of less than 100 μm (stokes equivalent diameter) to pass to the filter surface". Reference is then made in 40 CFR 50 to an article by C.D. Robson and K.E. Foster titled "Evaluation of Air Particulate Sampling Equipment", published in the American Industrial Hygiene Association Journal, Vol. 23, No. 5, P 404, Sept.-Oct. 1962. This article should also be carefully read because it does not present Hi-Vol sampling efficiency data and only makes the general statement that "the diameter of the particles collected is in the 100 to 0.1-micron range". This is the statement copied into 40 CFR 50, App. B and cited by Anderson. It is unfortunate that the Code of Federal Regulations does not provide a better discussion of the Hi-Vol sampler and what it collects.

From the above referenced articles and the analysis by Anderson it should be apparent that the dust chamber will collect particles of about the same size as the standard Hi-Vol Sampler.

2) Emission factor measurements for a 10 foot drop was requested (as you stated in your March 17th letter, many of

these items had been requested at a January meeting and the information had since been provided).

These data were provided in the February 1, 1983 report. The 10 foot drop tests (tests #73, 74, 75 and 76) indicate the dust emission rate is about twice that for the standard 5 foot drop test. It would seem reasonable to assume a linear relationship between freefall height and dust emission rate over a limited height range. Once the product reaches freefall velocity, further height would not increase dust emission. These data would suggest an emission factor of 0.0016 #/Ton per foot of fall (at ~4% moisture).

3) Emission factor measurements for a drop onto a 45 degree angle plate were requested.

Data for the dust emission rate resulting from a 5 foot drop onto an angle plate at 0° (flat), 15°, 30° and 45° were obtained and presented in the February 28, 1983, Addition #1 report. A slight, but insignificant, emission factor decrease was observed with increasing angle. The particles kinetic energy upon impact is the same for all angles. It appears that the impact velocity is the important parameter and determines the product breakup and the production rate of dust. This was further demonstrated in a second series of tests in which repetitive tests were conducted by dropping samples onto a previously dropped material base. This previously dropped material built up as a pile (at an angle) and could be considered a similar test. Again, a slight but insignificant decrease in dust emission rate resulted. These data are plotted as Figures 4 and 5 of the February 28, 1983 report.

4) Conduct repeated drops of a product sample to determine if progressive dust generation results; that is, an increase, decrease or constant generation rate with additional handling.

Data in Table I of the February 1, 1983 report provides information on this question. Tests #1 through #3 and #4 through #6 clearly show a significant decrease in dust emission rate with additional drops. Tests #7 through #10 and #11 through #14 show the decrease in dust emission after 10, 20 and 40 tumbles. Each tumble represents a 5 foot drop in a metal pipe. All of these tests (#1 through #14) were for a prilled sulfur with an approximate 4% moisture content. Although additional surface area is created on every drop, the moisture binds the smaller particles to the courser material. Fine dust emitted on a previous drop is no longer available for emission. Therefore, only the recently formed fines can be released, but they are effectively tied onto the larger particle by the presence of the moisture.

The effect and importance of moisture on dust emissions can clearly be seen in Figures 1 and 2 of the February 1, 1983 report. In Figure 1 an outside stored sample was retested after weekly exposures. The dust emission factor increased and decreased with product moisture content (resulting from rain fall). The indoors stored sample showed

a dramatic increase in dust generation with decreasing moisture. After the product had dried to a low moisture value the dust generation rate remained approximately constant.

5) Determine the wettability of prilled sulfur.

Before answering this question, let me state that while the wettability of sulfur is important to the rule inquiry and a permit application for a storage facility like Big Bend, it is inapplicable to South Pierce. The prilled sulfur at this site will be processed and melted as soon as it arrives. The material will not have an opportunity to dry out, much less be rewetted. Consequently, reasonable assurances can be provided by a simple permit proviso prohibiting the unloading of dry sulfur.

As I was unaware of any quantitative and meaningful measure of wettability (appropriate to this question), my answer must be somewhat indirect. It should first be emphasized that the sulfur prill being tested is formed in water and starts out with an approximate 5% moisture content (any additional water will drain off). The concern is then whether the product will remain wet or, if it is allowed to dry for any reason, if it can be re-wet to a desired 3% to 5% moisture level.

Data plotted in Figure 1 of the February 1, 1983 report clearly shows that natural rain fall did effectively wet the previously naturally dried sulfur prill and, when re-wet, the dust emission rate did drop to the original wet product value.

Water has been sprayed onto sulfur prill samples in the laboratory. If the sulfur sample is wet the water is rapidly taken up by the prill. If the sulfur sample has been dried, the water requires some time (several minutes) to wet the prill. The as-received samples P-1, P-2, P-3 and P-4 of wet formed PVC prill were allowed to dry from about 4% moisture to about 1.5% moisture. Dust emission increased from about 0.008 to 0.07 #/Ton. A light spray of water reduced the dust emission to about 0.02. Saturation with water (~6% moisture) reduced the dust emission to 0.0035 #/Ton.

My testing did not quantitate the wetting ability of sulfur. It is possible to demonstrate that wet prill can easily be maintained in a wet condition by the simple application of a water spray. It was also possible to demonstrate that dry sulfur can be re-wet and that once re-wet the dust emission factor assumes its initial low value. These data are all tabulated in the February 1, 1983 report.

Orlando Laboratories, Inc. presented results of a re-wettability test for sulfur prill in their report dated April 22, 1983. They refer to using the Supplemental Water Behavior Test #1 specified in Sampling and Testing Sulfur Forms (SUDIC). I have not evaluated the meaning or usefulness of these data. They do show that sulfur can be re-wet however.

6) Repeat the standard dust emission factor test at least 30 times.

The February 1, 1983 report tabulates the results of 80 tests. The February 28, 1983 Addition #1 report tabulates data from 25 additional tests. All data are plotted in Figure 6 showing the effect of moisture content on product dustiness. In almost all cases replica tests were run providing data for statistical analysis. Test repeatability is excellent.

7) Provide standard sieve analysis on the sub 300 μ m (50 mesh) fraction of the sulfur prill both before and after a drop test. Provide several size fractions with the smallest cut at about 75 μ m (200 mesh).

I have been reluctant to perform sieve analysis tests because I do not believe the data obtained are useful. Furthermore, if these data are improperly used it will lead to incorrect conclusions, as has been done in the past.

Item 5 in the February 28, 1983, Addition #1 report discusses the difficulty of performing a sieve analysis of wet sulfur prill. A 100 gram sample of 3.5% moisture sulfur prill was hand sieved with essentially none of the material passing a 40 mesh sieve (420 μ m opening). A second sample of this material was oven dried to 0.1% moisture and hand sieved, with the results tabulated in the report. About 6% of the material passed through a 40 mesh sieve and about 1% passed through a 200 mesh sieve. When re-wet this same sample failed to pass through a 40 mesh sieve.

My objection to the sieve test is based on the inability of the sieve test procedure to quantitate the effect product moisture has on product dustiness. A clean dry formed prill in a dry condition may have very few fines by the sieve test but may have a fairly high emission factor measured by the dust test.

Wet formed prill will break down from repeated handling and the sieve test may show this. However, if the product remains "wet" the dust emission rate remains very low. Therefore, I believe a measure of the dust emission factor is more meaningful and useful in predicting product dustiness.

Orlando Laboratories, Inc. in their April 22, 1983 report present results of sieve sizing of both PVC and Coastal wet prill sulfur at various moisture levels. Determinations were made using a Gilson sieve tester fitted with screens of 20, 40, 70, 140 and 200 mesh. Standard 8 inch sieves were used to machine sieve 100 gram product samples for an 8 minute time period. About 16 complete sieve analysis were provided at various product moisture over the range from 4.8% down to 0.3%. Two sieve analysis were also provided for Gulf dry formed sulfur prill at moistures listed as 0.18% and 0.014%. These data appear excellent and may be quite useful to you in that they show the fraction of fines increase as the product moisture decreases.

The concern I have is in my inability to use these data to predict product dustiness. The Gulf dry formed prill has a very low percentage of fines and my dust tests show the

dust generation rate is reasonably low (0.04 #/Ton on test #41, 42 and 43). The PVC and Coastal wet formed prill have a much higher percentage of fines when sieve tested dry, as shown in the Orlando Laboratories, Inc. report. A dust generation test on the aged, air dried wet prill showed a very high dust emission rate (0.4 %/Ton at 0.04% moisture from tests #55 and 56). When the Gulf dry prill and the PVC wet prill were both saturated with water to about 6% moisture both had a dust emission factor of about 0.0035 #/Ton (test #65, 66, 67 and 68). It appears to me that the dust emission factor is primarily dependent on moisture level at high moisture (perhaps 3% to 6% moisture) but is dependent on both moisture level and sieve size distribution (or % fines) at low moisture (~2% moisture). However, at both high and low moisture a definite conclusion can be reached. At high moisture level (~4%) all sulfur prill products tested had very low dust emission factors. At low moisture levels (<0.1%) the dust emission factor is always quite high. Product formation method, handling history and sieve analysis are all of secondary importance. These data are more difficult to obtain and document and are of less relevance to a determination of product dustiness. Product moisture is fairly easy to determine and is very relevant to product dustiness.

8) Provide test data at various product moisture contents. Also provide sieve analysis at various product moistures.

Dust emission factor data have been obtained at various moistures, under various test conditions and for various sulfur prills. These data are tabulated in Table 1 and plotted in Figure 3 of the February 1, 1983 report. An additional plot of all data is presented as Figure 6 of the February 28, 1983 report Addition #1.

Orlando laboratories, Inc. has presented sieve sizing data for various moisture level prill in their April 22, 1983 report. These data were discussed in the previous section.

9) Show that what was collected on the Hi-Vol filter represented essentially all of the dust that became airborne as a result of the drop.

This question was addressed in the February 28, 1983 Additional #1 report. Background concentrations were first determined in tests #81, 82, 83, 93 and 111. The equivalent background converted to 0.0002 #/Ton. This value should be subtracted from the dust emission factor determinations but was not, because it is negligible.

An analysis was then conducted on dust deposited on the test chamber walls. It was not possible to clean the chamber after only one test and conduct a sieve analysis. Therefore, after the 25 tests described in the Addition #1 report were completed the dust chamber was very carefully cleaned to determine the amount and size distribution of material on the chamber walls. These data are presented and described in the February 28 report. Of the material collected on the walls only 4.2% was less than 200 mesh (77 μ m). This would be

equivalent to about 0.002 #/Ton of fines. As the Hi-Vol does not collect all particles up to 77 μ m you cannot add all of this sieve fraction onto the dust chamber filter catch. These results indicate most of the airborne fines are collected by the Hi-Vol filter. It is not possible to quantitate this accurately from the data obtained. As a worse case one could add on the 0.002 #/Ton to the measured emission factor. This is not correct however, because some of these fines are attached to the larger particles collected on the chamber walls.

Topic Titled: EVALUATION OF INFORMATION SUBMITTED

Bone-dry Prill Emission Factors Section

In preparing answers to the 9 questions asked in the "REQUESTED INFORMATION" section of your letter I have provided answers or partial answers to some of the concerns expressed in this section. When helpful, I will refer back to these responses.

Before answering these questions, I need to advise you that many of your concerns while appropriate to the rule inquiry and Big Bend are inapplicable to South Pierce. For example, on page 5, paragraph 2 you ask whether wind stripping is an important factor. As you know, the material at South Pierce will not be subjected to the wind. Also, on page 6, paragraph 4 you ask "What assurances does the Department have that the prill will be kept in a moist state?" This question is irrelevant to this particular permit application, since only moist prill will be unloaded and it will be melted as soon as it arrives.

You first refer to my sieve test of bone dry sulfur prill and the results of that sieve test which show about 1% of the dry material passed the 200 mesh screen (the $< 77 \mu\text{m}$ fraction). This 1% would then convert to the 20 #/Ton figure you used. This 1% value is about three times higher than the approximate 0.3% values presented in the Orlando Laboratories, Inc. report of April 27, 1983.

If all of these silt fines (the name you used to indicate the sub $77 \mu\text{m}$ size fraction) were to become airborne an obvious problem would result. It is therefore reasonable for you to be concerned. On the following page you refer to settleable fines (defined as particles in the approximate $75 \mu\text{m}$ to 250 or $300 \mu\text{m}$ size range) as being an additional 2 or 3% of the bulk sulfur. This again agrees with my bone dry sieve analysis data in the February 28 report. Orlando Laboratories, Inc., show this value varies from about 0% at high moisture (4.65%) to about 3% at low moisture (0.3%). Based upon the low moisture sieve analysis data it is very reasonable to be concerned about settleable particles.

The mass of airborne particulate matter in the atmosphere in the greater than $1 \mu\text{m}$ size range (or ~ 1 to $300 \mu\text{m}$) results primarily from the dry dispersion of solid materials (or the dispersion of salt water over the oceans). Dispersion is an energy consuming process, the more energy provided the greater the quantity of material that can be dispersed and the finer the material (or particle size) produced. Not all materials disperse the same or with the same ease. Properties of the material are very important in determining its tendency to breakup or be dispersed. A truck moving rapidly down a lime rock road produces a large cloud of dust when the road is very dry but produces almost no dust when the road is very wet. The available energy in both cases is the same and is due to the velocity and weight of the truck. The wind serves to transport the formed dust, but is not primarily responsible for its

generation.

The above analogy can also be applied to a material transfer process such as a sulfur unloading operation. The energy for dispersion of the product (dust generation) comes primarily from the products motion. As you correctly inferred, the drop of sulfur from a 10 foot height may be expected to produce twice the dust as a drop from a 5 foot height. My test results appear to verify this. In a like manner, one may conclude that a 5 foot drop onto an angle plate may not be significantly different than a drop onto a flat surface because the energy at impact is the same. If the angle were great (60° or 70°) the energy would not all be lost and a difference would be expected. My test results show a small but unimportant effect as the angle increased from flat (0°) up to 45° . Again, I would expect this because the products energy was about the same in all cases.

Again, what the above is inferring is that the energy for dispersion comes from the products energy and not the wind. The wind serves to move or transport the formed dust. This cannot be true without limit however. Kinetic energy varies as velocity squared. Winds of 1 mph have essentially no energy for dispersion of dust. Winds of 10 mph have 100 times as much energy and can aerosolize certain products under certain conditions. By direct observation, I could see no dust generated by a 10 mph wind from a surface of wet sulfur prill.

I do not expect a 10 mph wind to have a significant effect on the measured dust emission factors for free falling sulfur over the 5 to 10 foot height range that would normally occur in the field. A free fall of only 4 feet will result in a product velocity of approximately 10 mph.

In summary, moisture can bond the sulfur fines to the coarser sulfur particles with sufficient force to prevent any significant release (or dispersion) from a 5 foot fall or from a 10 mph wind. The actual mass of fines released has been quantitatively measured in a reproducible and meaningful test. Product moisture is the most important parameter affecting dust release and should be maintained at about 4% to minimize dust emissions. Emission of settleable fines would not be expected to exceed the emission of suspended fines by more than the sieve fraction ratio. Sieve analysis produced a weight ratio of about 3 for the 77 to 250 μm fraction divided by the less than 77 μm fraction. This results in an insignificant release.

Agrico Chemical Co. will provide water sprays at product transfer points.

The fourth paragraph on page six asks why the department should believe that the actual emissions from moist prill would only be 1/500th or so of the settleable particulate fines content of the dry prill and what assurance the department has that the prill will be kept moist.

Although I have already pointed out that the inquiry is irrelevant to South Pierce, I want to add a few comments or restate my opinion on this matter.

It is difficult to aerosolize a pre-formed powder into its original size distribution, even though it is often very easy to disperse a wettable powder in a liquid. Particle to particle

adhesive forces are quite different in the two fluids (gas vs liquid). Any liquid coating, such as water, applied to a dry powder increase the particle to particle contact area and increases the forces of adhesion. The liquid acts as a glue holding the particle together. The force that a moving air stream can exert on a very small particle (of a few μm diameter) varies as the first power of particle diameter. The force of adhesion of the same small particles to a larger particle varies as the surface contact area or the second power of particle diameter. The net result is that it takes a higher gas velocity (or more energy) to dislodge or aerosolize a smaller particle. This phenomenon is qualitatively well known but is difficult to predict or qualitate for various powders. It is well known and excepted that almost all powders become sticky and difficult to pour when moist.

This discussion hopefully serves to explain why moisture content should and does have a dramatic effect on the dustiness of certain powers or products such as the granular sulfur prill. With a low energy break up process, such as a 5 foot product drop test, it is not unexpected to find that few micrometer size particles are dislodged from the moist millimeter size particles.

Moist Prill Emission Factor Section

The first statement asks what effect moisture has on various kinds of prilled or pelletized sulfur. Comments were made in the previous section that both the dry formed polish prill from Gulf (samples G-1 and G-2, tests #65 and #66) and the wet formed prill from P.V. Commodity Systems (samples P-1 and P-2, test #67 and #68) when wet to a 6.5% moisture content have the same dust emission factor (~ 0.0035 #/Ton). Another wet formed prill obtained from Coastal Sulphur (samples #5 and #6) were wet by natural rainfall, after a 4 week outdoor storage, to a moisture content of about 5%; these samples had a dust emission factor of about 0.006 #/Ton (test #35 and #36). Dry formed prill (samples G-1 and G-2) and wet formed prill (samples P-1 and P-2) were both sprayed with water to raise the moisture content to 1.5%. The wet formed prill emission factor of 0.019 was only slightly lower than the dry formed prill factor of 0.026 (tests #59 through #62). Samples of wet formed Coastal Sulfur also produced emission factor measurement of about 0.02 for moisture in the 1.5 to 2.4% moisture range (tests #25, 26, 31, 32, 33 & 34). Moisture content certainly affected these three sulfur products in a very similar manner.

I do not disagree with you that sieve size distribution data would be interesting and would indicate the effect various moisture levels have on binding the fine particle to the course partially. The previously referenced Orlando Laboratories, Inc.'s report of April 22, 1983 presents this exact data for PVC and Coastal sulfur samples. Two distributions are also presented for the Gulf dry formed prill. All of these data are for undropped prill.

Although it would be interesting to review sieve size data for dropped sulfur, the cost and time required to obtain these data outweigh any benefits. Data on sulfur dropped 20 times are not representative in that a sulfur sample will not be handled (dropped) that much in transfer from original manufacturer (prill formation) to the final use (prill melting). Data have been provided and discussed on the dustiness of both dry and moist prill dropped several times (Figures 1 and 2 and Table I of the February 1, 1983 report).

You refer to my demonstration in your office and my discussion indicating that the dust emission resulting from a product dropped several times is no greater than for a product dropped one time (on a per-drop basis). I believe this is very clearly demonstrated by referred to data in Figures 1 and 2 and other data in Table I. I also discussed this in the last section. Having conducted over one hundred tests, many of which involved more than one drop of a product sample, I believe this has been adequately demonstrated. Therefore, I believe additional data are unnecessary to provide reasonable assurance for this permit, even though it may be of basic interest.

I believe that a product moisture of 3% to 5% should be required because it can be achieved and can be maintained and will produce the lowest possible dust emission factor. I

personally see no need or reason for you to require less than the best possible, even if it is lower than the allowable emission rate.

Waste heat will be used to dry the prill before melting, making the cost of this drying a minor cost factor. I also believe this is irrelevant to the environmental issue.

While the frequency and methodology for a storage pile spray system is important to provide assurances that the moisture content will be maintained in a facility like Big Bend (where the material is stored in open piles) it is unnecessary for South Pierce (where sulfur will not be stored).

Conclusions Section

I believe that water sprays should be used at all transfer points to maintain a product moisture between 3% and 5%. The exact quantity of water needed can (and must) be determined after the system is installed and operating. Product moisture levels greater than about 6% can not be held by the product and would produce no added benefit.

In every operation good house keeping must be observed. Poor or careless procedures can and will cause an environmentally undesirable situation. A poor situation would result even though the dust emission rate was low. There is a very significant economic incentive for Agrico to provide very good, rather than only adequate, house keeping procedures.

In conclusion, I believe the dust emission factor data I have provided is as accurate as existing technology is able to determine. I believe these numbers can be used with greater confidence than can other presently obtainable numbers.

Settleable particles and suspended particles are both parts of the same aerosol distribution generated when handling a bulk granular product. From fundamental considerations they both must vary together. Handling of a moist product will minimize both.

After a careful review of all dust emission factor data obtained for sulfur prill, I recommend use of a $4 \pm 1\%$ moisture content prill and suggest using a high and very conservative emission factor of 0.002 #/Ton/foot of fall (for suspended dust in the approximate size range from 1 μm up to about 30 to 60 μm aerodynamic diameter).

Settleable dust (associated with particles in the general size range from 30 to 300 μm diameter) will vary significantly with wind speed. At zero wind there will be no settleable dust because it will all fall to the ground with the product.

In closing this response, I want to thank you and your staff for your personal interest in this project. I am confident you have acquired a good understanding of the problem and I appreciate the careful and conservative consideration you are giving to it. It is always easier to remain quiet than to risk making a mistake by speaking out. I have spoken out and must therefore take some risk. My initial involvement in emission factor determination was in January of 1979. Since then I have gained great confidence in this method of emission factor estimation and I hope my data and explanation will help you gain a similar understanding and confidence. After

approval and construction of this sulfur handling facility, I look forward to being involved in varifying the emission factor prediction. Once this emission prediction technique is verified it should prove very useful to both industry and air pollution agencies for making decisions on other facilities.

After you and your staff have had time to digest this response I will be available to discuss it and answer any questions.

Sincerely,

A handwritten signature in cursive script that reads "Dale A. Lundgren". The signature is written in dark ink and is positioned below the word "Sincerely,".

Dale A. Lundgren, Ph.D., P.E.
Professor, Env. Eng. Sci.

DAL/sr

ONE OF THE WILLIAMS COMPANIES
AGRICO MINING COMPANY
A DIVISION OF AGRICO CHEMICAL COMPANY
P. O. BOX 1110
MULBERRY, FLORIDA 33560
813 428-1431

DON R. MORROW
GENERAL MANAGER

May 13, 1983

Mr. Steve Smallwood
Bureau of Air Quality Management
Florida Department of Environmental Regulation
2600 Blair Stone Road
Tallahassee, FL 32301

Dear Steve:

In reference to Agrico's Application AC53-55780 pertaining to an air permit for wet prilled sulfur handling at our South Pierce plant, I respectfully submit the following modifications.

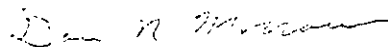
Although Agrico feels that we demonstrated that wet prilled sulfur can be handled at our South Pierce plant without any environmental problems, we realize that you still have some concern about dust emission. Agrico's application did not include any dust suppression equipment due to the low dust emission factor determined by Dr. Lundgren and the precedents set by others who handle this material in our country without suppression devices.

There are only two areas where dust could occur in our design if the material were, in fact, dusty. They are the dumping hopper and the discharge into the 100-ton storage hopper. The remaining facility is a closed system.

As an extra safeguard Agrico is willing to install a mist spray system in the dumping hopper and a wet scrubber in the head house of the 100-ton storage hopper. Attached is our original conceptual diagram (Supplement 6) and a revised diagram dated May 11, 1983, which illustrates the additional dust suppression and collection equipment.

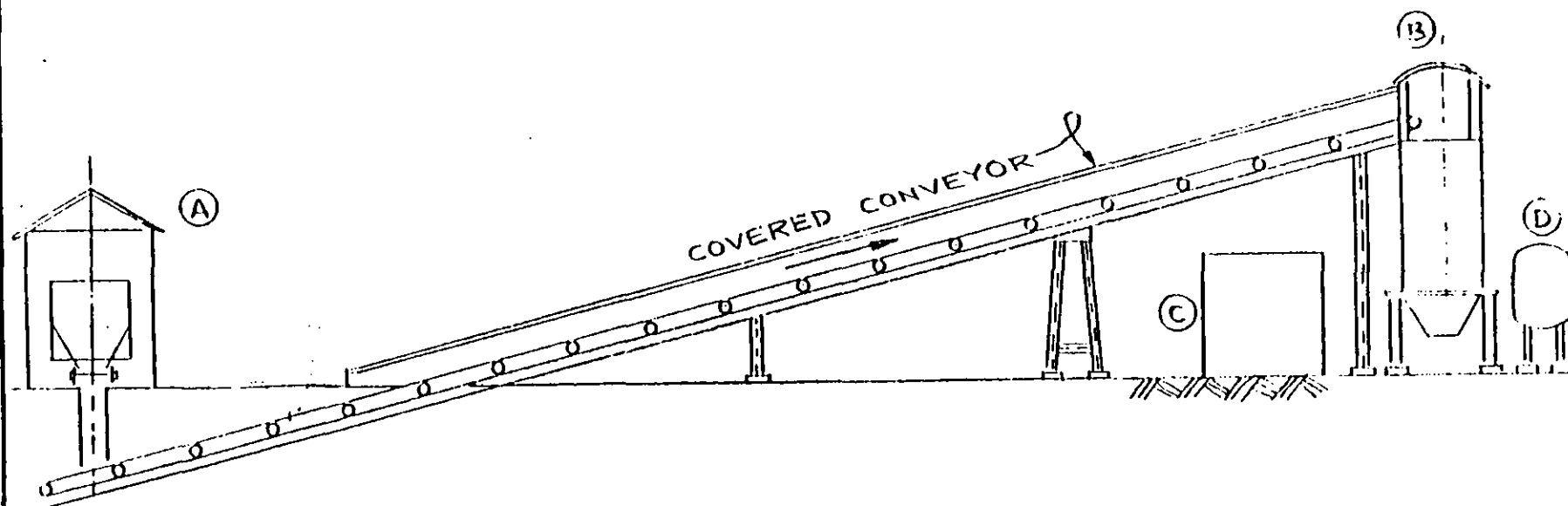
At some time in the future, when Agrico has accumulated substantial operating experience with the wet prill product and has proved through abundant scientific testing that these dust suppression and collection facilities are not needed, we will want to petition you for their removal.

Very truly yours,



Don R. Morrow

DRM:GNC
Attachments



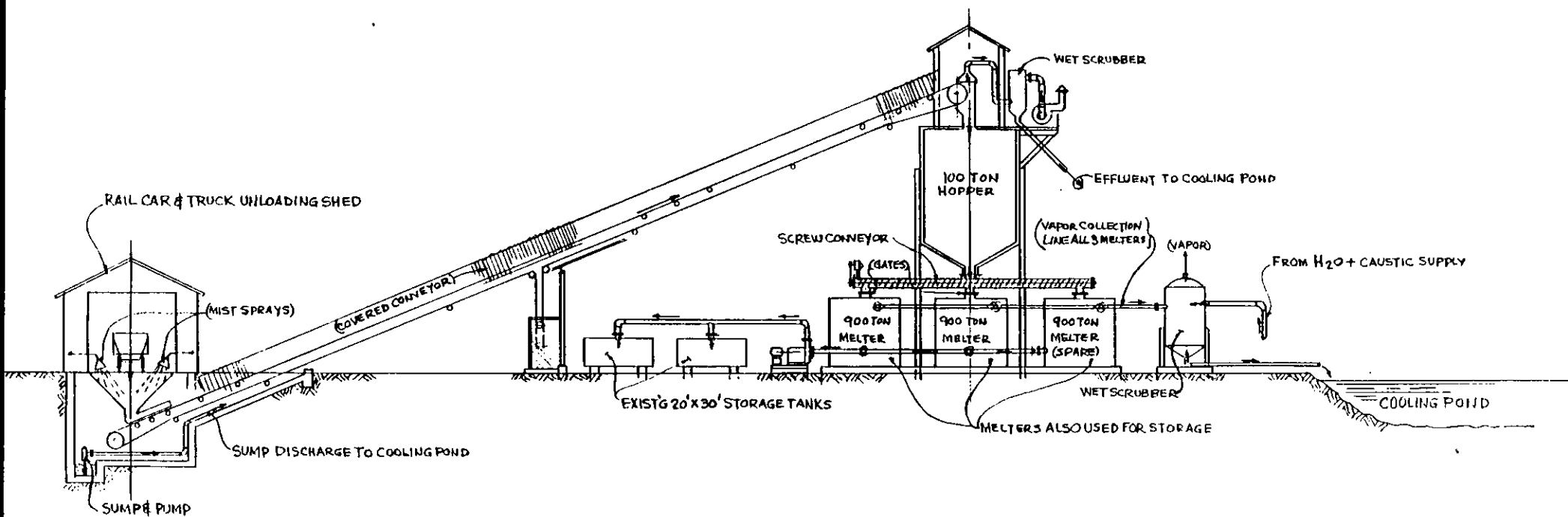
- (A) RAILCAR AND TRUCK UNLOADING SHED
- (B) COVERED STORAGE HOPPER
- (C) COVERED MELTERS
- (D) H₂S SCRUBBER

SUPPLEMENT #6



BARNARD AND BURN ENGINEERS
& CONSTRUCTORS, INC.

TITLE FLOW DIAGRAM
PRILLED SULPHUR HANDLING
CLIENT AGRICO - SOUTH PIERCE



FLOW DIAGRAM FOR PRILLED SULPHUR HANDLING
 AGRICO-SOUTH PIERCE CHEM WORKS
 5-11-83

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May 11, 1983

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REPLY TO:

Miami

Mr. Steve Smallwood
Chief, Bureau of Air Quality Management
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32301

Re: Sulphur Rulemaking; Radio Interviews

MAY 16 1983


Dear Steve:

BAQM

I thought you would be interested in the enclosed transcripts of radio broadcasts which were aired in the Tampa area on April 8, 1983.

Sincerely,

PEEPLS, EARL, REYNOLDS &
BLANK, P.A.


Paul H. Amundsen
For the Firm

PHA:nir

Enclosures

cc: Clair Fancy
John Svec

TRANSCRIPT OF RADIO PROGRAM

WSUF-FM - April 8, 1983

MR. KINNEY: "Based on previous experiences in the Tampa Bay area, any kind of solid form of sulphur is not popular with the environmental people because of the problem with dusting that they had in previous years. However, now the new technology is suggesting that because of this new form of sulphur -- that is the prill form -- this dusting problem won't exist to the extent that it did before."

REPORTER: The Tampa Bay market represents approximately 80 percent of the total world market for sulphur. The demand is so great here because the phosphate industry uses sulphur to make sulphuric acid, which is needed to make phosphate fertilizer. Currently, sulphur is imported into the area in a liquid form and has been for several years because of environmental reasons. Now some twenty years later, a local phosphate company wants the rules changed on how sulphur is handled in Florida. Agrico Mining Company of Polk County has applied to the Department of Environmental Regulation for a permit to import solid sulphur in a prilled form to their facility in Tampa. Agrico has been trying to get their permit approved for five years. Environmental regulators in Tallahassee say they'll make a decision some time next year. Manager of Environmental Control for Agrico, Harold Long, says that the incentive behind the proposal was one of economics.

MR. LONG: "Agrico became involved in the sulphur project simply as an economical matter. Sulphur prices have gone up over the period of years. We use a considerable amount of sulphur, and therefore, our involvement is one of economics."

REPORTER: "Can you explain why it's more economical?"

MR. LONG: "The wet prill form is cheaper simply because it's a by-product of either the sour gas operations in Canada or the petroleum industry in the United States. It is not the main product. It is a by-product, and they recover it, and therefore, it's cheaper than your Frasch molten sulphur."

REPORTER: Long says that over a period of time, the change from liquid to solid sulphur would affect the consumer in lower fertilizer costs.

MR. LONG: "From the standpoint of economics, as I say, it would eventually show up as a reduced cost to the consumers. From the standpoint of employment, we're not talking too much of too many more employees. It would mean some greater tariff to the Port of Tampa for ships entering, offloading. We would not impact the road system, because it would be a straightout transfer, and transportation, where we're now receiving molten sulphur, we would just be replacing that with the solid wet prill."

MR. EARL: "It's been called the invisible industry right now -- the liquid industry -- because you don't see it. Tampa is the

world's largest port of importation in the world for sulphur and no one knows about it. But when you start having these piles all over Tampa Bay and you start having the dust on the water, people will know about it and I think the very real environmental problems will become apparent."

REPORTER: Bill Earl is an environmental attorney from Miami who represents Freeport Chemical, which supplies sulphur to phosphate companies. Freeport strongly disagrees with the conversion and sees no economical advantages. Earl explains that the existing liquid sulphur industry thinks it's pretty unfair that the new people won't have to invest in pollution control technology that, for instance, Freeport has invested in.

MR. EARL: "If they can bring it in cheaper, they can bring it in cheaper because they don't have to invest in the pollution control equipment that our clients have. The tankers, for example, the tank trucks, the tank farms. You just have to charter yourself any old tramp steamer and load it with this solid sulphur up in Canada and bring it down and dump it on the dock in Tampa. You don't have to spend the millions and millions of dollars to build the tankers and materials you need to bring in liquid sulphur."

REPORTER: "And that's the real issue. So it's not -- maybe, it's partially an environmental issue as well as an economical one. Is it one more than the other?"

MR. EARL: "Our client has a very real and economic interest. Our client has always said that we have an economic interest because we've invested in this technology which we think is cleaner. With that aside, there are some valid, environmental concerns here which is one of the reasons why the Florida Department of Environmental Regulation is right now undertaking rulemaking."

REPORTER: Earl also argues that solid sulphur is too dangerous to work with in regards to its potential damage to the environment, whereas there is little to no risk handling the liquid sulphur.

MR. EARL: "Even when it's brought in in solid form, they have to remelt it over in the fertilizer plants, and they again have storage tanks and storage areas where they have liquid sulphur. So on balance, there's very little problem with liquid sulphur. There are some minor amounts of gas generated, but you don't have the dust. You don't have the risk of explosion which you have with solid sulphur dust. There is some potential explosivity of liquid sulphur, but it's nothing like the problems caused by fires and explosions in solid sulphur. Sulphur dust is something like 20 times more explosive than coal dust. It's highly explosive, which is generally why you have open storage piles in a lot of areas. You don't see too many enclosed facilities."

REPORTER: But Harold Long from Agrico disagrees. He says that his company has spent time and money proving that wet prilled

sulphur is environmentally sound and wouldn't make such a proposal unless it was safe.

MR. LONG: "In all of my viewing of wet prill in Canada and in Houston, Texas and other places that I've seen it handled, manufactured, stored, shipped, I have never seen an environmental problem with it. No dusting. So we at Agrico didn't envision this large of a problem that we're facing now."

REPORTER: "How are you coping with the problem, the environmental issue?"

MR. LONG: "Just recently, I made a trip to Canada and to Houston, Texas, and we obtained samples of various prills -- wet formed prills and dry formed prills. We sent those samples to Dr. Dale Lundgren at the University of Florida that is doing some emission test work for us. We provided samples to Dr. John Fitzgerald at the University of Georgia for bacteriological work. We have provided also the samples to the Orlando laboratories for further testing. So, we have made a very concerted effort to supply the State, DER, and the Hillsborough County environmental people with as much data as we can so that they can make a good, accurate judgment on our permit application."

REPORTER: Another approach Agrico has taken to gather factual information involves showing what kind of effect that sulphur has in the real world. Last week Agrico sent four environmental

representatives from Florida to Immingham, England on the North Sea where the importation of prilled sulphur is currently being carried on. Air Quality Chairman of the Hillsborough County Environmental Coalition, Mike Kinney, was one of the observers.

MR. KINNEY: "First of all, we saw the potential for alot of particulate emissions from this handling facility in the way of --in a couple of different areas. One was when the ship was being unloaded by a clam shell digger. It was physically taking the sulphur from the hold in the ship and putting it in a hopper, and the hopper was transferring it to a conveyance system which took the sulphur and put it into a large building where they stored the solid sulphur until they're ready to use it."

REPORTER: And that was bad?

MR. KINNEY: Well, there were areas where there was a lot of dusting, and those areas in particular were near the ship. Any place where the material was being lifted from the holds of the ship to the hopper generally were a source of dusting from the sulphur. The sulphur particles themselves, some of which fell back into the ship, others were picked up by the wind and transported some distances downwind. It was interesting to note that most of the sulphur that was transported downwind remained on the site, on the facility site. And this was just from my own visual inspection. I didn't have any equipment there to sample the air around the site, but from my own visual perspective, I

could see that most of the dust was falling within the facility itself and not being transported to neighboring areas."

REPORTER: But Kinney notes that the dusting could be controlled under different conditions.

MR. KINNEY: "A lot of it had to do with operator technique. A lot of human error was involved. For instance, the men that were operating the clam shells, that were removing the sulphur from the ship and putting it into the hopper, some of them were real good at it and others weren't good at all. It depended on the technique that they were using. And it was interesting to note that the owner of the facility had no control over who was operating the clam shells because that was a separate union. And if they were doing a sloppy job, he couldn't go up there and tell them to do it any better, because if he did, that would create some union problems. So, that was a significant problem in this particular site. However, it was something that was totally out of the control of the owner of the facility."

REPORTER: One of the issues surrounding the change to solid sulphur is how to store the substance. Kinney learned that the storage facility at Immingham where the sulphur was kept until it was ready to be used was a large building.

MR. KINNEY: "Rainwater was kept from hitting, from landing on the sulphur pile, and the building kept wind from blowing the

sulphur from the point in which it was deposited to to areas outside of the facility. I was interested to note this because the current proposal here in Tampa is that the sulphur will not be allowed to be covered because there was a fear that if there was a dusting problem, that the facility -- any kind of a building that was containing the sulphur would have the potential for exploding."

REPORTER: "So which is better?"

MR. KINNEY: "I think that if Agrico and other companies are allowed to import solid sulphur and store it on and around Tampa, that it should be covered, because my major concern is the distribution of the sulphur once it's been deposited, once it's been stored here from wind and from rain. I fear that with the amount of rainfall that the Tampa Bay area has, that there's a real good potential for the rain landing on the sulphur pile and transporting the sulphur into the neighboring waterways in the way of water contamination. And I'm also concerned that the wind blowing across the sulphur pile would pick up sulphur and blow it to areas outside of the sulphur importing facility. I feel that if this material was covered with a building, then that would significantly cut down on this particular problem. As far as the explosive nature of the sulphur, it was my observation that with this prilled sulphur there wasn't, in fact, that much dust generated when they were moving the sulphur into the building,

and I don't really think that the explosive characteristic of the sulphur would be a problem in this particular situation."

REPORTER: "So do you think if Agrico modifies their proposal, then it's possible they will get what they want?"

MR. KINNEY: "Well, I think that if they are given permission to do what they want to do, that is, import solid sulphur, then there should be some major modifications made to their permit, one of which is the covering of the sulphur on the site. Another major modification that I would like to see is that they are proposing to transport the sulphur by trucks from the site of Tampa Bay to a site 30 miles away. And in the current permit, they are not required -- in fact, they're required not to cover the trucks with any kind of tarp or any kind of canvas. I fear that if they don't do this, the sulphur will be allowed to escape from the truck and deposited on the roads, and that could be another source of pollution outside of the boundaries of their proposed facility."

REPORTER: The Florida Department of Environmental Regulation is interested in the information gathered by Kinney in Inningham. DER began its own study of the issue last year and has been able to come up with only a few facts and a lot of opinion. Steve Smallwood is Chief of the Bureau of Air Quality Management at DER. He says that the technical investigation should be finished within two months. Where then, a public workshop will be held in

the Tampa area on the subject just to see how the public feels on changing the rules.

MR. SMALLWOOD: "Certainly one of the questions has to be, 'Is it acceptable to allow a of conversion from molten handling technology to solid?' And looking at that issue, you need to make some estimate of what the environmental impacts of one versus the other would be. That requires factual information about emissions. Once you have that information, and you can make a reasonable estimate as to how much pollution may be emitted into the atmosphere from one versus the other, if it's about the same, and I'll just say "if" because that's part of what we're trying to find out -- whether it is or is not, and there's various opinions expressed -- let's say that it is, that one is one type of pollutant and the other is another, then certainly these other factors are things that the State Environmental Regulation Commission can consider in determining what the rule should be -- basically in terms of what is the overall best for the environment without requiring an exorbitant cost."

REPORTER: After the public meeting in the Tampa area, Smallwood will draft a report and send it to Secretary Tschinkel, who will make recommendations based on that information. Those are then sent to the State Environmental Regulation Commission where a final decision will be made some time next year. But that

timetable could be upset if anything arises in the process that suggests there's some general public benefit from deferring the decision. Smallwood says a possible delay could come up because there's a real need for a demonstration project in the Bay area.

MR. SMALLWOOD: "Under varied, controlled conditions, we might allow some prilled sulphur to be brought in for a demonstration test for a lot of people who have never seen this material and find out what it really looks like, and necessary tests can be run under Florida conditions to find out what it actually would do here. If that were to be done, and there was to be general agreement on doing that, that might result in some delay of the final rulemaking until the results of that kind of a test were completed."

REPORTER: This issue will be setting a precedent if Agrico is allowed to bring in solid sulphur, and that's something that worries Freeport. Attorney Bill Earl:

MR. EARL: "If in fact it is brought in cheaper, all the companies may in fact be forced to go to the solid sulphur. So instead of one little 50,000-ton pile sitting down by Big Bend, all over the Tampa Bay area you'll have piles of solid sulphur. Our client might, in fact, be forced to bring in solid sulphur and store it in piles -- open storage piles. So, the cumulative impacts of this are enormous in terms of environmental impacts. You'll have four million tons a year -- what no one sees now in

the liquid form -- of open, visible storage and open, visible environmental problems and nuisances."

TRANSCRIPT OF RADIO PROGRAM

WMNF - April 8, 1983

REPORTER: Agrico, a company which has a plant in Hillsborough County, put in a request to the County to be allowed to switch from using liquid sulphur at its plant to a solid form of sulphur. The use of dry sulphur would involve the shipment of the chemical in and out of Tampa Bay to Agrico's plant at the Big Bend Terminal, and would necessitate the stockpiling of sulphur in the county.

MR. ESSENFELD: "After hearing a couple of environmental people and from the discussion of our own members, our members were unanimous that we were absolutely opposed to changing this technology and going to dry sulphur in Hillsborough County, whether it's Agrico or anybody else who wants to do it."

REPORTER: That's how the Chairman of the Hillsborough County Citizens' Advisory Committee . . . [inaudible]. The Citizens' Advisory Committee, which is appointed by the Board of County Commissioners, is designed to collect information on a broad range of proposals put before the commission and then make a recommendation. In this case, the committee voted unanimously not to allow the stockpiling of dry sulphur which would be necessary at Agrico's plant. Agrico in fact says that the sulphur is not even dry but in pellet form, which is moist. Here's the chemical company's Manager of Environmental Control, Harold Long:

MR. LONG: "We've never proposed bringing in dry sulphur. We have always proposed to bring in wet, solid prilled sulphur with a [inaudible] of around the two to three percent moisture content."

REPORTER: One of the major concerns that the Citizens' Advisory Committee has is the dust which they say will be created by the transport of the sulphur pellets or prills. Again, Agrico disagrees. Harold Long says that prill doesn't create any dust.

MR. LONG: "No it does not. That's what we're maintaining. I have actually seen work of prill handled, loaded, transported in Canada and in Houston, Texas. We just had some people over in Immingham, England that use it, and they reported no dust also and I've never seen any dust either.

REPORTER: In a letter to County Commissioner Jan Platt earlier this week, Citizens' Advisory Committee Chairman David Essenfeld said of the dust question that he had personally seen much evidence to prove that dust would be a problem if the stockpiling were allowed.

MR. ESSENFELD: "Well, the Citizens' Advisory Committee in its regular meeting last month saw the videotapes of sulphur being handled, including sulphur being handled in the form that Agrico wishes to use. We saw some loading of sulphur up in Northern Canada onto ships on the coast. I think it's British Colombia.

An enormous amount of dust is kicked up by this operation. Even if the sulphur is in prilled form -- this granular or pelletized form -- just the fact that these big scoops grab it and crush it and rub it and drop it into holds of ships causes some friction and causes a lot of dusting to happen, and it causes the sulphur to break down -- the form of the sulphur to break down -- and we saw an awful lot of dusting taking place on the loading site. We then saw -- we saw fire, in fact, aboard one of these ships where the dust -- the sulphur dust -- apparently exploded, and it caused a big problem."

REPORTER: And even one of the environmentalists who went to Immingham, England at Agrico's expense found sulphur dust to be a problem in transporting and stockpiling it in prill form. Mike Kinney, an environmental consultant with the Hillsborough County Environmental Coalition, is one of the people Harold Long of Agrico spoke of.

MR. KINNEY: "I was asked by one of the companies who wants to import solid sulphur to go over to Immingham and view a facility over there to show -- they wanted me to see the process in action so that I can formulate my own opinions, and basically, I was impressed with the potential dustiness of the industry. They had the technology available to control the emission along the conveyance system by covering it, and then they covered the stockpile with a -- they put it in a large building. So, the

environmental effects weren't present at those two points. However, when they were unloading the material from the boat, there was some dust generated at that point because of the lack of emission control, and generally, I think, poor housekeeping measures and poor technique by the crane operator."

REPORTER: Major problems associated with sulphur dust threaten the environment, property and public safety according to the Citizens' Advisory Committee, but Committee Chairman David Essenfeld is particularly fearful of two dangers posed by the dust.

MR. ESSENFELD: "Sulphur dust has two major problems. Number one, it corrodes anything it touches, especially metals. When it comes into contact, for example, with metals, it forms an action almost like your battery in your car, and it will literally eat away metal. It's an extremely corrosive product. The second thing is that dust itself is highly explosive so that the right concentration of dust and the spark could cause an explosion and a fire. The proposed stockpiles are right in the lightning belt that we have. We have a very active area for lightning, especially in the summertime here in Hillsborough County. One of the most active lightning strike areas in the whole world as a matter of fact. A lightning strike on this exposed sulphur stockpile would cause an explosion and a sulphur fire. Sulphur fire produces SO_2 , which is sulphur dioxide, which is itself intolerable. And the risk, the danger of that, is enormous."

REPORTER: As an alternative there is the old method of bringing sulphur into Agrico for its production of sulphuric acid. That is to bring it in in liquid form. The liquid form of sulphur has been used in the Tampa Bay area for several years, and according to most environmentalists, poses few of the hazards posed by sulphur in prill form. Again, here's David Essenfeld:

MR. ESSENFELD: "The liquid sulphur industry in Tampa has been described as an invisible industry. They have not harmed the ecology that we are aware of. You hardly ever hear of any problems. No one complains of sulphur leaks the way you hear about oil spills and things like that. No one -- I don't recall ever hearing about a sulphur spill, whereas with the exposed dry sulphur stockpiles, you have a continuous leaching into the ground, runoff into the bay, blowing of the dust, and all the problems that brought."

REPORTER: Agrico, however, has told the County Commission and the public that shifting its operation to prill form sulphur is safe. The bottom line for Agrico is costs. While sulphur in prill form would be brought in for much further weight, it would still be cheaper, according to Agrico's spokesperson Harold Long.

MR. LONG: "Well, we are a large consumer of sulphur in the manufacture of sulphuric acid, and as in any business, you're always looking for, you know, a cheaper product that can be utilized and which is simply one of economics. The prill that

was manufactured in Houston or in Canada is essentially a by-product, and therefore, they recover it as a by-product, and it's not their main product; therefore, logically it's cheaper. So it is just an economical consideration."

REPORTER: The recommendation to the Hillsborough County Commission from the Citizens' Advisory Committee is not binding in any way, though in the past committee recommendations have weighed heavily on commission decisions. The permitting process for the change to sulphur in prill form has been going on since 1977 and is sure to drag on for some time to come. The County Commission, for its part, can't comment on the issue until it comes up before them formally. Until then, however, environmentalists in the County and officials from Agrico are gearing up for a long battle.

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WRITER'S DIRECT NO.:

May 10, 1983

Mr. John P. Svec
Bureau of Air Quality Management
State of Florida
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32301-8241

DER
MAY 16 1983
BAQM

Dear Mr. Svec:

Thank you for your information submittal of April 29, 1983.
Enclosed please find a check for reproduction services in the
amount of \$7.80, made payable to the Department.

In addition, during the course of my review of your files, there
were four particular documents of interest which were of a rela-
tively voluminous nature and I did not ask to be copied at that
time. I would appreciate, however, if you would have those docu-
ments copied and remitted to me with your copying charge. More
specifically, the documents are as follows:

1. Technical memorandum: Review of Dr. Dale
Lundgren's Emission Factor for Prilled
Sulpher--Report dated October 18, 1982, by
Gale Hoffnagle (CCM) and John E. Yocum (PE);
2. Emission Tests Work and Testimony of Dr. Dale
Lundgren on Prilled Sulpher which includes an
"Analysis of Proposed Dust Control Program
and Predicted Air Impact for Agrico Chemical
Company's Big Ben Sulpher Nugget Terminal",
dated February, 1978;

3. Freeport's response to the Questionnaire sent by DER to assist in proposed rule-making;
4. Agrico's response to the Questionnaire sent by DER to assist in proposed rule-making.

In addition, this office is representing Gardinier, Inc., in matters presently pending before the Department dealing with potential rules and regulations governing sulphur handling. As a result, we would respectfully request that we be placed on your mailing list for information and copies of all materials dealing with the potential rule-making and reports of the Department in this regard. We would also like to be notified of any and all meetings, workshops, etc., dealing with the subject so that we may make arrangements to attend.

Finally, we are also interested in the proceedings dealing with the pending Agrico permit and would appreciate copies of all documents filed in that matter and we will, of course, pay for any reproduction services involved.

Your cooperation in this matter would be greatly appreciated as has been your cooperation to date.

Yours very truly,



CLIFFORD A. SCHULMAN

CAS/bwp

cc: Mr. O. E. Morris
Mr. David Buff

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TELEPHONE (904) 222-8396

April 22, 1983

DER

APR 22 1983

BAQM

Mr. Steve Smallwood, P. E.
Chief
Bureau of Air Quality Management
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, FL 32301

Re: Sulfur inquiry; H₂S data

Dear Steve:

Subsequent to our last meeting in Tampa, on March 21, 1983, I conveyed your request for data on H₂S emissions from liquid sulfur handling to the Texasgulf technical staff. I am now in receipt of such data, and am forwarding the same to you.

Enclosed please find a memorandum from Doug Anderson to Leonard Mason dated March 11, 1981, summarizing test results on H₂S emissions, obtained at Texasgulf's new Tampa terminal. The tests were conducted in February, 1981. The results reflect a very low level of H₂S emissions from the operation of the facility.

I trust the data proves useful to the Department as it moves forward in the sulfur inquiry. Please let me know if we can be of further assistance to you.

Sincerely,


Segundo J. Fernandez

SJF:dg
Enclosure

Texasgulf memo

DER

Date March 11, 1981

APR 22 1983

To Mr. Leonard Mason Location

From D. H. Anderson Location

BAQM

Subject Sulphur Tank Air Emissions at New Tampa Terminal.

This is a report of the sulfur tank air emissions testing at the Tampa Terminal on February 27 and 28, 1981 performed for the purpose of obtaining hydrogen sulphide emission data to be used in the application for an operation permit. The testing was done by Douglas Anderson and Roger Gay of the Newgulf laboratory. The estimated hydrogen sulphide emissions for tank filling are 0.306 lb/hr and 0.092 tons/year and for holding and loadout are 0.0903 lb/hr and 1.1 tons/year for a total emission of 1.192 tons/year. The predicted emission in the construction permit application was 0.034 tons/yr. A comparison of the permit values and test results are shown in Exhibit A.

The major cause of the discrepancy is the air flow rate from the vents during holding and loadout. The original data were from measurements made with a pitot tube at the Beaumont Terminal. Some of the difference is because the measurements were made during lower wind speeds than were at Tampa during the time of testing. Also, a pitot tube is not a good measuring instrument at the low velocities encountered because of the low pressure drop developed. The Tampa measurements were made with a Biram vane type anemometer which should give a more accurate measurement because it is designed for low air flows. Another difference in the values was caused by the proposed off loading of one million long tons of sulphur per year instead of 300,000 as in the permit.

The hydrogen sulphide test procedure was basically that of EPA method 11 with revisions to allow the use of Smith-Greenburg impingers instead of midget impingers. The procedure is described in more detail in Appendix A. The following is a list of Exhibits that are attached that further describe the testing procedures and results.

Exhibit	Subject
A	Comparison of Permit Values and Test Results
B	Emission Calculations for Tank Filling
C	Emission Calculations for Holding and Loadout
D	Sampling Operation Summary, Wind Speed, Wind Direction and Temperature
E	Weather Data for Test Days
F	Hydrogen Sulphide Concentration Intermediate Calculations
G	Hydrogen Sulphide Calculation Example
H	Solution Standardizations
I	Air Flow Calculations from the Holding Tank (Tank #3)
J	Dry Gas Meter Calibration
K	Rotameter Calibration
L-1 thru L-14	Raw Data Sheets

Sulphur Tank #3 held 1051 L tons of sulphur with 0.08% carbon content. Tank #1 initially held sulphur of about 0.08% carbon which changed to about 0.06% after loading and Tank #2 initially had about 0.05% carbon and this increased to about 0.07%. The carbon from the Marine Texan was approximately 0.05%.


D.H. Anderson

DHA/jr
xc:Mr.E.R.Thrasher(2) (w/attachments)
Mr. C.J.Staffa(w/attachments)
Mr.R.J.Staffa(w/attachments)
Mr.B.N.Soderman(no/attachment)

EXHIBIT A
COMPARISON OF CONSTRUCTION PERMIT
VALUE AND TEST RESULTS

	PERMIT VALUES	TEST RESULTS
TANK FILLING:		
Long Tons/yr	300,000	1,000,000
Fill rate, LT/hr	3000	1658
Concentration H ₂ S, PPM	112	202 & 168
Pounds H ₂ S/hr	0.439	0.306
Tons H ₂ S/yr	0.033	0.092
HOLDING AND LOADOUT:		
Concentration H ₂ S, PPM	112	49
Gas flow rate, SCFM	0.142	302
Pounds H ₂ S/hr	0.00009	0.0903
Tons H ₂ S/yr	0.00117	1.1
TOTAL:		
Tons H ₂ S/yr	0.034	1.192

EXHIBIT B
HYDROGEN SULPHIDE EMISSIONS
DURING TANK FILLING

Sulphur density = 112.5 lb/ft³
Fill rate = 1658 long tons/hour

$$\left(\frac{1658 \text{ L tons}}{\text{hr}} \right) \left(\frac{2240 \text{ lb}}{\text{L ton}} \right) \left(\frac{\text{ft}^3}{112.5 \text{ lb}} \right) \left(\frac{\text{hr}}{60 \text{ min}} \right) = 550.21 \text{ ACFM}$$

ACFM = Actual cubic feet per minute

Measured flow rate:

North Hatch	114 ACFM
South Hatch	444 ACFM
	<hr/> 558 ACFM

81% of the flow area is in these two hatches, also, the vents are longer than the hatches and are curved so the majority of the flow should be leaving the two large hatches during tank filling.

$$\begin{aligned} \text{Standard CFM} &= (550.21 \text{ ACFM}) \left(\frac{460 + 70}{460 + ((225 + 250)/2)} \right) \\ &= 418 \text{ SCFM} \end{aligned}$$

Assume 81% of the flow is out the two large hatches and 19% out the vents. Use the sulphur fill rate to determine gas flow. Use the average of the hydrogen sulphide concentrations measured at the large hatches for the hatch concentration and average for the vent concentrations for the remaining 19% of the flow.

$$\begin{aligned} 0.81 (418 \text{ SCFM}) &= 339 \text{ SCFM hatches} \\ 0.19 (418 \text{ SCFM}) &= 79 \text{ SCFM vents} \end{aligned}$$

$$\frac{178 + 226}{2} \text{ mg/m}^3 = 202 \text{ mg/m}^3 \text{ hatches}$$

$$\frac{235 + 149 + 42 + 167 + 251}{5} \text{ mg/m}^3 = 168 \text{ mg/m}^3 \text{ vents}$$

$$1 \text{ lb H}_2\text{S/hr} = 3.743 \times 10^{-6} (339) (202) = 0.256$$

$$1 \text{ lb H}_2\text{S/hr} = 3.743 \times 10^{-6} (79) (168) = 0.050$$

$$\text{Total } 1 \text{ lb H}_2\text{S/hr} = 0.306$$

$$\left(\frac{1,000,000 \text{ L tons sulphur}}{\text{year}} \right) \left(\frac{\text{hr}}{1658 \text{ L tons}} \right) = 603 \text{ hrs}$$

$$\begin{aligned} \left(\frac{603 \text{ hrs}}{\text{yr}} \right) (0.306 \frac{\text{lb H}_2\text{S}}{\text{hr}}) &= 185 \text{ lb H}_2\text{S/yr} \\ &= 0.092 \text{ tons H}_2\text{S/yr} \end{aligned}$$

EXHIBIT C
YEARLY EMISSIONS FOR SULPHUR TANKS
DURING HOLDING AND LOADOUT

Since the air flows, gas concentrations and temperatures were not taken at the same time and since conditions changed continually during the day it should not be expected that the flow rate and concentration from a given vent should necessarily be used together. An assumption was made that the vents with the higher flows had the higher concentrations and that outflow temperatures were 130°C and inflow temperatures were 80°C. The flow rates and concentrations were ranked and paired according to size and the emission rate was figured on this basis.

ACFM	mg/m ³	Assumed Temp. °C	SCFM 20°C	lb H ₂ S/hr
77.0	117	130	56	0.0245
75.0	90	130	55	0.0185
71.7	90	130	52	0.0175
62.3	60	130	45	0.0101
57.1	56	130	42	0.0088
50.3	56	105	39	0.0082
-19.7	14	80	-16	0.0027
Total				0.0903

$$\frac{(X \text{ mg}) \left(\frac{\text{gm}}{1000 \text{ mg}} \right) \left(\frac{\text{lb}}{454 \text{ gm}} \right) \left(\frac{\text{m}^3}{1000 \text{ l}} \right) (28.32 \text{ l}) (Y \text{ ft}^3) (60 \text{ min})}{\left(\frac{\text{m}^3}{\text{ft}^3} \right) (\text{min}) (\text{hr})}$$

$$= XY \left(\frac{(28.32)(60)}{454 \times 10^6} \right)$$

$$= 3.743 \times 10^{-6} XY \text{ lb/hr}$$

Calculations for vent with major flow into it. Assume the concentration of H₂S in the air when it is flowing out of the vent is 56 mg/m³. This assumption was made because the concentration is probably not as high as that of the high concentrations because of mixing with the incoming air and not as low as the lowest because

Page 2.

there was also some incoming air in that sample. Assume during the period of outflow the outflow rate is the same as the average of the five high flow values (50SCFM). Assume the average outflow flowrate for one minute is $(14 \text{ mg/m}^3)/(56 \text{ mg/m}^3)$ or 25% of the average high flow rate or $0.25 \times 50 = 13 \text{ SCFM}$.

$$\text{lb H}_2\text{S/hr} = (3.743 \times 10^{-6}) (56 \text{ mg/m}^3) (13 \text{ SCFM}) = 0.0027$$

$$\text{Hours per year} = 365 \times 24 = 8760$$

$$\begin{aligned} \text{Hours filling tank} &= 603 \\ (\text{From permit application}) \end{aligned}$$

$$\text{Holding and loadout time} \quad 8157 \text{ hrs}$$

$$\begin{aligned} (3 \text{ tanks}) \left(\frac{8157 \text{ hrs}}{\text{yr}} \right) \left(\frac{0.0903 \text{ lb}}{\text{hr}} \right) &= 2209 \text{ lb/yr} \\ &= 1.1 \text{ tons/yr} \end{aligned}$$

EXHIBIT D

SUMMARY OF SULPHUR TANK SAMPLING AT TAMPA TERMINAL

SAMPLE NO.	TANK	VENT	Mg H ₂ S/M ³	PPM H ₂ S	WIND DIREC.	WIND SPEED Mi./Hr	TEMP. F	DATE	TIME START
1	1	S.Hatch	178	126	29	2.5	62	2/27/81	07:48
2	1	N.Hatch	226	160	29	3.5	78	2/27/81	09:16
3	1	7	235	167	24	4.0	80	2/27/81	10:27
4	1	5	149	105	207	5.5	74	2/27/81	11:39
5	2	5	42	30	67	10.0	81	2/27/81	12:40
6	2	4	167	118	56	8.0	83	2/27/81	13:58
7	2	1	251	178	261	7.0	80	2/27/81	16:10
8	3	1	90	64	202	5.5	75	2/28/81	10:54
9	3	2	56	39	218	6.5	78	2/28/81	12:27
10	3	3	56	39	218	10.0	78	2/28/81	13:06
11	3	4	60	42	213	15.0	77	2/28/81	13:59
12	3	5	90	63	213	15.0	75	2/28/81	-----
13	3	6	14	10	218	14.5	74	2/28/81	15:22
14	3	7	117	83	234	14.0	72	2/28/81	16:24

Samples 1-7 while filling tank

Samples 8-14 during holding

EXHIBIT E

WEATHER DATA FOR TEST DAYS AT TAMPA SULPHUR TERMINAL

Source: National Weather Service/National Oceanic
and Atmospheric Administration Broadcast
Station At Ruskin, Florida

DATE	2/27/81	2/28/81
TIME	4:05 PM	3:30 PM
TEMPERATURE, °F		79
BAROMETRIC PRESSURE, in. Hg	30.11	30.11
RELATIVE HUMIDITY, %	41	32
WINDS, DIRECTION & SPEED, MPH	WNW-8	SW-10
CLOUD COVER	CLEAR	CLEAR

EXHIBIT F
HYDROGEN SULPHIDE SAMPLING DATA
TAMPA TERMINAL 2/27/81 & 2/28/81

SAMPLE	AIR VOLUME	CH ₂ S,mg/m ³ Impinger 1	CH ₂ S,mg/m ³ Impinger 2	CH ₂ S,mg/m ³ Impinger 3	CH ₂ S TOTAL mg/m ³	CH ₂ S TOTAL PPM
Tank 1 South Hatch	6.109	176.1	1.4	0.0	177.5	125.6
Tank 1 North Hatch	2.992	216.9	8.6	0.0	225.5	159.6
Tank 1 Center Vent	5.874	226.7	8.7	0.0	235.4	166.6
Tank 1 Vent 5	5.847	146.0	2.9	0.0	148.9	105.4
Tank 2 Vent 5	8.713	42.1	0.0	0.0	42.1	29.8
Tank 2 Vent 4	8.691	160.7	5.9	0.0	166.6	117.9
Tank 2 Vent 1	5.771	242.1	8.9	0.0	251.0	177.6
Tank 3 Vent 1	5.874	84.3	5.8	0.0	90.1	63.8
Tank 3 Vent 2	5.839	49.7	5.8	0.0	55.5	39.3
Tank 3 Vent 3	5.871	46.5	8.7	0.0	55.2	39.1
Tank 3 Vent 4	17.720	58.8	1.0	0.0	59.8	42.3
Tank 3 Vent 5	5.915	80.8	8.7	0.0	89.5	63.3
Tank 3 Vent 6	5.945	14.4	0.0	0.0	14.4	10.2
Tank 3 Vent 7	11.923	111.7	5.7	0.0	117.4	83.1

EXHIBIT G
HYDROGEN SULPHIDE
CALCULATION EXAMPLES

I. Volume Corrections

We will consider the Barometric Pressure to be constant during each run. Temperature correction must be made according to the Ideal Gas Law:

$$P_1 V_1 = n_1 R T_1$$

$$P_2 V_2 = n_2 R T_2$$

$$\text{If } n_1 = n_2$$

$$P_1 V_1 T_2 = P_2 V_2 T_1$$

since $P_1 = P_2$ From Above

$$V_1 T_2 = V_2 T_1$$

$$V_2 = V_1 T_2 / T_1$$

II. Volume Determination, Dry Gas

$$V_{mSTD} = V_m Y \left[(T_{std}/T_m) (P_{Bar}/P_{std}) \right]$$

$$Y = 1.0648$$

$$T_{STD} = 293 \text{ } ^\circ K$$

$$P_{STD} = 760 \text{ mm Hg}$$

For example, consider Tampa Terminal Tank 1, North Hatch

$$V_m = 0.10 \text{ Ft}^3 \times 28.3 \text{ l/Ft}^3 = 2.83 \text{ l}$$

$$T_m = 75.5^\circ F = 24.2^\circ C = 297^\circ K$$

$$P_{BAR} = 30.11 \text{ inHg} = 765 \text{ mm Hg}$$

$$V_{mSTD} = (2.83 \text{ l}) (1.0648) [293/297] (765/760) = 2.992 \text{ l}$$

III. Sulphur Concentration

Consider Tampa Terminal Tank 1, North Hatch, 1st Impinger

$$CH_2S = K \left[(V_{1T} N_1 - V_{TT} N_T)_{\text{sample}} - (V_{1T} N_1 - V_{TT} N_T)_{\text{Blank}} \right] / V_{mSTD}$$

$$K = 17.04 \times 10^3$$

$$V_{1T} = 10$$

$$N_1 = 0.0094$$

$$V_{TT} = 5.60$$

$$N_T = 0.01002$$

$$V_{mSTD} = 2.992 \text{ l}$$

Sample

$$V_{1T} = 10$$

$$N_1 = 0.0094$$

$$V_{TT} = 9.40$$

$$N_T = 0.01002$$

Blank

$$CH_2S \text{ 1st Impinger} = 216.9 \text{ mg/m}^3$$

$$CH_2S \text{ 2nd Impinger} = 8.6 \text{ mg/m}^3$$

$$CH_2S \text{ 3rd Impinger} = 0.0 \text{ mg/m}^3$$

$$CH_2S \text{ Total} = 225.9 \text{ mg/m}^3$$

EXHIBIT H
SOLUTION STANDARDIZATIONS

I. February 27, 1981

A. Normality of 0.1N Thiosulphate

$$N_S = 2039 \text{ W/V}_S$$

W = 8.0017 g/l = 2.0004 g/250 ml = Weight of Dichromate Standard

V_S = Volume of Thiosulphate Titration, ml.

Trial (1) = 40.75 ml Trial (2) = V_S = 40.70 ml V_{Savg} = 40.725 ml

$$N_S = 0.1002$$

B. Normality of 0.01N Thiosulphate = 0.1 x Normality of 0.1N Thiosulphate

$$N_S = 0.01002$$

C. Normality of 0.01N Iodine

$$N_I = N_T V_T / V_I$$

N_T = Normality of 0.01 Thiosulphate = 0.01002 N

V_I = Volume of 0.01 N Iodine = 25 ml

V_T = Volume of 0.01N Thiosulphate, ml.; Trial (1) = 23.45 ml;
Trial (2) = 23.50 ml

V_{Tavg} = 23.475 ml

$$N_I = 0.00940$$

II. February 28, 1981

A. Normality of 0.1N Thiosulphate

mls Thiosulphate: 40.70 (2) 40.70 AVG: 40.70 N_S = 0.10022

B. Normality of 0.01N Thiosulphate

$$N_S = 0.01002$$

C. Normality of 0.01N Iodine

mls Thiosulphate: (1) 23.50 (2) 23.50 AVG: 23.50 N_S = 0.00942

Standardization Procedures in Federal Regulations Followed

EXHIBIT I
TANK #3 AIR FLOW

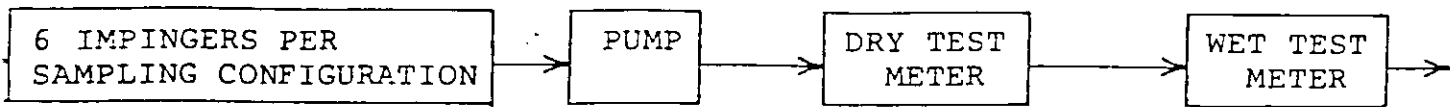
VENT	1	2	3	4	5	6	7
Time Stop	3:37:45	3:47:55	3:59:20	3:28:45	4:20:34	4:43:36	4:11:11
Time Start	3:32:33	3:42:43	3:54:01	3:21:37	4:15:00	4:37:54	4:05:44
Elapsed Time	5:12 5.20	5:12 5.20	5:19 5.32	7:08 7.13	5:34 5.57	5:42 5.70	5:3 5.5
Feet of Flow (- if into tank)	1616	974 (+,-)	2043	2550	1586	-560	207
Time Stop		4:30:49					
Time Start		4:25:45					
Elapsed Time		5:04 5.07					
Feet of Flow		1592 (+,-)					
Temp. °C 4:40 PM	130	129	70.80	129	130	80	130
Q, ACFM	62.3	37.6 63.0 50.3 avg	77.0	71.7	57.1	-19.7	75.0

DIAM. of 6" PIPE = 6.065"

$$A = \frac{\pi D^2}{4(144)} = 0.2006 \text{ ft}^2$$

$$Q = (\text{FT.}) \left(\frac{1}{\text{Min}} \right) (A \text{ FT}^2)$$

EXHIBIT J
 DRY GAS METER CALIBRATION
 FLOW CONFIGURATION



Dry Test Meter: Rockwell 175-S; Ser.No. 717426,DT2

Calibration Standard: Precision Wet Test Meter by Precision Scientific Co., G2SS, WT 1

Barometric Pressure = 30.14 in - Hg = 766 mm Hg

DRY TEST METER				WET TEST METER				Rotameter Float	Flowrate	VDT/ VwT
Temp. In	Temp. Out	Meter Reading ft ³	Standard Volume liters	Temp. In	Meter Reading ft ³	Standard Volume liters				
INITIAL CALIBRATION										
25	25.6	214.2 213.5 <u>0.7</u>	19.6	23.5	98.1635 97.4158 <u>0.7477</u>	21.10	80-90		0.929	
25.3	26.1	214.4 214.2 <u>0.2</u>	5.6	23.9	98.3770 98.1635 <u>0.2135</u>	6.014	90-91		0.931	
25.3	26.1	214.8 214.4 <u>0.4</u>	11.10	23.9	98.8087 98.3770 <u>0.4317</u>	12.06	95		0.921	
25.3	26.1	215.0 214.8 <u>0.2</u>	5.55	23.9	99.0180 98.8087 <u>0.2093</u>	5.85	95		0.952	
POST-TEST CALIBRATION										
24.4	24.4	225.35 225.05 <u>0.3</u>	8.36	23.9	1.4073 1.0880 <u>0.3193</u>	8.92	85		0.937	
24.4	24.4	225.65 225.35 <u>0.3</u>	8.36	23.9	1.7250 1.4073 <u>0.3177</u>		85		0.943	
24.4	24.4	225.75 225.65 <u>0.1</u>	2.79	23.9	1.8315 1.7250 <u>0.1065</u>	2.97	85		0.937	

EXHIBIT K

Calibration of Dry Testmeter Rotameter F3 on Dry Test Meter DT 2

Rotameter: Matheson, Tube No. 602

Calibration Standard: SKC 2 liter soap bubble flowmeter(only the first liter was used)

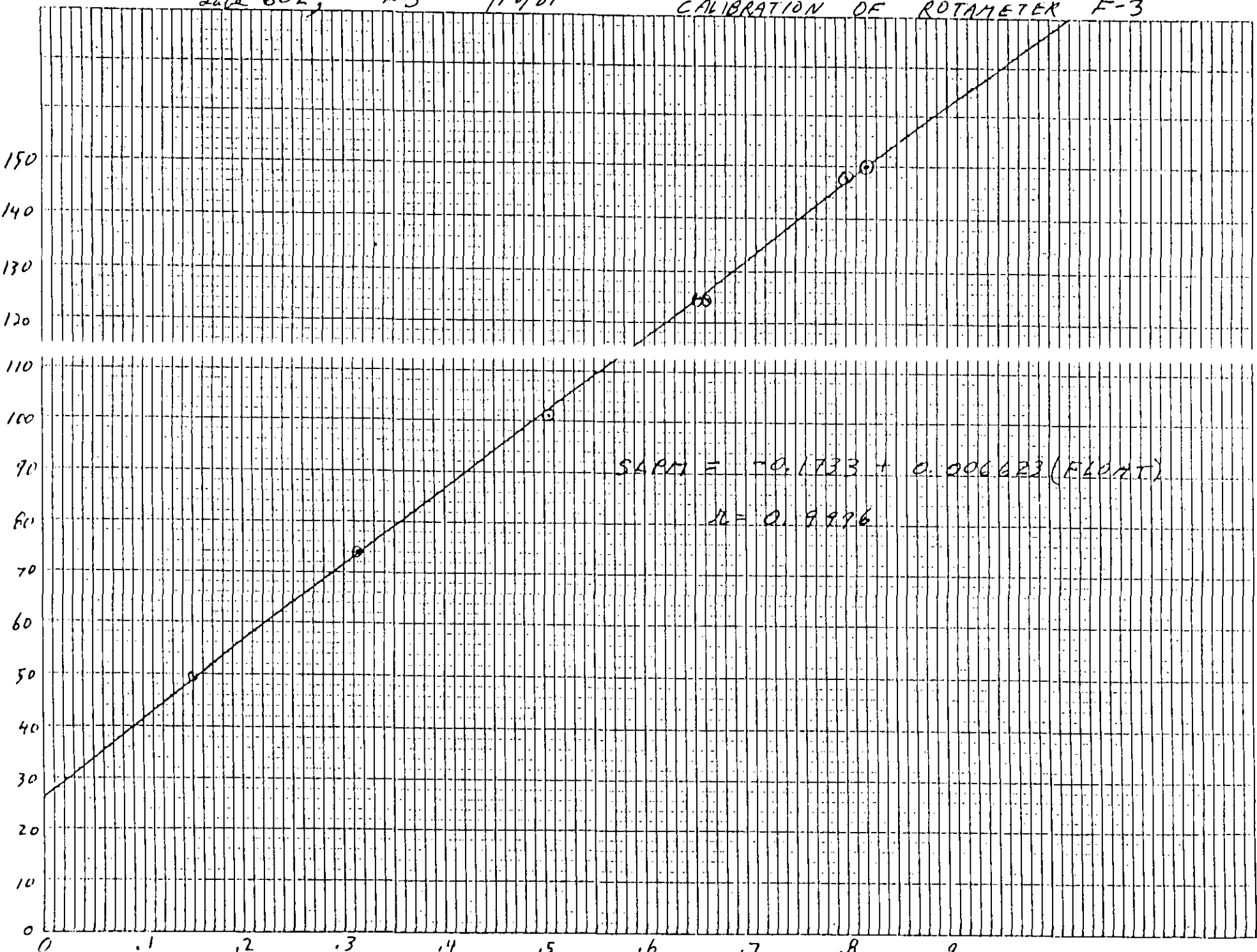
Temperature = 24.5°C

Barometric Pressure = 767 mmHg

Stainless Steel Float Setting	Time for 1 liter, Seconds	Actual LPM	Standard LPM
49.5	397.8	0.151	0.150
	398.05		
74	average 397.95		
	189.89	0.315	0.313
	191.06		
101	average 190.48		
	117.89	0.509	0.506
	117.97		
	average 117.93		
124.5	91.61	0.658	0.654
	90.70		
	average 91.16		
148	74.35	0.805	0.800
	74.66		
	average 74.51		
125	90.13	0.666	0.662
	89.94		
	average 90.04		
150	73.41	0.820	0.815
	73.18		
	72.91		
	73.05		
	average 73.14		

Tab 602, F3 1/19/81

CALIBRATION OF ROTAMETER F-3



$$SAPM = -0.1733 + 0.006623(FLOWRATE)$$

$$R = 0.9996$$

EXHIBIT L-1

LEASE: TEJASGOLF TAMPA TERMINAL TANK 1 MAP NUMBER _____

SAMPLE POINT: South Hatch

WELL NUMBER: 1

DATE: 2/27/81

TIME: 7:48 start
8:05:38

SAMPLING TIME: 17 min, 38 sec

ATMOSPHERIC PRESSURE: 30.11 in. Hg

TEMPERATURE: 67 68
59 62

T (average) = 64

TEST METER:

VOLUME (stop) = 218.202

VOLUME (start) = 218.002

0.200 x (0.9833) = _____ ft³

DHA

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>10</u>	<u>0.00940</u>	<u>3.10</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.35</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	_____

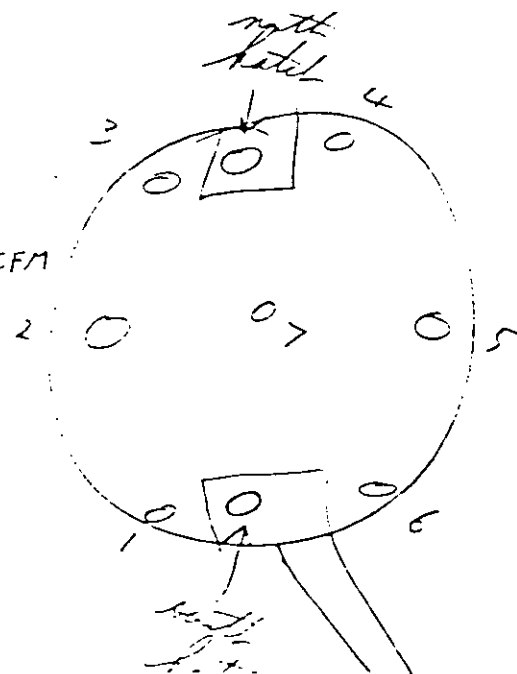
Flow = 75

Leak test OK

DT 2

$$\text{FLOW: } \left(\frac{769 \text{ ft}}{5 \text{ min.}} \right) \left(23 \text{ in. diam} \right)^2 \left(\frac{\pi}{4} \right) \left(\frac{1 \text{ ft}^2}{144 \text{ in.}^2} \right) = 443.8 \text{ ACFM}$$

$$443.8 \text{ ACFM} \left(\frac{460 + 70}{460 + 225} \right) = 343 \text{ SCFM}$$



769.5 ft
1000 ft
1000 ft

EXHIBIT L-2

LEASE: EXXON TAPPA TERNAL TANK 1

MAP NUMBER _____

SAMPLE POINT: North Vent (Harcu)

SAMPLE NUMBER: 2

DATE: 2/27/81

TIME: ~~8:16~~ 9:16:15 - 9:26:05

SAMPLING TIME: 9 min 50 sec

ATMOSPHERIC PRESSURE: 30.11 in. Hg

TEMPERATURE: 72 80
71 79

T(average) = 75.5

TEST METER:

VOLUME (stop) = 218.40

VOLUME (start) = 218.40

0.10 x (0.9643) = _____ ft³

DHA

TITRATION:

ml	IODINE n	ml S ₂ O ₃	n	Difference
<u>10</u>	<u>0.00940</u>	<u>5.60</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.25</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.50</u>	<u>0.01002</u>	_____

T_{amb} = 75

Vac. P = 22

225 PPM Nitrogen 8014-120c

FLOW:

$$\left(\frac{380 \text{ ft}}{9.65} \right) \left(23 \right)^2 \left(\frac{\pi}{4} \right) \left(\frac{1}{144} \right) = 113.6 \text{ ACFM}$$

250 °F

$$(113.6 \text{ ACFM}) \left(\frac{460 + 70}{460 + 250} \right) = 64.9 \text{ SCFM}$$

9:34:11

9:24:32

9:39

380 feet

9.65

EXHIBIT L-3

LEASE: TEASGULF TAMPA TERMINAL TANK 1

MAP NUMBER

SAMPLE POINT: Center vent10:46:07SAMPLE
SERIAL NUMBER: 310:27:02DATE: 2/27/81TIME: 9:25:30SAMPLING TIME: 19 min, 5 sec.ATMOSPHERIC PRESSURE: 30.11 in HgTEMPERATURE: 85 85T (average) = 85

TEST METER:

VOLUME (stop) = 218.908VOLUME (start) = 218.7080.200 x (0.9643) = ft³

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>10</u>	<u>0.00940</u>	<u>1.60</u>	<u>0.01002</u>	<u> </u>
<u>10</u>	<u>0.00940</u>	<u>9.10</u>	<u>0.01002</u>	<u> </u>
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	<u> </u>
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	<u> </u>

Flow = 831.25 = VacuumClear sky, lower
level slightly hazy

FLOW:

10:59
Drugs 1/2 tube
12-13 ppm on
walkway downwind
of with batch

EXHIBIT L-4

LEASE: TELOSQUE TAMPA TERMINAL, TANK 1

MAP NUMBER _____

SAMPLE POINT: Vent 5SAMPLE
PWC NUMBER: 4DATE: 2/27/81TIME: 11:39:30 - 11:56:22SAMPLING TIME: 16 min, 52 secATMOSPHERIC PRESSURE: 30.11 in. HgTEMPERATURE: 48 86
90 88T (average) = 88

TEST METER:

VOLUME (stop) = 219.300VOLUME (start) = 219.1000.200 x (0.9643) = _____ ft³

TITRATION:

ml	IODINE n	ml	S ₂ O ₃ n	Difference
<u>10</u>	<u>0.00940</u>	<u>4.40</u>	<u>0.01802</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.30</u>	<u>0.01802</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01802</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01802</u>	_____

Flow = 83Var = 2.7Vent 5 is

FLOW:

Wind Direction Changed, On downwind sideMore emissions from south hatch than north hatch

TEXACO Gulf TAMPRA TERMINAL

LEASE: Tank #2

MAP NUMBER _____

SAMPLE POINT: # Vent #5 - most upwind ventSAMPLE
NUMBER: 5DATE: (1/27/41TIME: 12:46:00~1:10 PM (13:10)2/27/81 WESSAMPLING TIME: 24 minATMOSPHERIC PRESSURE: 30.11 in. HgTEMPERATURE: 92 93T (average) = 91.5

TEST METER:

VOLUME (stop) = 219.81VOLUME (start) = 219.510.300 x (0.9643) = _____ ft³

TITRATION:

ml	IODINE n	ml	S ₂ O ₃ n	Difference
<u>10</u>	<u>0.00940</u>	<u>7.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.35</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.35</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.35</u>	<u>0.01002</u>	_____

Flar = 90Var = 2.0Wind direction changed again before test

FLOW:

EXHIBIT L-6

LEASE: TEIGULF TANK TERMINAL TANK 2 MAP NUMBER _____

SAMPLE POINT: Vent #4

SAMPLE SRC NUMBER: 6

DATE: 1/27/81

TIME: 1154 00

SAMPLING TIME: 2/27/81 NCS

ATMOSPHERIC PRESSURE: 30.11 in Hg

TEMPERATURE: 89 85
91 95

T (average) = 92.5

TEST METER:

VOLUME (stop) = 220.300

VOLUME (start) = 220.000

0.300 x (0.9643) = _____ ft³

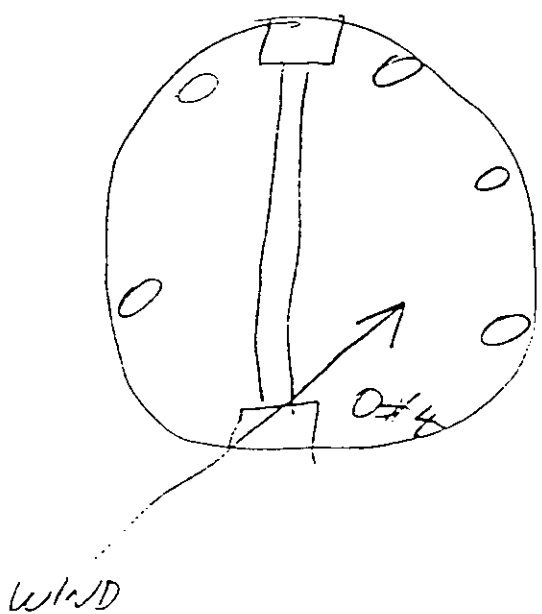
TITRATION:

ml	IODINE n	ml	S ₂ O ₃ n	Difference
<u>20</u>	<u>0.00940</u>	<u>10.60</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.10</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.40</u>	<u>0.01002</u>	_____

Flow = 90

γ_u = 2

FLOW:



LEASE: TRIPOLI TANK TECHNICAL TANK &

MAP NUMBER _____

SAMPLE POINT: Vent. F. /SAMPLE
NO. NUMBER: 7DATE: 1/27/81TIME: 4:10:15 —2/27/81 NKG

SAMPLING TIME: _____

ATMOSPHERIC PRESSURE: 30.11 in. HgTEMPERATURE: 90 100T(average) = 95

TEST METER:

VOLUME (stop) = 220.715VOLUME (start) = 220.5150.200 x (0.9643) = _____ ft³

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>20</u>	<u>0.00940</u>	<u>10.40</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>8.90</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00940</u>	<u>9.20</u>	<u>0.01002</u>	_____

FLAV = 87

VAC = 2

5

FLOW:

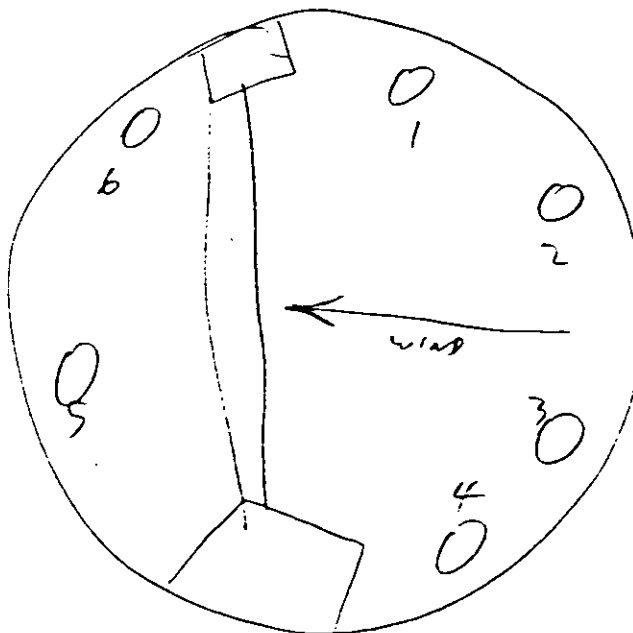


EXHIBIT L-8

LEASE: TEXASGULF TAMPA TERMINAL

MAP NUMBER _____

SAMPLE POINT: Tank 3 Vent 1

SAMPLE
SRC NUMBER: 9

DATE: 2/28/81

TIME: 11:09:18
10:54:45

SAMPLING TIME: 14 min., 53 sec.

ATMOSPHERIC PRESSURE: 30.11 in Hg

TEMPERATURE: 86 84

T (average) = 85

TEST METER:

VOLUME (stop) = 221.158

VOLUME (start) = 220.958

0.200 x (0.9643) = _____ ft³

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>20</u>	<u>0.00942</u>	<u>15.90</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.40</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.40</u>	<u>0.01002</u>	_____

FLOW: = 83

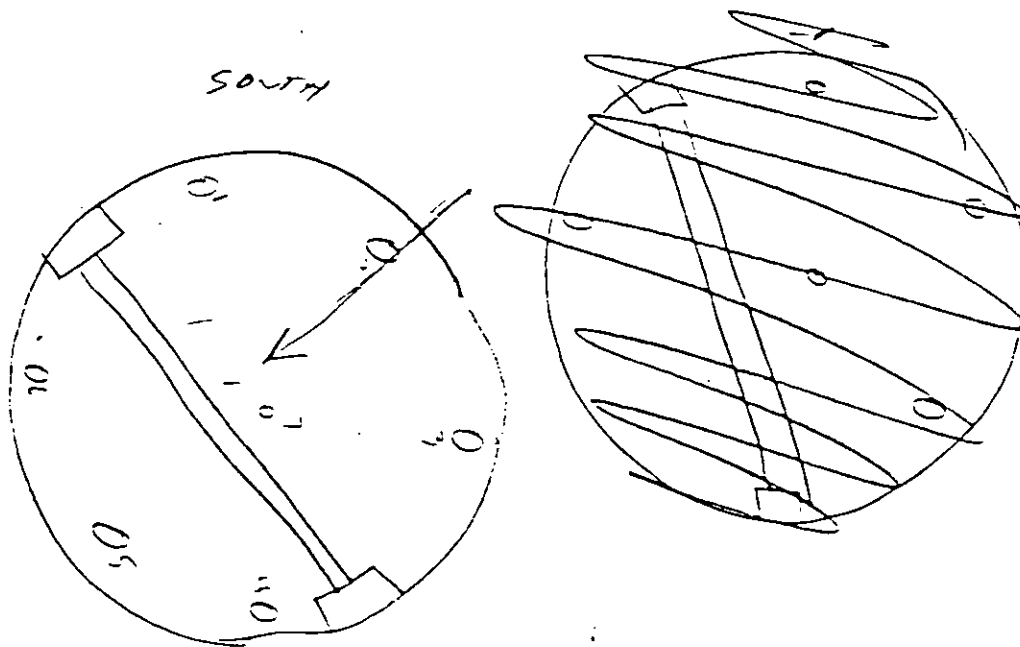


EXHIBIT L-9

TEASGULE TAYPO TERMINAL

LEASE: Link #3 quiescent MAP NUMBER _____

SAMPLE POINT: Vest #2

AMPLE NUMBER: 9

DATE: 1/26/81

TIME: 12:27:30 - 12:47:20

2/28/81 was

SAMPLING TIME: 15 min, 50 sec.

ATMOSPHERIC PRESSURE: 30.11 in. Hg

TEMPERATURE: 87 90
87 89

T (average) = 88.25

TEST METER:

VOLUME (stop) = 221.490

VOLUME (start) = 221.290

0.200 x (0.9643) = _____ ft³

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>10</u>	<u>0.00942</u>	<u>7.60</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.10</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.30</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.30</u>	<u>0.01002</u>	_____

VAC = 2

Summary
Kays
12:31:34
12:30:00

FLOW: = 85

Wind sensor as vent #1

EXHIBIT L-10

Texasgulf Targo Tidal

LEASE: TANK # 3

MAP NUMBER _____

SAMPLE POINT: Vent # 3SAMPLE NO. NUMBER: 10DATE: 1/28/812/28/81 WGSSAMPLING TIME: 13 min. 4 secATMOSPHERIC PRESSURE: 30.11 in. HgTIME: 13:06:10 13:19:14TEMPERATURE: 82 87
85 87T (average) = 85.25

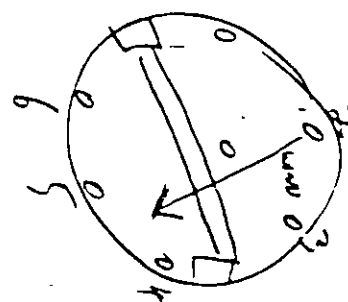
TEST METER:

VOLUME (stop) = 221.902VOLUME (start) = 221.7020.200 x (0.9643) = _____ ft³

TITRATION:

ml.	IODINE n	ml	S ₂ O ₃ n	Difference
<u>10</u>	<u>0.00942</u>	<u>7.60</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>8.90</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____

VAC = 2

FLOW: 84(first 0.05 ft³ @ 7 150)

TEXASGOLF TAMPA TERMINAL

LEASE: 7-16 #3

MAP NUMBER _____

SAMPLE POINT: 7-16 #4SAMPLE NUMBER: 11DATE: 3/28/81TIME: 11:59:00 - 2:42:23SAMPLING TIME: 43 min 23 secATMOSPHERIC PRESSURE: 30.11 in HgTEMPERATURE: 81 83T(average) = 82

TEST METER:

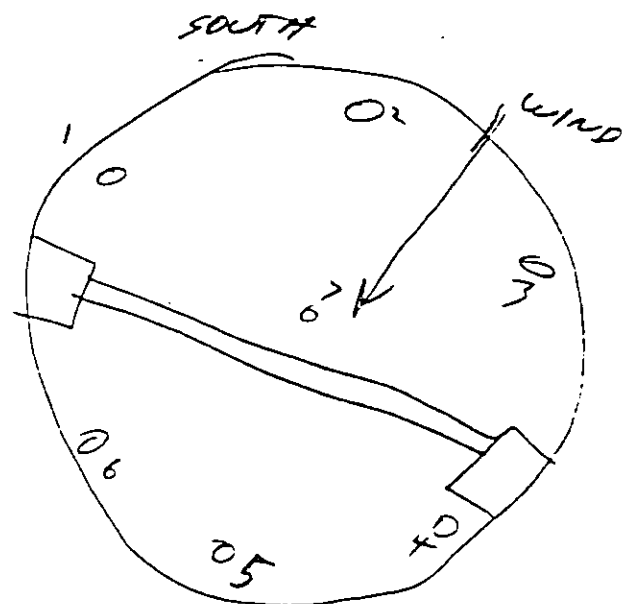
VOLUME (stop) = 222.700VOLUME (start) = 222.1000.600 x (0.9643) = _____ ft³

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>10</u>	<u>0.00942</u>	<u>3.10</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.10</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____

VAC = 1.5

FLOW: = 90



TEASQUE TAPA TERMINAL

LEASE: Trk #3

MAP NUMBER _____

SAMPLE POINT: Vent #5SAMPLE
SRC NUMBER: 12DATE: 2/28/81

TIME: _____

SAMPLING TIME: _____

ATMOSPHERIC PRESSURE: 30.11 in. HgTEMPERATURE: 81 81
82 81T (average) = 81.25

TEST METER:

VOLUME (stop) = 223.130VOLUME (start) = 222.9300.200 x (0.9643) = _____ ft³

TITRATION:

IODINE		S ₂ O ₃		Difference
ml	n	ml	n	
<u>10</u>	<u>0.00942</u>	<u>6.60</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.10</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.40</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.40</u>	<u>0.01002</u>	_____

VAC = 1.4

Wind same as vent #4

FLOW: = 85

EXHIBIT L-13

Terasgulf Tampa Terasgulf

LEASE: Leak #3

MAP NUMBER _____

SAMPLE POINT: #6SAMPLE
SEC NUMBER: 13DATE: 2/28/81TIME: 3:40:10 3:22:02SAMPLING TIME: 12 min, 8 secATMOSPHERIC PRESSURE: 30.11 in. HgTEMPERATURE: 79 78T(average) = 78.5

TEST METER:

VOLUME (stop) = 223.611VOLUME (start) = 223.4110.200 x (0.9643) = _____ ft³

TITRATION:

ml	IODINE n	ml	S ₂ O ₃ n	Difference
<u>10</u>	<u>0.00942</u>	<u>8.70</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____
<u>10</u>	<u>0.00942</u>	<u>9.20</u>	<u>0.01002</u>	_____

VAC = 1.4FLOW: 85

Texasgulf TOWER TERMINAL

LEASE:

7 1/2 # 3

MAP NUMBER

SAMPLE POINT:

7

SAMPLE
SRC NUMBER:

14

DATE:

2/28/81

TIME: 4:24:05

4:53:20

SAMPLING TIME:

29 min. 15 sec

ATMOSPHERIC PRESSURE:

30.11 in. Hg

TEMPERATURE:

77 77

T (average) =

77

TEST METER:

VOLUME (stop) =

224, 211

VOLUME (start) =

223, 811

0.400

x (0.9643) =

ft³

TITRATION:

IODINE

S₂O₃

ml

n

ml

n

Difference

20

0.00942

10.80

0.01002

10

0.00942

8.80

0.01002

10

0.00942

9.20

0.01002

10

0.00942

9.20

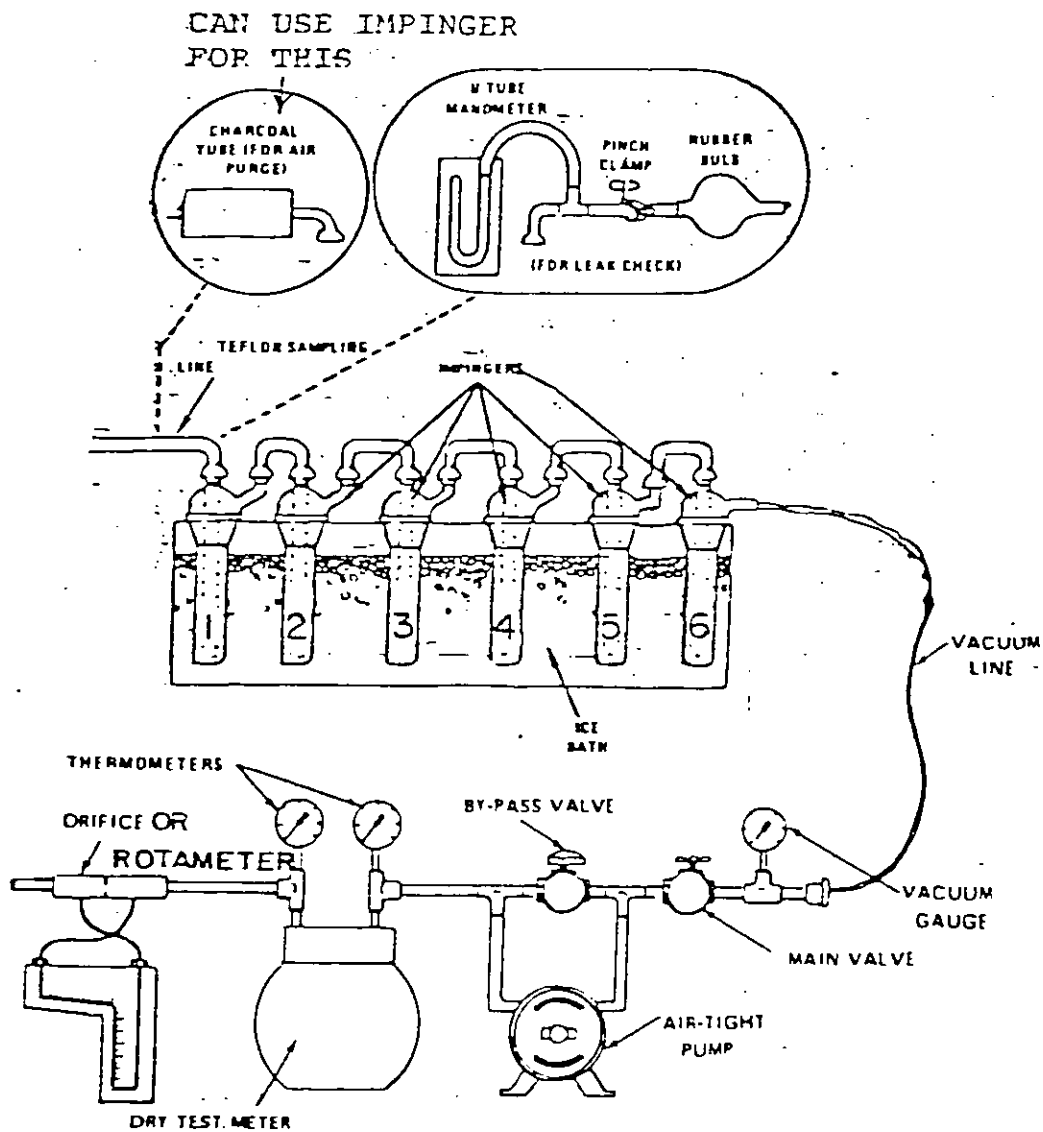
0.01002

VAC = 1.3

FLOW:

= 90

FIGURE 11-1



Impinger Number	Contents
1	Hydrogen peroxide
2	Empty, straight tube
3	Absorbing solution, straight tube
4	Absorbing solution
5	Absorbing solution
6	Silica gel, straight tube

APPENDIX A

METHOD - DETERMINATION OF HYDROGEN SULPHIDE CONTENT OF FUEL GAS STREAMS FROM SULPHUR STORAGE TANKS

1. Principle and Applicability. 1.1 Principle. Hydrogen sulfide (H_2S) is collected from a source in a series of Smith-Greenburg Impingers and absorbed in pH 3.0 cadmium sulfate (CdSO_4) solution to form cadmium sulfide (CdS). The latter compound is then measured iodometrically. An impinger containing hydrogen peroxide is included to remove SO_2 as an interfering species. This method is a revision of Method 11 and uses some procedures developed by the Texas Air Control Board and some from ASTM Method D 2385.
- 1.2 Applicability. This method is applicable for the determination of the hydrogen sulfide content of exiting gas flows from sulfur storage tanks.
2. Range and Sensitivity. The maximum of the range is approximately 7000 ppm.
3. Interferences. Any compound that reduces iodine or oxidizes iodide ion will interfere in this procedure, provided it is collected in the cadmium sulfate impingers. Sulfur dioxide in concentrations of up to 2,600 mg/m^3 is eliminated by the hydrogen peroxide solution. Thiols precipitate with hydrogen sulfide. In the absence of H_2S , only co-traces of thiols are collected. When methane and ethane-thiols at a total level of 300 mg/m^3 are present in addition to H_2S , the results vary from 2 percent low at an H_2S concentration of 400 mg/m^3 to 13 percent high at an H_2S concentration of 100 mg/m^3 . Carbon oxysulfide at a concentration of 20 percent does not interfere. Certain carbonyl-containing compounds react with iodine and produce recurring end points. However, acetaldehyde and acetone at concentrations of 1 and 3 percent, respectively, do not interfere. Entrained hydrogen peroxide produces a negative interference equivalent to 100 percent of that of an equimolar quantity of hydrogen sulfide. Avoid the ejection of hydrogen peroxide into the cadmium sulfate impingers.
- .0 Reserved.
- . Apparatus
 - .1 Sampling apparatus
 - 1.1 Sampling line. Teflon tubing and glass tubing to connect the sampling train to the sampling valve.
 - 1.2 Impingers. Six Smith Greenburg impingers, three with modified straight tube tip per Method 8, paragraph 2.1.6.
 - 1.3 Glass or Teflon connecting tubing for the impingers.
 - 1.4 Ice bath container. To maintain absorbing solution at a low temperature.

- 5.1.5 Drying tube. Use a straight tip impinger packed with 6 to 16-mesh indicating-type silica gel or equivalent to dry the gas sample and protect the meter and pump. If the silica gel has been used previously, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.
- 5.1.6 Volume meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate (1.0 liter/min) and conditions actually encountered during sampling. The meter shall be equipped with a temperature gauge (dial thermometer or equivalent) capable of measuring temperature to within 3°C (5.4°F). The gas meter should have a petcock, or equivalent, on the outlet connector which can be closed during the leak check. Gas volume for one revolution of the meter must not be more than 10 liters.
- 5.1.7 Flow meter. Rotameter or equivalent to measure flow rates in the range from 0.5 to 2 liters/min (1 to 4 cfm).
- 5.1.8 Graduated cylinder, 250 ml size.
- 5.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice-versa for elevation decrease.
- 5.1.10 U-tube manometer or equivalent. 0-30 cm water column. For leak check.
- 5.1.11 Rubber squeeze bulb. To pressurize train for leak check.
- 5.1.12 Tee, pinch clamp, and connecting tubing. For leak check.
- 5.1.13 Pump. Leak free and capable of drawing air through the impingers at the required sampling rate.
- 5.1.14 Tube packed with activated carbon. To filter air during purge.
- 5.2 Sample Recovery.
- 5.2.1 Pipettes, 10 ml to 100 ml. Size needed will depend on hydrogen sulphide concentration.
- 5.2.2 Graduated cylinders. 250 ml
- 5.2.3 Wash bottle
- 5.3 Analysis
- 5.3.1 Burette. 50 ml

6. Reagents. Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use best available grade.

6.1 Sampling.

- 6.1.1 Cadmium sulfate absorbing solution. Dissolve 41 g of $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ and 15 ml of 0.1 M Sulfuric acid in a 1-liter volumetric flask that contains approximately $3/4$ liter of deionized distilled water. Dilute to volume with deionized water. Mix thoroughly. pH should be 3 ± 0.1 .

- 6.1.2 Hydrogen peroxide, 3 percent. Dilute 30 percent hydrogen peroxide to 3 percent as needed. Prepare fresh daily.

- 6.1.3 Water. Deionized, distilled.

6.2 Sample recovery.

- 6.2.1 Hydrochloric acid solution (HCl), 3M. Add 240 ml of concentrated HCl (specific gravity 1.19) to 500 ml of deionized, distilled water in a 1-liter volumetric flask. Dilute to 1 liter with deionized water. Mix thoroughly.

- 6.2.2 Iodine solution 0.1 N. Dissolve 24 g of potassium iodide (KI) in 30 ml of deionized, distilled water. Add 12.7 g of resublimed iodine (I_2) to the potassium iodide solution. Shake the mixture until the iodine is completely dissolved. If possible, let the solution stand overnight in the dark. Slowly dilute the solution to 1 liter with deionized distilled water, with swirling. Filter the solution if it is cloudy. Store solution in a brown-glass reagent bottle. For high concentration of hydrogen sulphide a stronger solution may be required to reduce the total volume of liquid.

- 6.2.3 Standard iodine solution. 0.01 N. Pipette 100.0 ml of the 0.1 N iodine solution into a 1-liter volumetric flask and dilute to volume with deionized, distilled water. Standardize daily as in section 8.1.1. This solution must be protected from light. Reagent bottles and flasks must be kept tightly stoppered.

6.3 Analysis.

- 6.3.1 Sodium thiosulfate solution, standard 0.1 N. Dissolve 24.8 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) or 15.8 g of anhydrous sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) in 1 liter of deionized, distilled water and add 0.01 g of anhydrous sodium carbonate (Na_2CO_3) and 0.4 ml of chloroform (CHCl_3) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes and store in a glass-stoppered, reagent bottle. Standardize as in section 8.1.2.

6.3.2. Sodium thiosulfate solution, standard 0.01 N. Pipette 50.0 ml of the standard 0.1 N thiosulfate solution into a volumetric flask and dilute to 500 ml with distilled water.

6.3.3. Reserved.

6.3.4. Starch indicator solution. Bring to a boil 600 ml of deionized distilled water. Thoroughly mix 1 gram HgI_2 and 200 grams of soluble starch; the HgI_2 acts as a preservative. Make a paste of 2 grams of this mixture by adding a small amount of deionized distilled water slowly to the starch in a beaker while stirring. Add the paste to the boiling water. Cool and dilute to one liter with deionized distilled water.

NOTE. Test starch indicator solution for decomposition by titrating, with 0.01 N iodine solution, 4 ml of starch solution in 200 ml of distilled water that contains 1 g potassium iodide. If more than 4 drops of the 0.01 N iodine solution are required to obtain the blue color, a fresh solution must be prepared.

7. Procedure.

7.1 Sampling.

7.1.1 Assemble the sampling train as shown in Figure 11-1, connecting the six impingers in series. Place 200 to 250 ml of 3 percent hydrogen peroxide solution in the first impinger. Leave the second impinger empty. Place 200 to 250 ml of the cadmium sulfate absorbing solution in the third, fourth, and fifth impingers. Place silica gel in the sixth impinger. Place the impinger assembly in an ice bath container and place crushed ice around the impingers. Add more ice during the run, if needed.

7.1.2 Connect the rubber bulb and manometer to the first impinger, as shown in Figure 11-1. Close the petcock on the dry gas meter outlet. Pressurize the train to 25-cm water pressure with the bulb and close off the tubing connected to the rubber bulb. The train must hold a 25-cm water pressure with not more than a 1-cm drop in pressure in a one minute interval. Stopcock grease is acceptable for sealing around glass joints.

7.1.3 Purge the connecting line between the sampling valve and the first impinger by disconnecting the line from the first impinger and sucking air through the sample line with a rubber bulb. Reconnect the line to the impinger train. Open the petcock on the dry gas meter outlet. Record the initial dry gas meter reading.

- 7.1.4 Open the sampling valve and then adjust the valve to obtain a rate of approximately 0.3 liter/min. Maintain a constant (± 10 percent) flow rate during the test. Record the meter temperature.
- 7.1.5 Sample for at least 10 min. At the end of the sampling time turn off the pump and record the final volume and temperature readings.
- 7.1.6 Disconnect the impinger train from the sampling line. Connect the charcoal tube as shown in Figure 11-1. Purge the train with clean ambient air to ensure that all H_2S is removed from the hydrogen peroxide. For sample recovery, cap the open ends and remove the impinger train to a clean area that is away from sources of heat. The area should be well lighted but not exposed to direct sunlight.
- 7.2 Sample Recovery.
 - 7.2.1 Discard the contents of the hydrogen peroxide impinger.
 - 7.2.2 Pipette an exact known amount of iodine solution into each impinger that contains absorbing solution. The amount and concentration of iodine solution needed will vary with the amount of hydrogen sulphide collected and will probably have to be determined by experience. An excess of iodine must be added.
 - 7.2.3 Add 10 ml of 3M HCl to the solution. Place the top on the impinger and shake briefly. Once the HCl has been added the impinger must be kept closed at all times except for adding or removing solutions.
 - 7.2.4 Allow the impinger to stand about 30 minutes in the dark for absorption of the H_2S into the iodine, then complete the titration analysis as in section 7.3.

Note: Caution! Iodine evaporates from acidified iodine solutions. Samples to which acidified iodine has been added may not be stored, but must be analyzed in the time schedule stated in section 7.2.4.
 - 7.2.5 Prepare a blank by adding 45 ml of cadmium sulfate absorbing solution to an iodine flask. Pipette a known volume of 0.01 N iodine solution into the flask. Add 10 ml of 3 M HCl. Stopper the flask, shake briefly, let stand 30 minutes in the dark and titrate with the samples.

Note: The blank must be handled by exactly the same procedure as that used for the samples.

7.3 Analysis

Note-Titration analyses should be conducted at the sample-cleanup area in order to prevent loss of iodine from the sample. Titration should never be made in direct sunlight.

- 7.3.1 Using 0.01 N sodium thiosulfate solution, rapidly titrate each sample in each impinger using gentle mixing, until the solution is light yellow in color. Add 4 ml of starch indicator solution and continue titrating slowly until the blue color just disappears. Record V_{TT} , the volume of sodium thiosulfate used.

8. Calibration and standards.

8.1 Standardizations:

- 8.1.1 Standardize the 0.01 N iodine solution daily as follows: Pipette 25 ml of the iodine solution into a 125 ml Erlenmeyer flask. Add 2 ml of 3 M HCl. Titrate rapidly with standard 0.01 N thiosulfate solution until the solution is light yellow using gentle mixing. Add four drops of starch indicator solution and continue titrating slowly until the blue color just disappears. Record V_T , the volume of thiosulfate solution used. Repeat until replicate values agree within 0.05 ml. Average the replicate titration values which agree within 0.05 ml and calculate the exact normality of the iodine solution using equation 9.3. Repeat the standardization daily.
- 8.1.2 Standardize the 0.1 N thiosulfate solution as follows: Oven-dry potassium dichromate ($K_2Cr_2O_7$) at 180 to 200°C (360 to 390°F). Weigh to the nearest milligram, 2 grams of potassium dichromate. Transfer the dichromate to a 500 ml volumetric flask, dissolve in deionized distilled water, and dilute to exactly 500 ml. In a 500 ml iodine flask, dissolve approximately 3 grams of potassium iodide (KI) in 45 ml of deionized distilled water, then add 10 ml of 3 M hydrochloric acid solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of deionized distilled water, washing down the sides of the flask with part of the water. Titrate with 0.1 N thiosulfate until the solution is light yellow. Add 4 ml of starch indicator and continue titrating slowly to a green end point. Record V_3 , the volume of thiosulfate solution used (ml). Repeat until replicate analyses agree within 0.05 ml. Calculate the normality using equation 9.1. Repeat the standardization each week, or after each test series, whichever time is shorter.

8.2 Sampling train calibration. Calibrate the sampling train components as follows:

8.2.1 Dry Gas meter.

8.2.1.1. Initial calibration. The dry gas meter shall be calibrated before its initial use in the field. Proceed as follows: First, assemble the following components in series: Six impingers in the sampling train configuration, pump with valves, dry gas meter, and orifice or rotameter. Calibrate the dry gas meter (at the sampling flow rate specified by the method) as follows: Connect an appropriately sized wet test meter to the inlet of the drying tube. Make three independent calibration runs, using at least two revolutions of the dry gas meter per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the dry gas meter is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

8.2.1.2. Post-test calibration check. After each field test series, conduct a calibration check as in section 8.2.1.1. above, except that only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in section 8.2.1.1.), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the dry gas meter as in section 8.2.1.1. and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

8.2.2 Thermometers. Calibrate against mercury-in-glass thermometers.

8.2.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

8.2.4 Barometer. Calibrate against a mercury barometer.

9. Calculations. Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off results only after the final calculation.

9.1 Normality of the Standard ($\sim 0.1N$) Thiosulfate Solution.

$$N_S = 2.039W/V_S$$

where:

W=Weight of $K_2Cr_2O_7$ used, g.

V_S =Volume of $Na_2S_2O_3$ solution used, ml.

N_S =Normality of standard thiosulfate solution, g-eq/liter.

2.039=Conversion factor

$$\frac{(6 \text{ eq. } I_2/\text{mole } K_2Cr_2O_7)(1,000 \text{ ml/liter})}{(294.2 \text{ g } K_2Cr_2O_7/\text{mole})(10 \text{ aliquot factor})}$$

9.2 Reserved

9.3 Normality of Standard Iodine Solution.

$$N_I = N_T V_T / V_I$$

where:

N_I =Normality of standard iodine solution, g-eq/liter.

V_I =Volume of standard iodine solution used, ml.

N_T =Normality of standard ($\sim 0.01N$) thiosulfate solution: assumed to be $0.1N_S$ g-eq/liter.

V_T =Volume of thiosulfate solution used, ml.

9.4 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions ($20^\circ C$) and 760 mm Hg.

$$V_m(\text{std}) = V_m Y [(T_{\text{std}}/T_m) (P_{\text{bar}}/P_{\text{std}})]$$

where:

$V_m(\text{std})$ =Volume at standard conditions of gas sample through the dry gas meter, standard liters.

V_m =Volume of gas sample through the dry gas meter (meter conditions) liters

T_{std} =Absolute temperature at standard conditions, $293^\circ K$.

T_m =Average dry gas meter temperature, $^\circ K$.

P_{bar} =Barometric pressure at the sampling site, mm Hg.

P_{std} =Absolute pressure at standard conditions, 760 mm Hg.

Y=Dry gas meter calibration factor

- 9.5 Concentration of H_2S . Calculate the concentration of H_2S in the gas stream at standard conditions using the following equation:

$$C_{H_2S} = KI \{ (V_{1T}N_1 - V_{TT}N_T) \text{ sample} - (V_{1T}N_1 - V_{TT}N_T) \text{ blank} \} / V_m(\text{std})$$

where (metric units):

C_{H_2S} = Concentration of H_2S at standard conditions, mg/dscm.

K = Conversion factor = 17.04×10^3

$(34.07 \text{ g/mole } H_2S) (1,000 \text{ liters/m}^3) (1,000 \text{ mg/g}) / (1,000 \text{ ml/liter}) (2H_2S \text{ eq/mole})$

V_{1T} = Volume of standard iodine solution = 50.0 ml.

N_1 = Normality of standard iodine solution, g-eq/liter.

V_{TT} = Volume of standard ($\sim 0.01N$) sodium thiosulfate solution, ml.

N_T = Normality of standard sodium thiosulfate solution, g-eq/liter.

$V_m(\text{std})$ = Dry gas volume at standard conditions, liters.

dscm = Dry standard cubic meters

10. Stability. The absorbing solution is stable for at least 1 month. Sample recovery and analysis should begin within 1 hour of sampling to minimize oxidation of the acidified cadmium sulfide. Once iodine has been added to the sample, the remainder of the analysis procedure must be completed according to sections 7.2.2 through 7.3.1.
11. Bibliography.
- 11.1 Determination of Hydrogen Sulfide, Ammoniacal Cadmium Chloride Method. API Method 772-54. In: Manual on Disposal of Refinery Wastes, Vol. V: Sampling and Analysis of Waste Gases and Particulate Matter, American Petroleum Institute, Washington, DC., 1954.
- 11.2 Tentative Method of Determination of Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas, Natural Gas Processors Association, Tulsa, Okla. NGPA Publication No. 2265-65, 1965.

- 3 Knoll, J.E., and M.R. Midgett. Determination of Hydrogen Sulfide in Refinery Fuel Gases, Environmental Monitoring Series, Office of Research and Development, USEPA, Research Triangle Park, N.C. 27711, EPA 600/4-77-007.
- 1.4 Scheill, G.W., and M.C. Sharp. Standardization of Method 11 at a Petroleum Refinery, Midwest Research Institute Draft Report for USEPA, Office of Research and Development, Research Triangle Park, N.C. 27711, EPA Contract No. 68-02-1098, August 1976, EPA 600/4-77-088a (Volume 1) and EPA 600/4-77-088b (Volume 2).

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April 4, 1983

REPLY TO:

Miami

Mr. John Svec, P.E.
Department of Environmental
Regulation
Bureau of Air Quality Management
2600 Blair Stone Road
Tallahassee, Florida 32301

DER
APR 05 1983
BAQM

Re: Agrico South Pierce Application No. AC53-55780

Dear John:

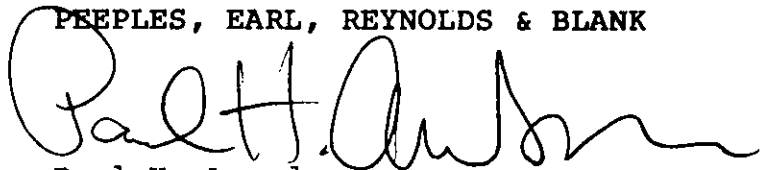
Recently we provided either you or Steve Smallwood with a particle size report on the dustfall at Burza and some materials on the control efficiency of water sprays.

We would request that these materials be made part of the above file. Enclosed are copies of those materials for this purpose.

Please let me know if you have any questions or if I can be of further assistance.

Sincerely,

PEEPLES, EARL, REYNOLDS & BLANK



Paul H. Amundsen
For the Firm

PHA:yp
Encls.

PEEPLS, EARL, REYNOLDS & BLANK

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March 16, 1983

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REPLY TO:

Miami

Mr. Steve Smallwood
Chief, Bureau of Air Quality
Management
Department of Environmental Regulation
2600 Blair Stone Road
Twin Towers Office Building
Tallahassee, FL 32302

Re: Control Efficiencies of Water Sprays on Solid Sulphur

Dear Steve:

It is my understanding that the Department has recently received a report estimating control efficiencies for water sprays for solid sulphur. It is also my understanding that this material was filed on behalf of Occidental in their proposal to vat sulphur at Swift Creek.

Because such control efficiency estimates may impact the ultimate outcome of the sulphur rulemaking inquiry, I put together the enclosed compilation of documents, submittals, and testimony in prior sulphur proceedings which bear upon the control efficiencies of water sprays for solid sulphur. The attached materials strongly suggest that the assumption of 90% is too high.

Mr. Steve Smallwood
March 16, 1983
Page 2

I hope these materials are of assistance. If you have any questions, please feel free to call. We look forward to working with you and your staff in rulemaking and other related matters.

Very truly yours,

PEEPLS, EARL, REYNOLDS &
BLANK, P.A.



William L. Earl
For the Firm

WLE/nir

Enclosures

CONTROL EFFICIENCIES OF WATER SPRAYS ON SOLID SULPHUR
-- COMPILATION OF MATERIALS OF RECORD --

March 11, 1983

I. Letter from EPA Commenting Upon Water Spray
Control Efficiencies for Solid Sulphur

In a letter from Gilbert H. Wood of EPA, dated March 31, 1978, (attached at Tab A), Mr. Wood discussed the varying estimates of 50% to 90% control efficiencies for sulphur water sprays and observed:

Considering the lack of information and the imprecise nature of the quoted control efficiencies, one can only estimate that the actual efficiency is probably in the range of 50 to 90 percent.

Mr. Wood further stated that the documents where these values appear "emphasize that the values should be used with caution and that the actual values may vary significantly from these estimates depending on the actual materials and the operating conditions." This EPA analysis was undertaken in response to a request from Agrico's consultant. The letter is part of the DER record in the Agrico case. (Freeport Exhibit #37)

II. Testimony and Submittals in LRACT Rulemaking

Dr. John B. Koogler, an experienced engineer in the field of air pollution, was retained by Freeport as a consultant for the LRACT rulemaking proceeding. Dr. Koogler's February 17, 1978, submittal in this rulemaking proceeding (attached at Tab B) includes the following statement:

In evaluating water sprays, I assumed a control efficiency of 50 percent for all activities.

Although Dr. Koogler apparently did not consider the use of a wetting agent, he nevertheless characterized his estimates as "in the reasonable to conservative range." On March 15, 1978, Dr. Koogler presented testimony before the DER's LRACT ad hoc committee on sulphur handling. Dr. Koogler testified:

Loading on the pile, Agrico assumed 90 percent and I assumed 50 percent. Again, you are taking a dry material and dropping it onto a pile. This material will have been sprayed with water, but again, water sprays aren't that effective on the control of dust, especially when you consider the fact that sulfur is not a wettable material.

Transcript at pp. 33-34, attached at Tab C.

III. Findings of Fact in the Agrico Case

At the Agrico hearing, considerable expert testimony focused on the issue of control efficiencies. Agrico's proposed finding of fact No. 72, which stated that 90% was an appropriate control efficiency for the water sprays with a wetting agent, was expressly rejected by the hearing officer as "not established by the evidence."

Affirmative findings of fact by the Department were consistent with the hearing officer's rejection of 90% as an appropriate control efficiency. For example, the following facts were found:

101. That maintaining solid-form sulphur in a moist condition is difficult.

108. That the addition of a surfactant to water sprays makes little or no difference in pollution control efficiency.

Freeport Proposed Findings of Fact Nos. 101 and 108.

The finding of ineffectiveness of a surfactant on sulphur was based upon the testimony of two expert witnesses from Canada and

an official from Vancouver Wharves, all of whom appeared on Agrico's behalf. See Transcript at 1011, 1166, 1221, 1254.

* * * * *

Based upon the foregoing materials on file with DER, it would appear that an estimated 90% control efficiency is unrealistic for solid sulphur handling.

Attachments

FX-37

5/11/77

Mr. Allen H. Fitzgerald, Director
 Air Quality Management Division
 William Prothero Engineering, Inc.
 Resources Sciences Center
 600 North Yale Avenue
 Tulsa, Oklahoma 74136

Dear Mr. Fitzgerald:

In response to your letter of 1/14/77, letter to Mr. John Haines, I have reviewed your seven enclosures, pertaining to estimates of particulate emissions from a proposed wet prill sulfur receiving facility in Hillborough County, Florida.

For the record, I have reviewed wet prill sulfur. Also, the document, Technical Guidance for Control of Industrial Process Fugitive Particulate Emissions, EPA-450/3-77-010, March, 1977, does not address wet prill sulfur. However, in the absence of specific data, one should be able to estimate emission rates for wet prill sulfur receiving operations by using data for similar material handling operations and using reasonable engineering judgment.

All seven of your enclosures appear to have quite similar estimates for the annual uncontrolled particulate emissions, despite different estimating methods. The differences in the enclosures are related to the assumed control efficiency and the consideration of peak emissions and ambient concentrations.

Basically, the proponents of wet prill sulfur claim that the chemical wetting agent used in the prill receiving process facilitates subsequent water washing to such a degree that 90 percent of the fugitive particulate emissions are controlled during subsequent handling and storage of the prills. Conversely, the opponents of wet prill sulfur postulate that the intermediate stage does not significantly improve subsequent water spray washing, a maximum of 50 percent of the fugitive particulate emissions are controlled. One should note that both the 50 percent and the 90 percent values are attributed to AP-42 and Technical Guidance for Control of Industrial Process Fugitive Particulate Emissions. The values are for well-designed and operated systems. Both documents emphasize that these values should be used with caution and that the actual values may vary significantly from these estimates depending on the actual material handling and operating conditions.

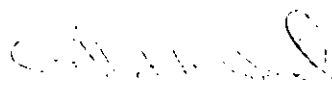
A precise value for the control efficiency of the system for the proposed facility cannot be determined without additional information. For example, the enclosures only state that a wetting agent will be used. No information is given in regard to the chemical formula, the performance, or even the name of the agent. Laboratory wind tunnel tests could be conducted to estimate the performance in regard to windblown dust. Ambient air monitoring of wet prill loading operations at other sites could also be conducted. Considering the lack of information and the imprecise nature of the quoted control efficiencies, one can only estimate that the actual efficiency is probably in the range of 50 to 90 percent.

An analysis of the potential impact of the particulate emissions should include not only the annual average ambient concentrations but also the short-term ambient concentrations. Only the Lundgren, et al, analysis and the Kogler, et al, analysis considered the peak emissions during ship unloading and the resultant peak ambient concentrations in any significant detail. None of the enclosures include adequate discussion of how the dispersion of the emissions was modeled.

In summary, additional information is necessary before one can determine precise particulate emission rates and resultant ambient air concentrations for the proposed wet prill sulfur receiving facility. The Florida Department of Environmental Regulation notice of intention to issue construction and operation permits for the proposed facility includes the requirement for an ambient air monitoring program. Such a program should provide the information necessary to properly evaluate the proposed operation and should also expand our data base to allow more precise estimations for future proposals.

We appreciate this opportunity to discuss our views to you and trust that these comments will be helpful to you in your evaluation of the various procedures used by the several consultants who have assessed the potential impacts of the proposed facility.

Sincerely yours,



Gilbert H. Reid
Industrial Studies Branch
Testing Standards and
Engineering Division

cc: Mr. John Barnes, Director
Mr. Tom Mahas, Deputy Director
✓ Mr. John B. Kogler
Sholtes and Kogler
Environmental Consultants
Mr. Rodger Stewart, Director
Hillsborough County Environmental
Protection Commission



SHOOK & MCKEE, INC. ENVIRONMENTAL CONSULTANTS
1213 N.W. 6th Street Tampa, Florida 33601 (804) 377-5822
SHEC 229-77-01

February 17, 1978

Florida Department of Environmental
Regulation
LRACT Ad Hoc Sulfur Committee
7601 Highway 301 North
Tampa, FL 33610

*Copy of letter
submitted to
LRACT
by Shook & McKee*

Re: Particulate Matter from Term Solid Sulfur Unloading
Facilities

Gentlemen:

In my initial document submitted to the Ad Hoc Sulfur Committee dated February 8, 1978 I pointed out the potential for fugitive dust emissions created by the handling of solid sulfur. In the document I made an assumption as to the "dust" content of prilled sulfur as received in Tampa.

Since preparing that material I have had the opportunity to review additional particle size data for various forms of solid sulfur and have also had the occasion to discuss "fugitive dust" further with the EPA Office of Air Quality Planning and Standards in Research Triangle Park, North Carolina.

The review and discussion have confirmed that the figures used in my original estimate of particulate matter emissions were conservatively low.

I assumed a dust content of prills, as received in Tampa, of 6 percent and an associated silt content of 1.4 percent. Dust is defined as particles less than 50 mesh (297 microns) and silt as particles less than 200 mesh (74 microns). Laboratory strength tests have shown that the dust content of water formed prills commonly exceeds 6 percent. Live tests, actual field measurements, also show the dust content of water formed prills to exceed 6 percent. In a report prepared by the Sulfur Development Institute of Canada (SUDIC) for the Sulfur Industry Training Committee entitled Sulfur Forms and Forming Processes (June 1977) and an Addendum thereto (Sept. 1977), the dust content of water formed prills was measured to be 6.5 percent in the hold of a ship. After transport to Tampa and off-loading there the dust content will be expected to be considerably higher.

Based on typical measured particle size distributions for sulfur dust, sulfur with 6 percent dust content passing a 50 mesh sieve can be expected to have 1.5 percent dust content passing a 200 mesh (74 microns) sieve.

In estimating fugitive dust emissions from materials handling there appears to be differing opinion. EPA uses the quantity of material less than 75 microns in diameter as a measure of fugitive dust potential (1.4 percent in my projections).

SUBIC & Alberta Sulfur Services, Ltd., however, use the quantity of material less than 297 microns as that which can cause fugitive dust problems (6 percent in my projections). As a point of reference, the high-volume sampler method (4-704-501 used by EPA is designed to collect particles less than 100 microns in diameter.

From the above, it is apparent that the fugitive emission rate I estimated using EPA criteria is conservatively low. Using Canadian criteria the potential for fugitive emissions could be four times higher.

If it is assumed that particles larger than 75 microns contribute significantly to fugitive dust generation it must also be recognized that some of these particles will settle out relatively close to the source because of their size and the associated settling velocity. The distance the larger particles (greater than 75 microns) travel will be dependent upon wind speed and the mechanical turbulence associated with higher wind speeds. In Tampa, the wind speed can be expected to exceed 20 mph about one percent of the time, and 12 mph 10 percent of the time. The annual average wind speed in Tampa is about nine miles per hour.

Because of sedimentation, ground-level concentrations of sulfur dust downwind of the proposed facilities would not be expected to increase in direct proportion to the estimated emissions. Even with the conservative assumptions considered in my initial calculations, i.e., fugitive emissions result only from particles less than 75 microns in diameter and good engineering control practices, the estimated ground-level particulate matter levels at typical receptor distances exceed the 24-hour particulate matter ambient standard of $150 \mu\text{g}/\text{m}^3$ by an order of magnitude. Increases in these levels resulting from less conservative emission estimates would only be accentuated because of the extreme levels involved.

When estimating the impact of sulfur emissions I assumed a control effectiveness I thought reasonable based on data published by EPA and my discussions with EPA staff. I assumed that fugitive emissions would be controlled by means proposed by the two permit applicants. In evaluating water sprays, I assumed a control efficiency of 50 percent for all activities. EPA reports the effectiveness of water sprays to be in the range of 34 to 50 percent depending upon the application. The point of greatest emissions in handling solid sulfur results is at dockside during ship unloading. At this point it would be naive to think water sprays could uniformly wet all the sulfur and provide the maximum degree of effectiveness (50 percent) for that type of control. At this point, therefore, water sprays would be expected to be less than 50 percent effective.

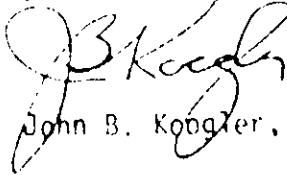
IRACT Ad Hoc Sulfur Committee
February 17, 1978
Page Three

As with other estimates used in my original projections I feel my estimates of control efficiency are in the reasonable to conservative range.

If there are any questions regarding this information or if further comment is required, I will be available at the Ad Hoc Committee meeting to respond. I have attached a copy of my professional resume for your general information.

Very truly yours,

SHOLTES & KOOGLER
ENVIRONMENTAL CONSULTANTS



John B. Koogler, Ph.D., P.E.

JBK:vw

Attachments: Mailing list
Resume

1 with the EPA people who have worked on these fugitive
2 dust projects, their estimates for the effectiveness
3 of water control, and again, this is not related to
4 sulfur handling facilities because there is no place
5 in this country where EPA could have done any work
6 on sulfur handling facilities. But, based on the
7 effectiveness of water sprays on bulk handling
8 systems, they estimate 38 to 50 percent. So, in
9 my estimate, I assumed 50 percent. And, considering
10 the fact that the sulfur dust, in my opinion, is
11 going to be dry, it's going to have less than one
12 and a half percent moisture, I think 50 percent is
13 a graciously conservative estimate.

14 On the transferring and conveying, this is
15 transferring material from the dockside hopper to
16 the open storage pile. Agrico assumed an efficiency
17 of 90 percent. I assumed 87 percent and I arrived
18 at mine through going through a series of steps and
19 adding it all up and it comes up to 87 percent.
20 So, 87 percent versus 90 percent is not bad.

21 Loading on the pile, Agrico assumed 90 percent
22 and I assumed 50 percent. Again, you are taking
23 dry material and dropping it onto a pile. This
24 material will have been sprayed with water, but as
25 water sprays aren't that effective on the control

1 dust, especially when you consider the fact that
2 sulfur is not a wettable material.

3 I think this movie Mr. McNamara showed us,
4 showing the sulfur floating on the water surface,
5 is an indication that the dust is not wettable.

6 So, going on down on the traffic, Agrico
7 assumed 90 percent control by water spray. Again,
8 I assumed 50 percent.

9 Items loading onto the pile, traffic around
10 the pile, loading out from the pile and wind erosion
11 are all emission factors relating to the open
12 storage pile. I have assumed 50 percent control by
13 using water spray for all of these activities.
14 Agrico assumed 90 percent except for load out and
15 they assumed 80 percent control. And on truck
16 loading, this is loading the solid material from
17 the pile to the truck for transport from the facility,
18 Agrico assumed 90 and I assumed 85. That's reasonably
19 close. So, adding it all up, Agrico comes with a
20 controlled emission, this is during unloading the
21 ship, of 52 pounds an hour and I come up with an
22 estimate of a hundred and twenty-seven pounds an
23 hour.

24 One thing you need to keep in mind is that
25 what you are talking about in handling the solid

PARTICLE SIZE ANALYSIS OF DUSTFALL
AT PRILLED SULFUR PRODUCTION PLANT

TRC Technical Memorandum 1945-J51-04
Prepared by: Gale F. Hoffnagle, CCM
John E. Yocom, P.E.

On October 28, 1982, TRC Environmental Consultants, Inc. obtained a representative sample of prilled sulfur at the Burza Resources, Ltd., production plant in Fort McMurray, Alberta. During the period samples were being collected, the vehicle used by the sampling team, a van, was observed to have received deposits of sulfur dust on its surface from a nearby railcar loading operation. This memorandum describes that dustfall sample and its subsequent particle size analysis.

Setting

The van was parked approximately fifty feet directly downwind from a railcar loading operation. Wind direction observations on-site and Canadian Weather Service data from the nearby Fort McMurray Airport confirm that the winds blew directly from the railcar loading point to the van. The windspeeds observed at the airport were 10 miles per hour. Railcar loading was being carried out from a simple conveyor belt with an approximately fifteen foot drop to the top of the open-topped railcar. No other sulfur handling operation was directly upwind of the van. Figure 1 shows the general layout with wind direction and speed. The railcar loading operations which resulted in the deposits on the van took place over approximately 45 minutes. The prill being loaded had just been produced and the surface moisture content was approximately 3.5% after drying, prior to entering the conveyor to the railcar (from the bottom of the storage silo, where the sample for this moisture content analysis was taken, the prill proceeds on one conveyor belt to a simple transfer point thence onto the railcar loading conveyor). Since

the point of impact of the falling stream of prill into the railcar was well below the sides of the car, little "splash" dust or wind erosion was expected. Although visible emissions could not be identified while looking at the falling prill, when standing in front of the van and using the back-drop of the dark railcars, sulfur particles could be seen falling to the ground. The mechanism for sulfur dust being stripped by the wind from the falling stream of prill and thence deposited on the van appeared to be, in part, droplets of water with entrained sulfur dust.

Sample Collection

To analyze the dustfall on the van, an approximately one-foot square area was marked out on the hood. The material in the area was gathered by gently scraping the surface with a plastic credit card (no other suitable method being available) and placed in a small plastic film case. Approximately 0.23 grams of sulfur dust was collected this way but significant quantities of sulfur dust blew away in the process. Once released from the dried droplet on the hood, many particles could and did readily become airborne during sampling. It is approximated that more than 50% of the sulfur in the one-foot square was not collected.

Sample Analysis

Upon return to TRC's laboratories the sample was weighed and two small portions of the sample were separated by quartering for microscopic analysis. The first portion of the sample was used to prepare slides for photomicrographs using fast developing film. Eight photomicrographs accompany this report including one of a reticule so that the size of the particles in the photomicrographs can be compared. The entire width of the reticule is 1000 microns, each major division is 100 microns and each of the finest

divisions is 10 microns.

The largest particle in these photomicrographs is about 1000 microns in its longest dimension. Most of the rest of the particles are less than 200 microns. Some of the photomicrographs show the large number of smaller particles. Particles less than 10 microns do not show up on any of the photomicrographs but several of the pictures are too dark to show small particles. However, as noted below, particles of this size range could be detected visually and were counted.

The second portion of the sample was used to prepare slides for manual sizing and counting. Using the reticule in the photomicrograph and another reticule with finest divisions to 16.25 microns, a total of 5618 particles were sized visually and the results are shown in Table 1.

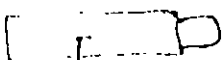
The particle size ranges were selected to be consistent with the reticule. These results show that the majority of particles are small and are expected to become airborne. For instance, more than 99% of the particles are less than 100 microns and more than 99.93% of the particles are less than 300 microns.

TABLE I

PARTICLE SIZE DISTRIBUTION
SULFUR DUSTFALL ON VAN
BURZA RESOURCES

<u>Particle Size Range (microns)</u>	<u>Number of Particles in Range</u>	<u>Percentage of Total Particles</u>	<u>Cumulative Percentage (%)</u>
Less than 16.25	5070	90.25	90.25
16.25 - 32.5	282	5.02	95.27
32.5 - 48.75	119	2.12	97.39
48.75 - 65.0	54	.96	98.35
65.0 - 81.25	39	.69	99.04
81.25 - 113.75	27	.48	99.52
113.75 - 162.5	15	.27	99.79
162.5 - 243.75	8	.14	99.93
243.75 - 325	3	.05	99.98
325 - 487.5	<u>1</u>	<u>.02</u>	100
	5618	100.00	

Sulfur
Truck



Sulfur
Tank

Water
Tank

Process
Building

Prill
Pile

Storage
Silo

Dryer

North

Transfer
Point

Van

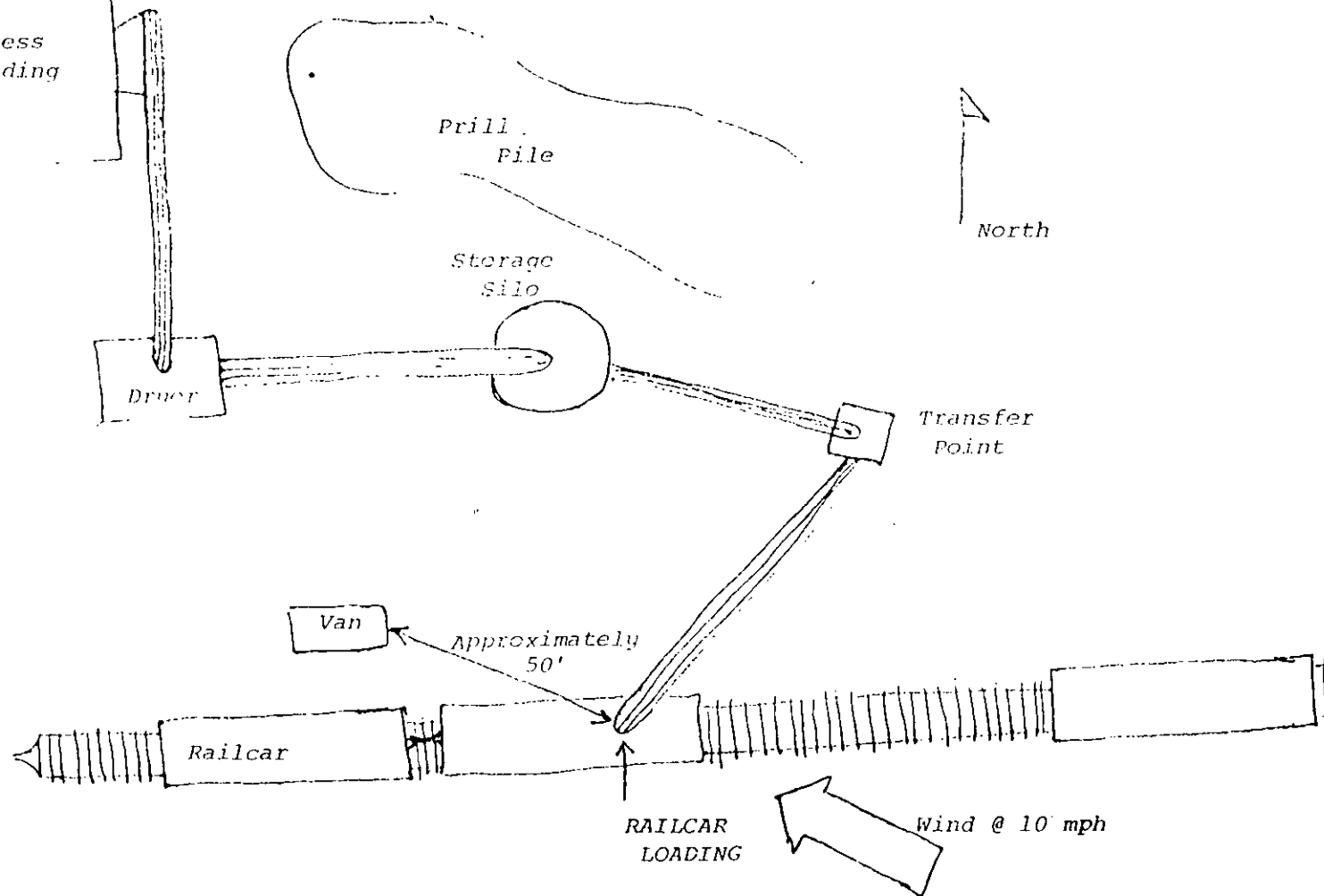
Approximately
50'

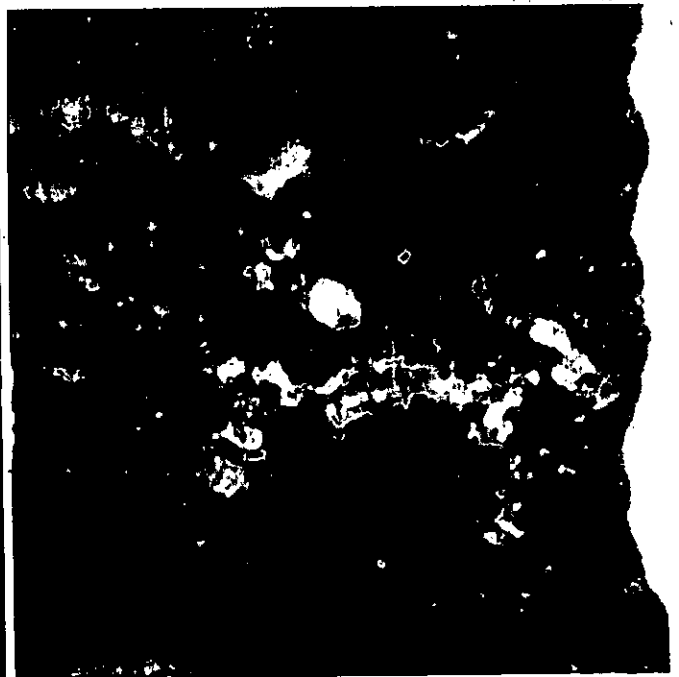
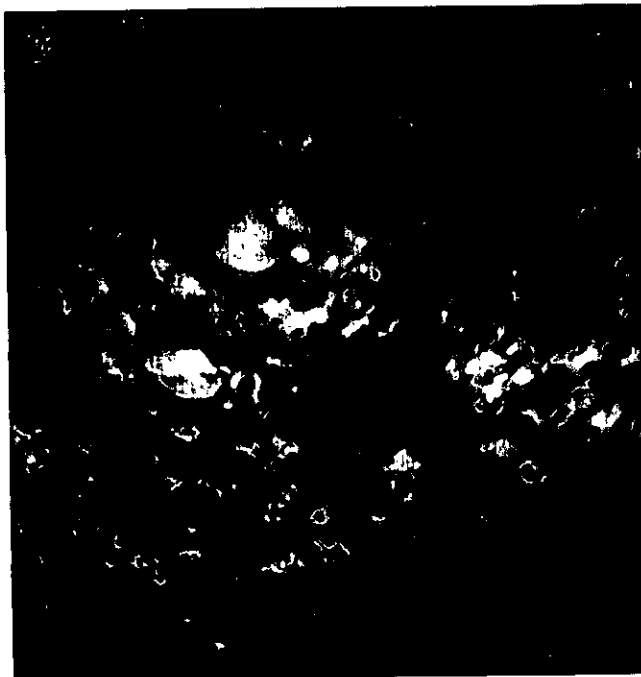
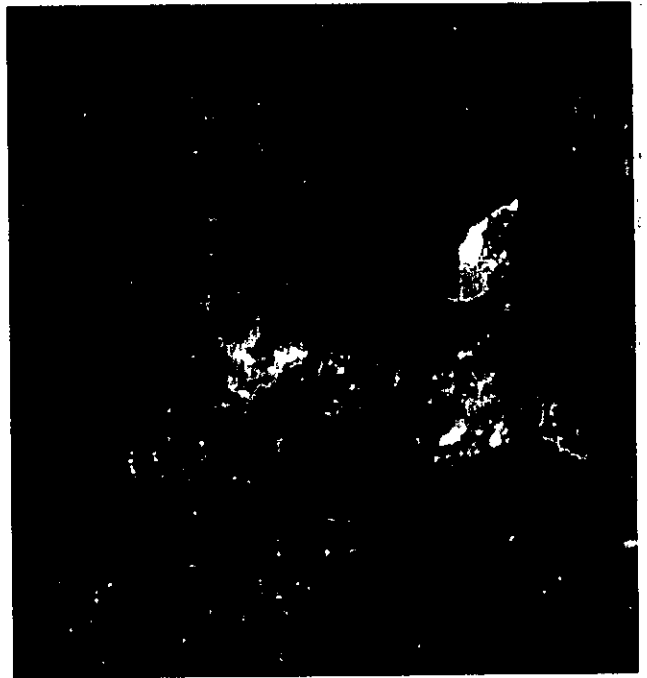
Railcar

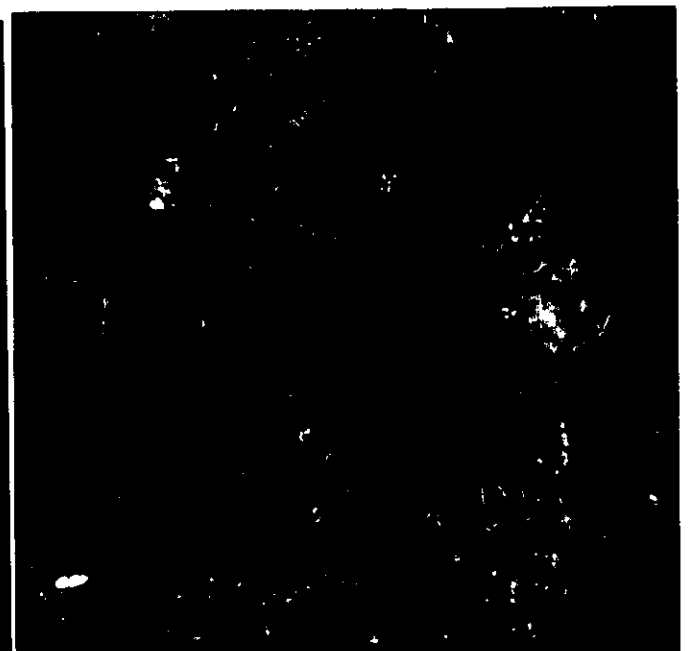
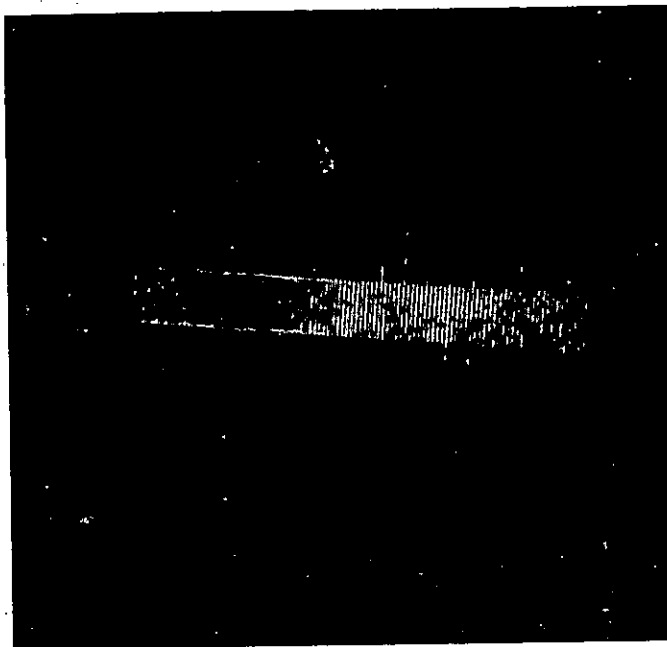
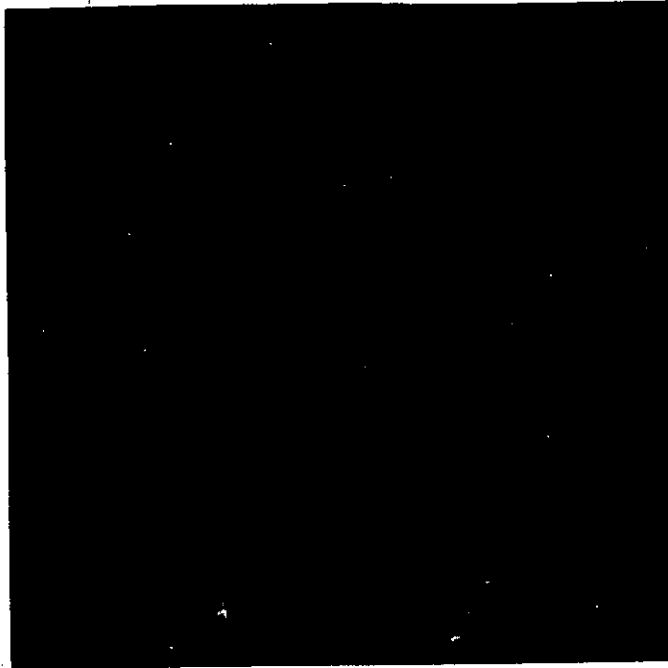
RAILCAR
LOADING

Wind @ 10 mph

Figure 1: Sketch of Bureau Resources
Layout (not to scale)







ECO/INTERFACE EVALUATIONS

Hal Scott

March 28, 1983

Mr. Steve Smallwood, P. E.
Chief, Bureau of Air Quality Management
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32301-18241

DER

APR 01 1983

Dear Steve:

BAQM

I appreciated the amount of time and effort which you and John Svec invested in the meeting on March 21st. Despite the obvious areas of disagreement that exist I felt the meeting was a constructive one.

In reviewing the notes I took, I remembered two topics that I had hoped we might be able to develop further, but which lack of time prevented our returning to.

First, you mentioned the extra energy required for each additional percent of moisture associated with the sulfur when remelting occurs. I am aware of that fact, but I was not sure that you were aware that the energy source for remelting at South Pierce is waste heat.

Second, you made note of the fact that "at 3% moisture prilled sulfur tends to dry in a day to a week." If I remember correctly, that conclusion was based on some of the data Dale Lundgren provided based on exposing a thin layer of sulfur to the air.

Every pile of wet prilled sulfur I have seen was wet despite the fact that none of the facilities was equipped with sprays to keep the material wet. You will recall that when you sampled a four month old pile of prilled material at Coastal Sulphur's facility in Houston you found it very wet an inch or so under the surface. That wetness continued on into the pile. The rain which Dennis Varner said had occurred a day or two previously would not explain that condition.

Agrico has a consulting firm studying a large pile of prilled sulfur to determine its drying characteristics. That information will be shared with you as soon as it is available.

Although I know you are aware that the way in which a large pile of wet prilled sulfur dries out can in no way be predicted by reference to the drying characteristics of a thin layer of the same material, I wanted to put those thoughts on record.

Sincerely,

Hal Scott

2918 WALNUT STREET / ORLANDO, FLORIDA 32806 / 305-894-6100

ECO/INTERFACE EVALUATIONS

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March 28, 1983

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Hal Scott

2918 WALNUT STREET / ORLANDO, FLORIDA 32806 / 305-894-6100

CHARACTERIZATION OF SULFUR PRILL
FROM CARGO SHIP GUNVER CORD (MOROCCO)
IN ACCORDANCE WITH SUDIC METHODS

BY

GEORGE V. ASEFF
LAW ENGINEERING TESTING COMPANY
LETCO JOB NUMBER G-8657



CHARACTERIZATION OF SULFUR PRILL
FROM CARGO SHIP GUNVER CORD (MOROCCO)
IN ACCORDANCE WITH SUDIC METHODS
LETCO JOB NUMBER G-8657

As authorized by Bill Earl, Esquire, the sampling and testing of a type of Fletcher sulfur prill was conducted in accordance with the standardized procedures published in the Sulphur Development Institute of Canada (SUDIC) 1978 Edition.

The purpose of the work was to characterize a Fletcher type sulfur prill reportedly sampled from cargo hold #3 of the Vessel Gunver Cord and rail car at Morocco over the period 12/29/82 through 1/3/83. The characterization tests included:

SUDIC METHOD

TEST PROCEDURE

S1-77

Sampling

S3-77

Particle Size Distribution by Washing the Fines prior to Sieving and Calculation of Fines on Dry Weight Basis.

S10-77

Surface Water Content of Sulfur Form by Constant Weight Determinations by Drying at $75 \pm 5^{\circ}\text{C}$ (4.1).

Trapped Internal Water Content by Constant Weight Determination at $75 \pm 5^{\circ}\text{C}$ followed by Karl Fisher Titration Procedure (4.2).

SAMPLING OF A FLETCHER - TYPE SULFUR PRILL
FROM CARGO SHIP GUNVER CORD AND RAIL CAR; MOROCCO

Mr. John Yocum, TRC, reports that all of the sampling of the Fletcher type prill was done by him. Representative sampling of the sulfur prill was done over the period

March 31, 1983
Page Two

12/29/82 through 1/3/83 from three different levels in cargo ship Gunver Cord, hold #3 and open rail car in Morocco. Mr. Yocom further reports that the shipment originated from Vancouver, Canada.

As sampled, the prill was taken and stored in thick wall, wide mouth plastic bottles with screw tops except for two samples stored in double wall bags. On 2/17/83 at TRC Laboratories in East Hartford, Connecticut G. V. Aseff reviewed each prill sample, summarized the label information and assigned test numbers to the original sequence sample numbers.

Data concerning the sampling of the sulfur prill are summarized in Table I.

VISUAL OBSERVATIONS OF PRILL SAMPLES BEFORE MIXING

The observations of the sulfur samples in the containers showed that the samples appeared to be 100 percent prill form with a relatively large range of particle sizes. The amount of fines appeared to be significant.

When the containers were opened for combining samples at the same cargo level, the samples appeared to be 100 percent prill. No slate portions were observed. Individual particles

of prill appeared to be abraded yielding a marketedly smooth surface as contrasted to the as-manufactured surface finish. These observations applied to Levels A, B and C of prill samples from hold #3 as well as the prill sample from the rail car (top surface). Some discoloration was observed in the sample from the rail car.

MIXING AND QUARTERING

The sulfur prill samples were then combined, mixed and quartered as per SUDIC Method S-1, as follows:

LEVEL A (Top) Hold #3, samples 1, 2, 3 and 4.

LEVEL B (Middle) Hold #3, samples 5, 6, 7 and 8.

LEVEL C (Bottom within 1 ft) Hold #3, samples 9, 10 and 12.

The combined samples from each level were quartered as required to obtain about 500g per sample (triplicate) for each level.

At level C, test samples 3.1 C-1, 3.1 C-2 and 3.1 C-3 upon visual examination during quartering showed less fines as compared with levels A and B. The test samples after quartering are shown as follows:

<u>LEVEL</u>	<u>TEST SAMPLE NUMBERS</u>
A (Top)	3.1 A-1 3.1 A-2 3.1 A-3

<u>LEVEL</u>	<u>TEST SAMPLE NUMBERS</u>
B (Middle)	3.1 B-1 3.1 B-2 3.1 B-3
C (Bottom)	3.1 C-1 3.1 C-2 3.1 C-3

The particle size distribution of sample 3.2 was comparable to samples 3.1A, 3.1B and 3.1C except for a contaminated condition.

PREPARATIONS AND TESTS PER SUDIC METHODS

The approximate 500g samples of as-received materials 3.1 A-1, 3.1 A-2, 3.1 A-3; 3.1 B-1, 3.1 B-2, 3.1 B-3; 3.1 C-1, 3.1 C-2, 3.1 C-3; 3.2-1, 3.2-2, 3.2-3) were placed in an oven at 75°C \pm 5°C after initial weighing and prior to drying to constant weight to determine surface water as per SUDIC S-10 (4.1).

At constant weight, as recorded for each approximate 500g sample, a small portion was taken (25 to 50g) for Karl Fischer method for trapped water as per SUDIC S-10 (4.2).

The balance of each 500g sample was then washed down to remove fines less than 50 mesh, air dried and then dried at 75° \pm 5°C to constant weight. Samples 3.1 A-1, 3.1 B-1, 3.1 C-1 and 3.2-2 were individually sieved per SUDIC S3-77 for particle size determinations. The weight of fines less than 50 mesh was added to the net weight of sieved material

SUDIC S-10, TITRATIONS - KARL FISCHER

The water equivalent of the Karl Fischer reagent was determined by the use of 15 μ L (15 mg) of high purity water and finding the equivalent volume of reagent to reach the end point. Results are shown below:

<u>RUN NO.</u>	<u>AMOUNT WATER (15 mg)</u>	<u>EQUIV. KF REAGENT (ml)</u>
1	15 mg	2.25
2	15 mg	1.85
3	<u>15 mg</u>	<u>2.15</u>
AVG	15 mg	2.08

$$2.08 \text{ ml}_{\text{KF}} = 15 \text{ mg H}_2\text{O}$$

$$1.00 \text{ ml}_{\text{KF}} = 7.21 \text{ mg H}_2\text{O}$$

$$\text{KF FACTOR} = \frac{7.21 \text{ mg H}_2\text{O}}{\text{ml KF}}$$

The correction factor, Karl Fischer Reagent, for the amount of water in the "anhydrous" methyl alcohol as used in SUDIC S-10 (4.2) was determined. Results are shown below.

<u>RUN NO.</u>	<u>AMT. "ANHY." METHYL ALC. (ml)</u>	<u>AMT. KF REAGENT (ml)</u>
1	25	0.40
2	25	0.40

Since 0.40 KF reagent was equivalent to 25 ml "anhydrous" methyl alcohol, then the correction factor of 0.08 KF was

calculated as equivalent to 5 ml methyl alcohol; the volume of extract used in each determination for trapped water per SUDIC S-10.

CHARACTERIZATION TESTS

All characterization tests on the prill were done in accordance with applicable SUDIC methods as previously stated.

SURFACE WATER AS FUNCTION OF DEPTH

Mixed and quartered test samples taken from the top, middle and near bottom elevations in hold #3 were placed in an oven at $75^{\circ}\text{C} \pm 5^{\circ}\text{C}$ after initial weighing, to dry to constant weight. The difference between the initial weight and constant weight divided by the constant weight and times 100 gave the percent surface water as per SUDIC S-10 (4.1). Measured values obtained for the percent surface water for sulfur prill as a function of depth are as follows:

<u>SULFUR TEST SAMPLE</u>	<u>DEPTH IN HOLD #3</u>	<u>PERCENT SURFACE WATER</u>
3.1 A-2	Top	0.9
3.1 A-2	Top	1.3
3.1 A-3	Top	<u>1.4</u>
	AVG	1.2
3.1 B-1	Middle	1.3
3.1 B-2	Middle	1.5
3.1 B-3	Middle	<u>1.5</u>
	AVG	1.4

<u>SULFUR TEST SAMPLE</u>	<u>DEPTH IN HOLD #3</u>	<u>PERCENT SURFACE WATER</u>
3.1 C-1	Near Bottom	1.1
3.1 C-2	Near Bottom	1.1
3.1 C-3	Near Bottom	<u>0.9</u>
	AVG	1.0
3.2.1	Top Rail Car	1.9
3.2.2	Top Rail Car	2.1
3.2.3	Top Rail Car	<u>1.8</u>
	AVG	1.9

TRAPPED WATER AS FUNCTION OF DEPTH

Trapped water in the sulfur prill was determined by SUDIC S-10 (4.2) Method on a 25 to 50g portion taken from the larger lot as mentioned above. The 25 to 50g portion at constant weight was covered with "anhydrous" methyl alcohol and later ground. A 5 ml volume of the extract was titrated with Karl Fischer reagent to an end point. Trapped water content was calculated as per SUDIC S-10 (4.2) as a function of depth of sulfur sample as follows:

<u>SULFUR TEST SAMPLE</u>	<u>DEPTH IN HOLD #3</u>	<u>PERCENT TRAPPED WATER</u>
3.1 A-1	Top	0.13
3.1 A-3	Top	<u>0.13</u>
	AVG	0.13
3.1 B-2	Middle	0.17
3.1 B-3	Middle	<u>0.15</u>
	AVG	0.16

<u>SULFUR TEST SAMPLE</u>	<u>DEPTH IN HOLD #3</u>	<u>PERCENT TRAPPED WATER</u>
3.1 C-1	Near Bottom	0.13
3.1 C-3	Near Bottom	<u>0.17</u>
		AVG 0.15
3.2.1	Rail Car Top	0.13

PERCENT FINES LESS THAN 50 MESH AS A FUNCTION
OF DEPTH IN HOLD #3

The previously mentioned test samples from the top, middle and near bottom elevations of hold #3 and from the top of the rail car at constant weight were thoroughly washed to remove fines less than 50 mesh, air dried and oven dried to constant weight for a second time.

The difference in weights was representative of the fines less than 50 mesh. The percentages were calculated as follows:

<u>SULFUR TEST SAMPLE</u>	<u>DEPTH IN HOLD #3</u>	<u>PRESENT FINES 50 MESH</u>
3.1 A-1	Top	4.1
3.1 A-2	Top	5.6
3.1 A-3	Top	<u>6.7</u>
		AVG 5.4
3.1 B-1	Middle	7.1
3.1 B-2	Middle	6.8
3.1 B-3	Middle	<u>8.6</u>
		AVG 7.5

<u>SULFUR TEST SAMPLE</u>	<u>DEPTH IN HOLD #3</u>	<u>PRESENT FINES 50 MESH</u>
3.1 C-1	Near Bottom	4.0
3.1 C-2	Near Bottom	3.3
3.1 C-3	Near Bottom	<u>5.6</u>
		AVG 3.4
3.2.1	Rail Car	5.6
3.2.2	Rail Car	6.4
3.2.3	Rail Car	<u>6.3</u>
		AVG 6.1

SIEVE ANALYSIS AFTER WASH DOWN OF FINES
LESS THAN 50 MESH AS A FUNCTION OF DEPTH, HOLD #3

After wash down of fines less than 50 mesh and drying to constant weight, sulfur prill test samples 3.1 A-1 (Top), 3.1 B-1 (Middle), 3.1 C-1 (Near Bottom) and 3.2.2 (Rail Car Top) were sieved in accordance with SUDIC Method S-3.

Results of the sieve analyses are shown in Figures 1P through 4P.

SUMMARY

In summary, the approximate 50 pounds of Fletcher type sulfur prill were sampled by John Yocum, TRC from the vessel Gunver Cord, hold #3 and rail car at Morocco over the period of 12/29/82 through 1/3/83 and brought to TRC, Hartford, Conn.

March 31, 1983
Page Ten

The sulfur prill was sampled and characterized by G. V. Aseff, Law Engineering Testing Company in accordance with SUDIC Methods S1-77, S3-77 and S10-77. Custody of all samples has remained with G. V. Aseff through the entire characterization program. All of the laboratory sampling and testing associated with this prill was conducted either by G. V. Aseff or under his direct supervision.

Representations of the test data of the Morocco prill are attached as Table I and Figures 1P through 4P.

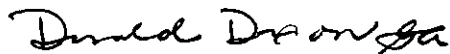
BY:

LAW ENGINEERING TESTING COMPANY



George V. Aseff, P.E.
Corporate Materials Consultant

REVIEWED BY:



Donald E. Dixon, P.E.
Corporate Materials Consultant

GVA:DED/ljh



JOB NO. G-8657 SHEET 1 OF 1

JOB NAME MOROCCO Sulfur Prill

BY G.V. Aseff DATE 3/15/83

CHECKED BY *[Signature]* DATE 3/17/83

LAW ENGINEERING TESTING COMPANY

geotechnical, environmental & construction materials consultants

396 PLASTER AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761

TABLE I - SUMMARY OF DATA ON SAMPLES OF TYPICAL SULFUR PRILL SHIPPED FROM VANCOUVER, B.C. AND SAMPLED FROM SHIP GUNVER CORD AND RAIL CAR AT MOROCCO 12/29/82 - 1/3/83

TEST SAMPLE NUMBER	ORIGINAL SAMPLE NO. (AS SAMPLED)	GROSS WEIGHT (g) * 165	LOCATION WHERE SAMPLED	LEVEL SAMPLED	SAMPLE CONTAINER	DATE SAMPLED	DIRECTION LOCATION SAMPLED
3.1.A-1 3.1.A-2 3.1.A-3	{ 1 } M 2 X 3 E 4 D	1440.0 1460.1 1468.6 1454.7	HOLD 3 LEVEL A (TOP) ↓	TOP ↓	PLASTIC BOTTLE ↓	12/29/82 ↓	WEST SOUTH EAST NORTH
3.1.B-1 3.1.B-2 3.1.B-3	{ 5 } M 6 I 7 X 8 D	1442.5 1445.3 1483.8 1410.0	HOLD 3 LEVEL B (MID) ↓	MID ↓	PLASTIC BOTTLE ↓	1/1/83 ↓	SOUTH SOUTH-MID NORTH-MID NORTH
3.1.C-1 3.1.C-2 3.1.C-3	{ 9 } M 10 I 11 X 12 D	1402.6 1406.7 6.4* 1429.5	HOLD 3 LEVEL C (BOTTOM) ↓	BOTTOM ↓	PLASTIC BOT. PLASTIC BOT. BAG PLASTIC BOT	1/1/83 ↓	SW NE SOUTH WEST
3.2.1 3.2.2 3.2.3	16	11.6*	LAND THIRD CAR	TOP	BAG	1/3/83	END OF CAR TOP

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

396 PLASTERS AVENUE N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 1P OF 4PJOB NAME Morocco Sulfur PrillBY G.V. Aseff DATE 3/15/83CHECKED BY G.V. Aseff DATE 3/19/83**3.1 A.1 PRILL FROM SHIP HOLD #3**
PARTICLE SIZE DISTRIBUTION
SUDIC 53-77Sulphur Form: PRILL, FLETCHER TYPE

Producer:

Sieve Size

Percent
Retained*Gross Sample Size: 447.3 g.

Date Formed:

1" (25.0 mm)

-

Date Received: 12/29/82

3/4" (19.0 mm)

-

Date Tested: 2/18/83

1/2" (12.5 mm)

-

Sieve Analysis: Wet ☒

3/8" (9.5 mm)

-

Dry ☐

No. 4 (4.75 mm)

16.1Fines Content (% Minus No. 50): 5.6 %

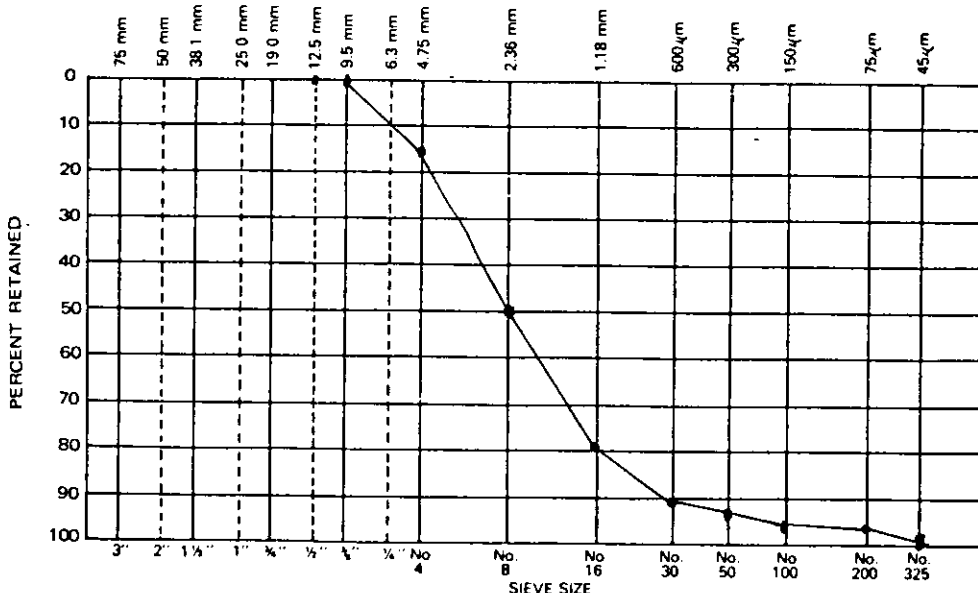
No. 8 (2.36 mm)

33.9Coefficient of Uniformity: 4.50

No. 16 (1.18 mm)

29.2Comments: WASHED FINES (%)No. 30 (600 μ m)11.4< 50 > 100 1.7No. 50 (300 μ m)4.0< 100 > 200 0.7

Minus No. 50

5.6< 200 1.8*To nearest
0.1%SIEVED FINES (%)< 50 1.4

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

396 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 2P OF 4PJOB NAME MOROCCO Sulfur PrillBY G.V. Aseff DATE 3/15/83CHECKED BY [Signature] DATE 3/19/83**3.1 B.1 PRILL FROM SHIP HOLD #3**
PARTICLE SIZE DISTRIBUTION
SUDIC 53-77Sulphur Form: PRILL, FLETCHER TYPE Percent
Producer: _____ Sieve Size Retained*Gross Sample Size: 408.39

Date Formed: _____

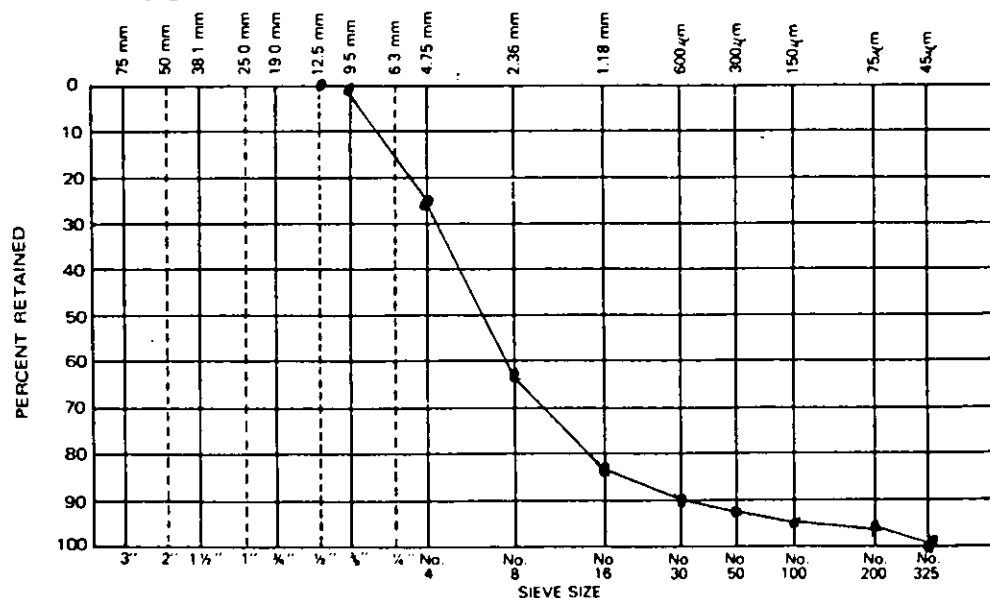
Date Received: 1/1/83Date Tested: 2/18/83Sieve Analysis: Wet ✓

Dry _____

Fines Content (% Minus No. 50): 7.4%Coefficient of Uniformity: ~5.92Comments: WASHED FINES (%)

<u>150 > 100</u>	<u>1.6</u>
<u>< 100 > 200</u>	<u>1.3</u>
<u>< 200</u>	<u>4.2</u>
<u>SIEVED FINES (%)</u>	
<u>< 50</u>	<u>0.3</u>

1"	(25.0 mm)	-
3/4"	(19.0 mm)	-
1/2"	(12.5 mm)	-
3/8"	(9.5 mm)	0.2
No. 4	(4.75 mm)	25.2
No. 8	(2.36 mm)	37.9
No. 16	(1.18 mm)	19.8
No. 30	(600 μ m)	7.0
No. 50	(300 μ m)	2.6
Minus No. 50		7.4

*To nearest
0.1%

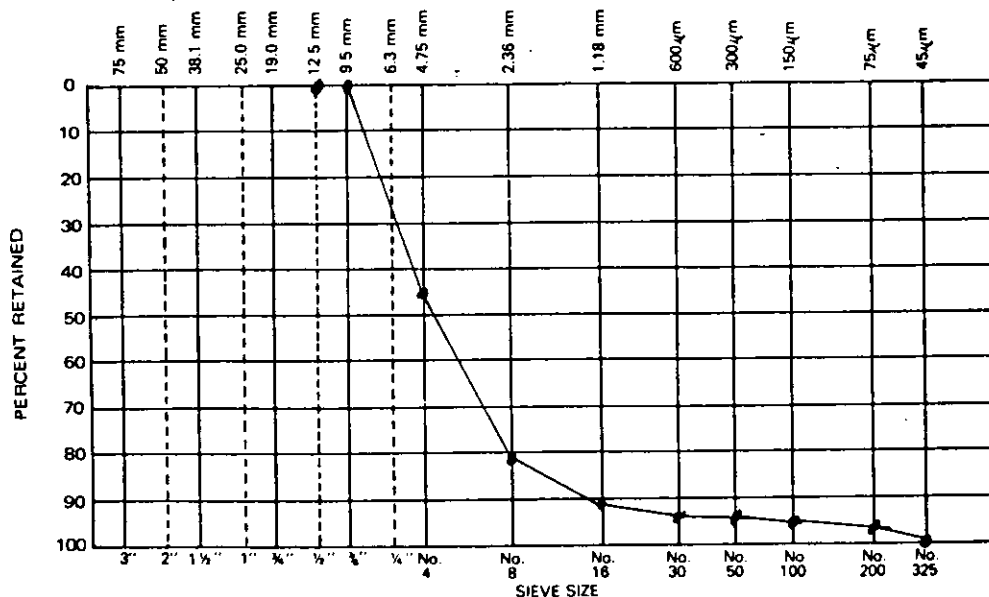
**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

396 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 3P OF 4PJOB NAME Morocco Sulfur PrillBY G.V. Aseff DATE 3/15/83CHECKED BY G.A. Aseff DATE 3/19/83**3.1.C.1 PRILL FROM SHIP HOLD #3**
PARTICLE SIZE DISTRIBUTION
SUDIC 53-77

Sulphur Form: PRILL, FLETCHER TYPE Percent Retained*
Producer: _____ Sieve Size
Gross Sample Size: 450.3 g.
Date Formed: _____
Date Received: 1/1/83
Date Tested: 2/18/83
Sieve Analysis: Wet ✓ Dry _____
Fines Content (% Minus No. 50): 4.2%
Coefficient of Uniformity: ~ 4.29 Minus No. 50

Comments: WASHED FINES (%)
< 50 > 100 0.84
< 100 > 200 0.80
< 200 2.33
SIEVED FINES (%)
< 50 0.20

*To nearest
0.1%

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

396 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 4P OF 4PJOB NAME Morocco Sulfur PrillBY G.V. Aseff DATE 3/15/83CHECKED BY [Signature] DATE 3/19/83**3.2.2 PRILL FROM RAIL CAR; TOP
PARTICLE SIZE DISTRIBUTION
SUDIC 53-77**Sulphur Form: PRILL, FLETCHER TYPE

Producer:

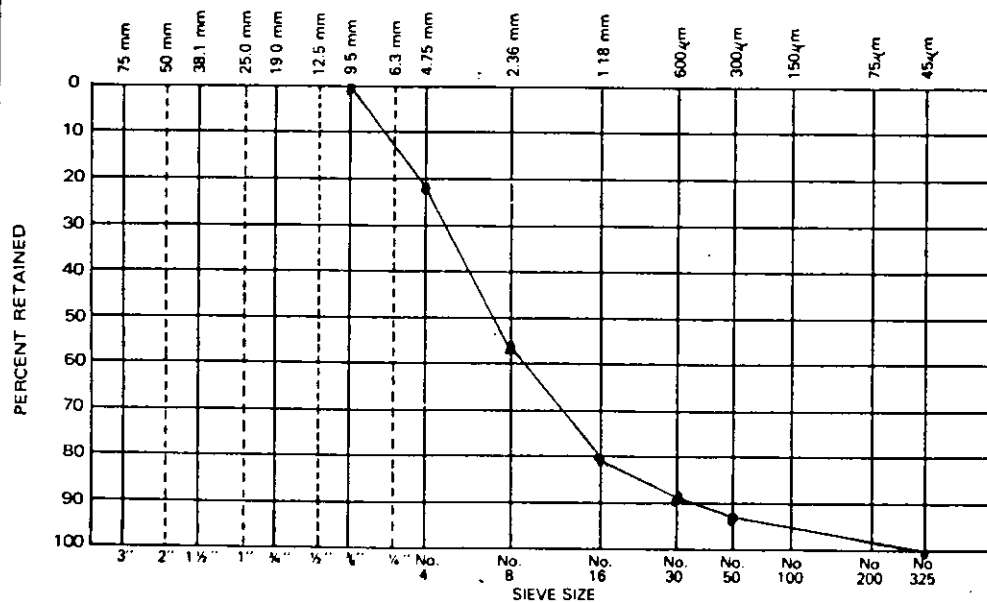
Gross Sample Size: 367.3 g

Date Formed:

Date Received: 1/3/83Date Tested: 2/18/83Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 7.0%Coefficient of Uniformity: 5.92

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	—
3/4" (19.0 mm)	—
1/2" (12.5 mm)	—
3/8" (9.5 mm)	0.25
No. 4 (4.75 mm)	23.25
No. 8 (2.36 mm)	34.41
No. 16 (1.18 mm)	23.25
No. 30 (600 μ m)	8.47
No. 50 (300 μ m)	3.40
Minus No. 50	6.97

*To nearest
0.1%

CHARACTERIZATION OF SULFUR PRILL

OBTAINED FROM BURZA

IN ACCORDANCE WITH SUDIC METHODS

BY

GEORGE V. ASEFF

LAW ENGINEERING TESTING COMPANY

LETCO JOB NUMBER G-8657



CHARACTERIZATION OF SULFUR PRILL
OBTAINED FROM BURZA
IN ACCORDANCE WITH SUDIC METHODS
LETCO JOB NUMBER G-8657

As authorized by Bill Earl, Esquire, the sampling and testing of a type of Fletcher sulfur prill was conducted in accordance with the standardized procedures published by the Sulfur Development Institute of Canada (SUDIC) 1978 Edition.

The purpose of the work was to characterize the sample of Fletcher type sulfur prill obtained from Burza, Ft. McMurray, Alberta, Canada on October 28, 1982. The characterization tests included:

SUDIC METHOD

TEST PROCEDURE

S2-77

Particle Size Distribution by
Dry Sieve Analysis

S3-77

Particle Size Distribution by
Washing the Fines Prior to
Dry Sieving

S4-77

Friability of Sulfur Form using
10 in. Diameter Tumbler Test

S5-77 Friability of Sulfur Form using
28 in. Diameter Tumbler Test

S10-77 Surface Water Content of Sulfur
Form by Constant Weight Deter-
minations by Drying at $75 \pm 5^{\circ}\text{C}$
(4.1).

Trapped Internal Water Content
by Constant Weight Determination
at $75 \pm 5^{\circ}\text{C}$ followed by Karl
Fischer Titration Procedure (4.2).

Total Water Content by Karl Fischer
Titration Procedure (4.3) and by
the Sum of Surface Water and
Trapped Internal Water (5.3).

SAMPLING OF FLETCHER - TYPE SULFUR PRILL

AT THE BURZA PLANT

FT. MCMURRAY, ALBERTA, CANADA

All of the sampling of the Fletcher type prill was done by
George Aseff in accordance with SUDIC Method S1-77.

Sampling of the sulfur prill was made on 10/28/82 at two
different transfer points at the BURZA Plant located in
Ft. McMurray, Alberta, Canada.

PLANT LOCATION NO. 1

Plant location number 1 at BURZA was considered as the
transfer point of the wet prill being released from the
prill tower into the conveyor belt to storage and/or to
partial drying. Sampling of the wet sulfur prill was made
from the conveyor belt at regular intervals and the prill

was systematically transferred to plastic drums marked 1-1, 1-2, 1-3 (40 gallons each) and 1-4 (5 gallons). The markings on the drums indicate the plant location was number 1 followed by the container numbered as 1, 2, 3 or 4.

PLANT LOCATION NO. 2

Plant location number 2 was considered as the transfer point of partially dried prill from the vertical storage tank by gravity onto a conveyor belt. The conveyor belt appeared to be used to transfer the prill to rail cars etc. Sampling of the partially dried sulfur prill was done from the bottom of the storage tank at regular intervals with the use of a 5 gallon plastic container. Each sample was then transferred to plastic drums marked as 2.1 (approx. 40 gallons) and 2.2 (5 gallons).

The "2" designation indicates that the sulfur prill had been through the partial drying stage at BURZA. The 2 - "1" and 2 - "2" designations indicate the sequence of the plastic drums filled with the partially dried prill.

The 40 gallon plastic drums contained plastic flexible liners which were closed after filling with BURZA prill.

Each of these drums was equipped with tight clamp-on lids. The two 5 gallon drums were equipped with slip-on squeeze lids. Each 5 gallon drum was placed in triple plastic bags. Each drum was signed, dated and sealed by George Aseff.

During the sampling of BURZA sulfur from the conveyor belt in conjunction with the tower, a fine mist of sulfur and moisture prevailed and appeared to be wind-blown.

During the sampling of the partially dried BURZA sulfur from the storage tank a significant amount of fine sulfur dust was observed being emitted and blown in a wide area. Automobiles and equipment driven to the vicinity were observed to be covered with fine sulfur powder and/or sulfur mist.

Sealed (approximately 40 gallon) plastic drums 1-1, 1-2, 1-3 and 2-1 were transported to Alltrans (carrier) in Fort McMurray on 10/28/82 for shipment to Connecticut. The four drums were placed on an oversized wooden palate and strapped together. The drums were then boxed-in on all sides for shipment as a rigid crate.

The 1-4 and 2-2 five gallon plastic drum samples of BURZA prill were hand carried by George Aseff and were in his

March 31, 1983
Page Five

custody at all times. Drum sample 2-2 was opened by U.S. Customs at Minot, North Dakota on 10/29/83 and George Aseff resealed this drum while maintaining custody. The 1-4 and 2-2 drum samples of BURZA prill were hand delivered to TRC Labs in Hartford, Connecticut on 10/29/83 and placed under lock and key by George Aseff.

On 11/12/82 George Aseff drove to Pilot Freight Carrier, Inc. in Wallingford, Connecticut to obtain custody of prill samples 1-1, 1-2, 1-3 and 2-1 delivered and inspected by U.S. Customs. The seal on drum 2-1 had been broken by U.S. Customs and a small sample removed. Drum 2-1 was resealed by George Aseff. All four drums of sulfur prill were delivered to TRC Labs on 11/12/82 and placed under lock and key with only George Aseff in possession of the key.

SUDIC characterization tests were conducted on BURZA sulfur prill samples 1-4 and 2-2 starting on 10/29/82 while tests were conducted on samples 1-1, 1-2 and 2-1 starting on 12/1/82.

Sulfur prill gross weights, net weights and drum weights were determined as follows:



<u>DRUM SAMPLE NUMBER</u>	<u>GROSS WEIGHT (lbs.)</u>	<u>DRUM TARE WEIGHT (lbs.)</u>	<u>PRILL NET WEIGHT (lbs.)</u>
1.1	505	25	480
1.2	512	25	487
1.3	529	25	504
1.4	59	8.6	50.4
2.1	532	25	507
2.2	62	8.6	53.4

CHARACTERIZATION TESTS

As previously mentioned, all characterization tests on the type 1 (as produced by BURZA) and type 2 (partially dried by BURZA) sulfur prills were conducted in accordance with applicable SUDIC methods. These tests were conducted at the TRC Laboratories in Hartford, Connecticut.

SURFACE WATER AS FUNCTION OF DEPTH

Prior to mixing and quartering of the 1.1, 1.2 and 2.1 drums of sulfur prill, a series of tests was conducted to determine the surface water content of the sulfur as a function of depth A (top zone), B, C and D (bottom zone). Each zone was approximately 8 inches deep. Samples were taken at four different quadrants in each zone and combined resulting in four combined samples from each drum.

Large drum sample 1.3 was left as sealed at the BURZA plant in Canada. This drum was not opened by U.S. Customs at Pilot Trucking in Wallingsford, Connecticut.

The surface water content of prill at various depths are summarized in Table 1.

MIXING DRUMS 1.1 AND 1.2

After sampling for testing the surface water of prill as a function of depth, large drums 1.1 and 1.2 were combined to make about a 1000 pound pile. Excess water was estimated as about 1500 ml. The pH of this excess water was found to be about 3 indicating high acidity. After the drums were opened, a distinct, strong odor of hydrogen sulfide gas persisted in the building.

The mixed sample of 1.1 and 1.2 was quartered and requartered repeatedly to yield about ten 5 gallon and twenty-four 2 gallon plastic buckets of the mixed prill.

Large drum 2.1 was mixed and quartered in a similar manner resulting in a total of five 5 gallon and twelve 2 gallon plastic buckets of mixed prill.

WATER CONTENT

The surface water, trapped water and total water of the BURZA sulfur prill were conducted as per SUDIC S10-77; applicable paragraphs on 1.1/1.2 (mixture), 1.4, 2.1 and 2.2 samples. Water content data are summarized in Table 2.

RATE OF AIR DRYING OF BURZA SULFUR PRILL

The rate of air drying or the loss of surface water was determined in six samples of 1.4 and six samples of 2.2 BURZA sulfur prill. These data are plotted in Figures 1 and 2.

SIEVE ANALYSES AND FRIABILITY OF BURZA SULFUR PRILL

Sieve analyses of as-received 1.1/1.2, 1.4, 2.1 and 2.2 samples of BURZA prill was conducted in accordance with S2-77 and S3-77 method as applicable. In addition, the friability of the 1.1/1.2 and 2.1 sulfur samples was determined as per SUDIC methods S4-77 (10 in. drum) and S5-77 (28 in. drum).

Using the results of these tests, the total fines content, particle breakdown modulus, coefficient of uniformity and particle size distribution were calculated and/or plotted.

March 31, 1983
Page Nine

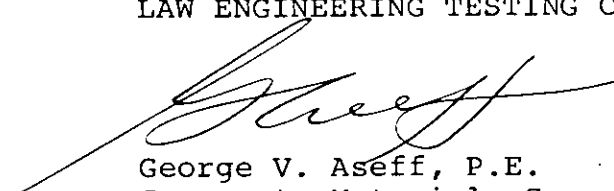
Table 3 is a summary of the data and references some 41 pages of curves and tabulations (1P-29P) and (1F-12F).

SUMMARY


In summary, the approximate 2100 pounds of BURZA (Fletcher Type) sulfur prill, as produced and as partially dried at BURZA has been sampled and characterized according to SUDIC Methods S1-77, S2-77, S3-77, S4-77, S5-77 and S10-77, as required. All of the sampling of the BURZA prill was done by George Aseff. Custody of all samples has remained with Mr. Aseff through the entire characterization program. All of the sampling and testing associated with the BURZA prill was conducted either by George Aseff or under his direct supervision.

BY:

LAW ENGINEERING TESTING COMPANY


George V. Aseff, P.E.
Corporate Materials Consultant

REVIEWED BY:


Donald E. Dixon, P.E.
Corporate Materials Consultant

GVA:DED/ljh



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JOB NO. G-8657 SHEET 1 OF 1

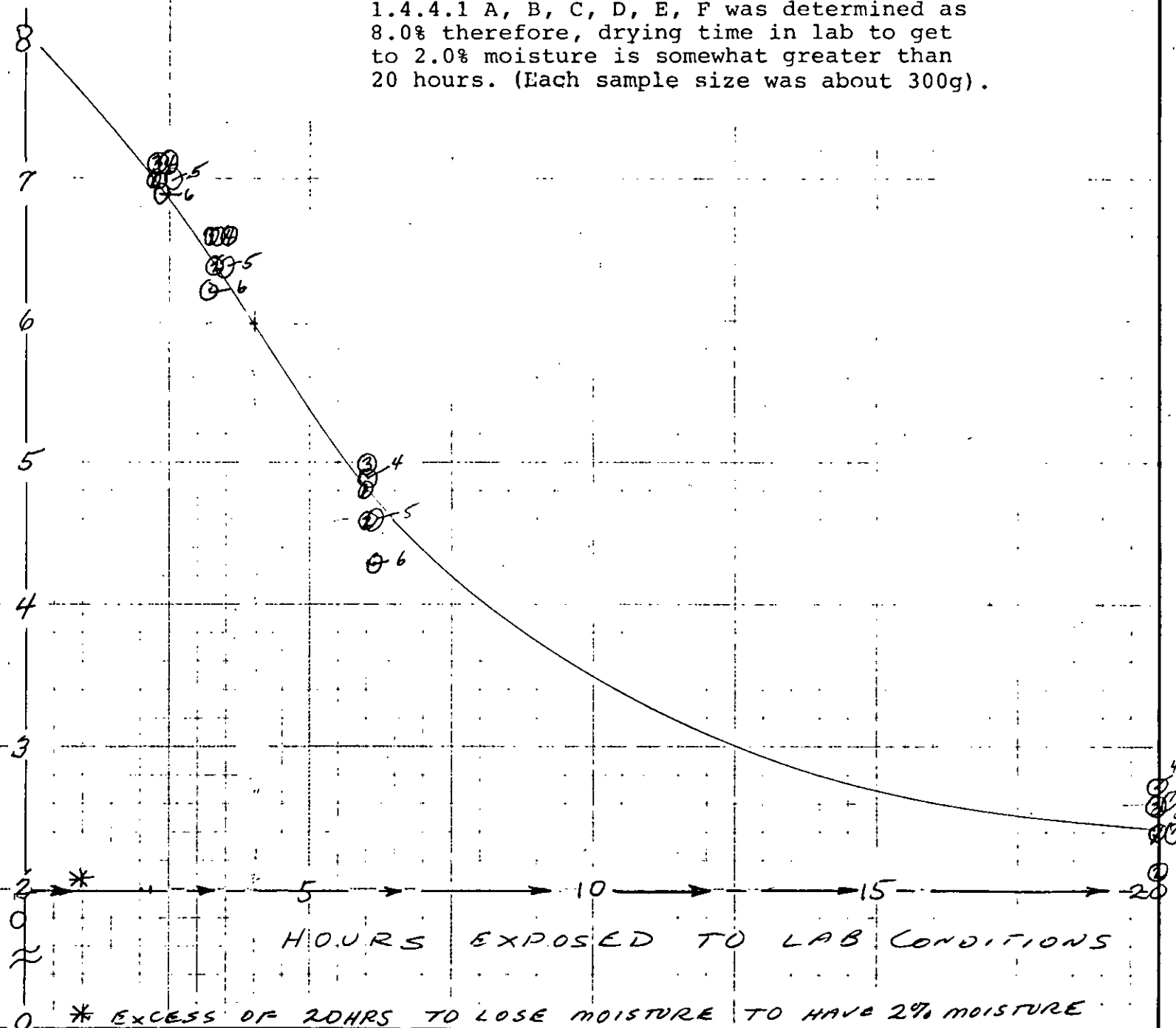
JOB NAME BURZA Sulfur Prill

BY G.V. Aseff DATE 1/24/83

CHECKED BY *[Signature]* DATE 1/25/83

FIGURE 1 RATE OF AIR DRYING OF BURZA PRILL 1.4 SAMPLES

PERCENT MOISTURE CONTENT AT LAB CONDITIONS



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JOB NAME BURZA Sulfur Prill

BY G.V. Aseff DATE 1/24/83

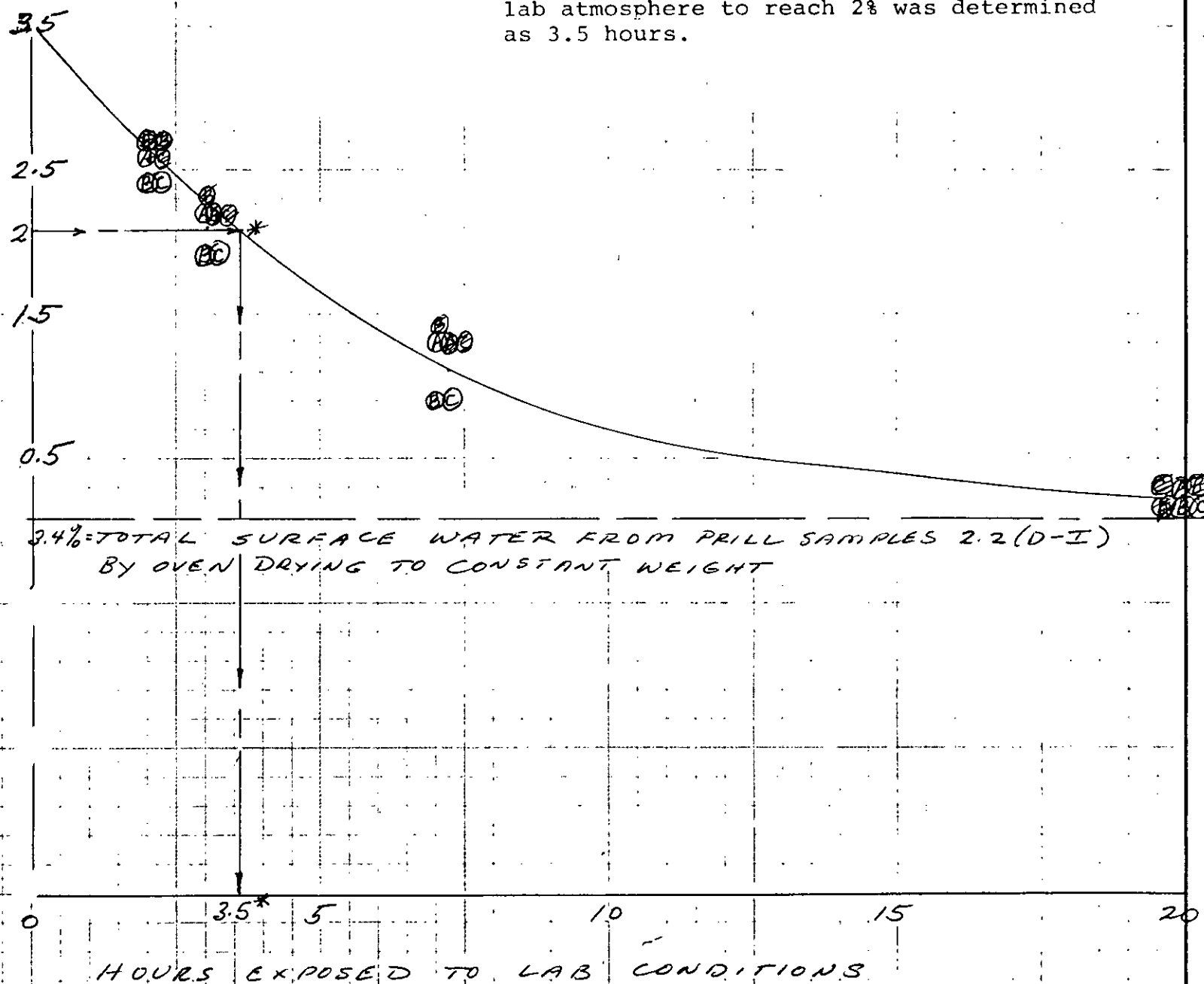
CHECKED BY gta DATE 1/25/83

FIGURE 2. RATE OF AIR DRYING OF BURZA PRILL 2.2 SAMPLES

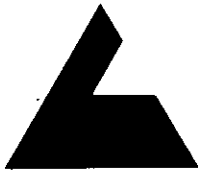
PERCENT MOISTURE CONTENT AT LAB. CONDITIONS

REF P54

NOTE: Average surface moisture of 2.2 samples was determined as 3.5%. Drying time in lab atmosphere to reach 2% was determined as 3.5 hours.



* 3.5 HRS TO LOSE MOISTURE TO HAVE 2% MOISTURE REMAINING

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(404) 873-4761JOB NO. G-8657 SHEET 1 OF 1JOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/20/83

TABLE 1 SUMMARY OF DATA ON THE SURFACE WATER
CONTENT OF AS-RECEIVED BURZA SULFUR
PRILL AS A FUNCTION OF DEPTH IN LARGE
DRUM SAMPLES 1.1, 1.2 & 2.1 PER SUDIC
METHOD 510-77 (4.1)

PRILL SAMPLE	DEPTH IN DRUM (ZONE)	SURFACE WATER (%)	REF
1.1	A TOP	4.8	36,37
1.1	B	4.9	
1.1	C	5.3	
1.1	D BOTTOM	10.3	
1.2	A TOP	4.0	36,37
1.2	B	6.7	
1.2	C	5.4	
1.2	D BOTTOM	10.3	
2.1	A TOP	2.7	38
2.1	B	3.4	
2.1	C	3.2	
2.1	D BOTTOM	3.4	

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TABLE 2 CHARACTERIZATION TESTS USING SUDIC METHODS TO DETERMINE SURFACE, TRAPPED AND TOTAL MOISTURE CONTENT OF BURZA (FLETCHER TYPE) SULFUR PRILL

SUDIC METHOD	SAMPLE NUMBER	TEST PROCEDURE	RESULTS	REF.
S10-77(4.1)	1-4-4.1 A,B,C	WEIGHT DIFFERENCE SURFACE MOISTURE	6.8% (AV.)	6
S10-77(4.1)	2-2-4.1 A,B,C	WEIGHT DIFFERENCE SURFACE MOISTURE	3.9% (AV.)	6
S10-77(4.2)	1-4-4.1 A,B,C	KARL FISCHER TITRATION TRAPPED WATER	0.23% (AV.)	9
S10-77(4.2)	2-2-4.2 A,B,C	KARL FISCHER TITRATION TRAPPED WATER	0.40% (AV.)	10
S10-77(4.3)	1-4-4.3 A,B,C	KARL FISCHER TITRATION TOTAL WATER CONTENT	6.6% (AV.)	9
S10-77(4.3)	2-2-4.3 A,B,C	KARL FISCHER TITRATION TOTAL WATER CONTENT	3.4% (AV.)	10
S10-77(4.1)	1-4-4.1 A,B,C	WEIGHT DIFFERENCE SURFACE MOISTURE	8.0% (AV.)	17
	1-4 D,E,F		8.0% (AV.)	18
	2-2 D,E,F (~100g)		3.5% (AV.)	18
S10-77(4.1)	2-2 (D→I) (~600g)	WEIGHT DIFFERENCE SURFACE MOISTURE	3.4% (AV.)	19
S10-77(4.2)	1-4 D,E,F (~50g)	KARL FISCHER TITRATION TRAPPED WATER	0.35% (AV.)	20
	2-2 D,E,F (~50g)		0.33% (AV.)	20
S10-77(4.1)	1-1/1-2 S2A,B,C	WEIGHT DIFFERENCE SURFACE MOISTURE	7.6% (AV.)	41
S10-77(4.1)	2-1 S2/S3 A,B,C	WEIGHT DIFFERENCE SURFACE MOISTURE	3.7% (AV.)	42
S10-77(4.2)	1-1/1-2 A,B,C	KARL FISCHER TITRATION TRAPPED WATER	0.24% (AV.)	45
			0.28% (AV.)	45

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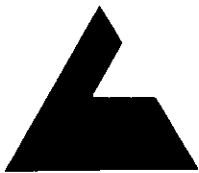
TABLE 3 SUMMARY OF DATA ON TOTAL FINES CONTENT, PARTICLE BREAKDOWN MODULUS AND COEFFICIENT OF UNIFORMITY PER SUDIC METHODS ON BURZA FLETCHER TYPE SULFUR PRILL

PRILL SAMPLE NUMBER	SUDIC METHOD	TOTAL FINES CONTENT (%)	"PBM"	COEFF. OF UNIFORMITY	REFERENCE PAGES	
					PARTICLE SIZE	FRIABILITY
1.4 F	S2-77, DRY SIEVE	0.8	—	2.4	1P	—
2.2 F	S2-77, DRY SIEVE	1.7	—	3.1	2P	—
1.4	S3-77, WET SIEVE	1.4	—	2.1	3P	—
2.2 E	S3-77, WET SIEVE	2.4	—	2.4	4P	—
1.4.4.1A	S3-77, WET SIEVE	1.1	—	2.2	5P	—
1.1/1.2(A)(B)(C)	S2-77, DRY SIEVE	1.0-1.3 AV. 1.2	— —	2.4-2.5 AV. 2.5	6P, 7P, 8P	— —
2.1(A)(B)(C)	S2-77, DRY SIEVE	2.0-2.7 AV. 2.3	— —	2.7-3.5 AV. 3.2	9P, 10P, 11P	— —
1.1/1.2 S2A, B, C	S3-77, WET SIEVE	1.1-1.7 AV. 1.3	— —	2.0-2.5 AV. 2.3	12P, 13P, 14P	— —
2.1 S3(A)(B)(C)	S3-77, WET SIEVE	2.5-2.7 AV. 2.6	— —	3.3-3.3 AV. 3.3	15P, 16P, 17P	— —

FRIABILITY TESTS; SUDIC 54-77 & 55-77 FOLLOWED BY 53-77 WET SIEVE

1.1/1.2 S4A, B, C	S3-77, WET SIEVE AFTER 54-77	6.7-7.8 AV. 7.2	19.81-21.55 AV. 20.6	3.1-5.7 AV. 4.6	18P, 19P, 20P	1F, 2F, 3F
2.1 S4A, B, C	S3-77, WET SIEVE AFTER 54-77	4.7-5.4 AV. 5.0	12.5-14.3 AV. 13.6	3.0-3.4 AV. 3.3	21P, 22P, 23P	4F, 5F, 6F
1.1/1.2 S5A, B, C	S3-77 WET SIEVE AFTER 55-77	5.5-5.8 AV. 5.7	25.0-25.8 AV. 25.4	3.3-3.6 AV. 3.4	24P, 25P, 26P	7F, 8F, 9F
2.1 S5A, B, C	S3-77 WET SIEVE AFTER 55-77	4.9-5.3 AV. 5.1	22.7-23.1 AV. 22.8	3.4-3.4 AV. 3.4	27P, 28P, 29P	10F, 11F, 12F

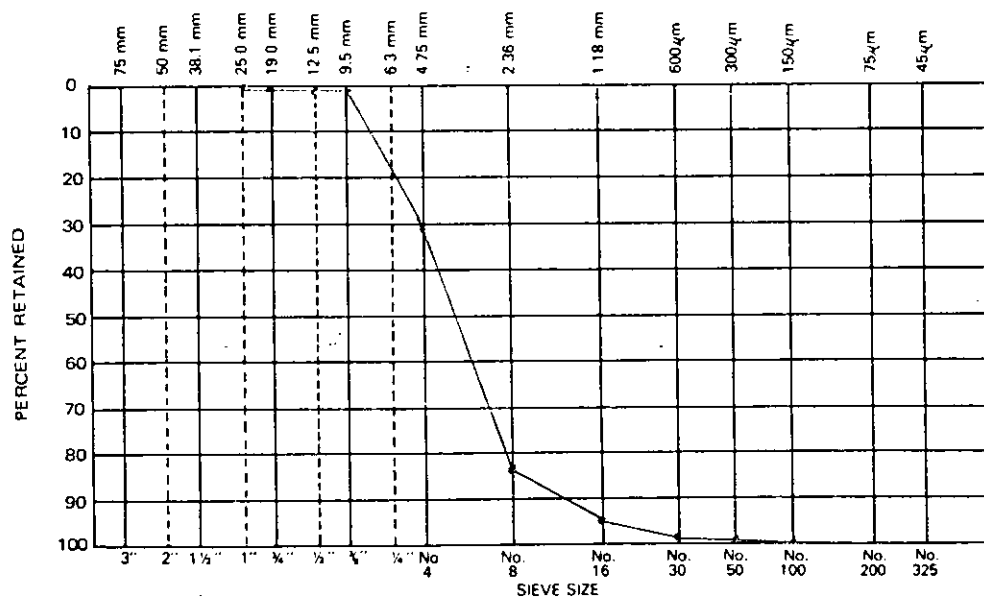
"PBM" PARTICLE BREAKDOWN MODULUS

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 1P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/831.4 F, AS PRODUCED PRILL, SUDIC 52-77
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILLProducer: BURZA, ALBERTA, CAN.Gross Sample Size: 384.9 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 11/10/82Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 0.8%Coefficient of Uniformity: 2.4Comments: Need to wash downfines clinging to larger particles

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	30.7
No. 8 (2.36 mm)	53.3
No. 16 (1.18 mm)	11.2
No. 30 (600 µm)	3.3
No. 50 (300 µm)	0.8
Minus No. 50	0.8

*To nearest
0.1%

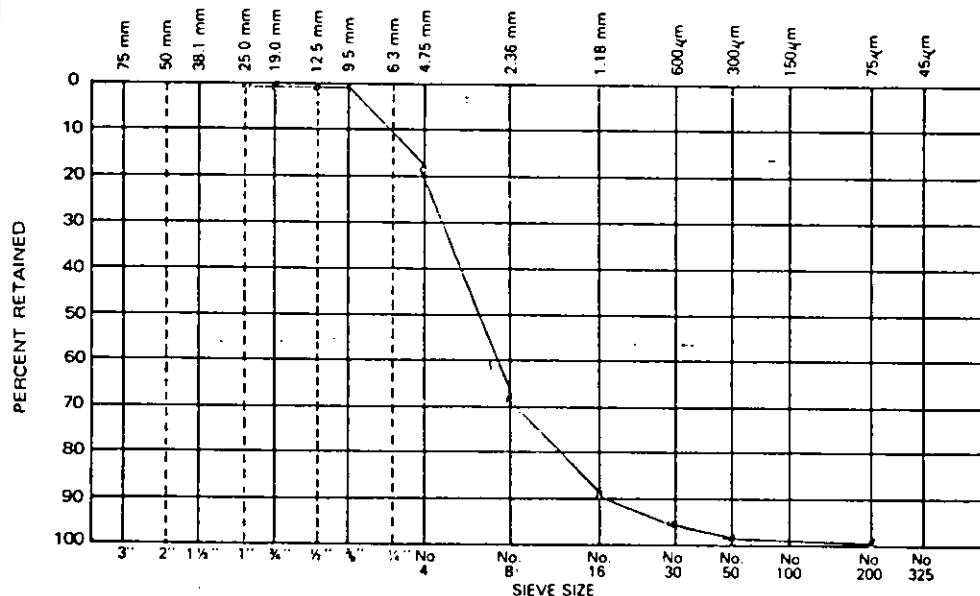
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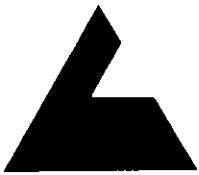
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 2P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY sa DATE 1/25/83**2.2 F, 4.5 PRODUCED PRILL, SUDIC 52-77
PARTICLE SIZE DISTRIBUTION**Sulphur Form: PRILLProducer: BURZAGross Sample Size: 350.8 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 11/10/82Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 1.7Coefficient of Uniformity: 3.1

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	18
No. 8 (2.36 mm)	50.1
No. 16 (1.18 mm)	20.9
No. 30 (600 μ m)	6.9
No. 50 (300 μ m)	2.4
Minus No. 50	1.7

*To nearest
0.1%

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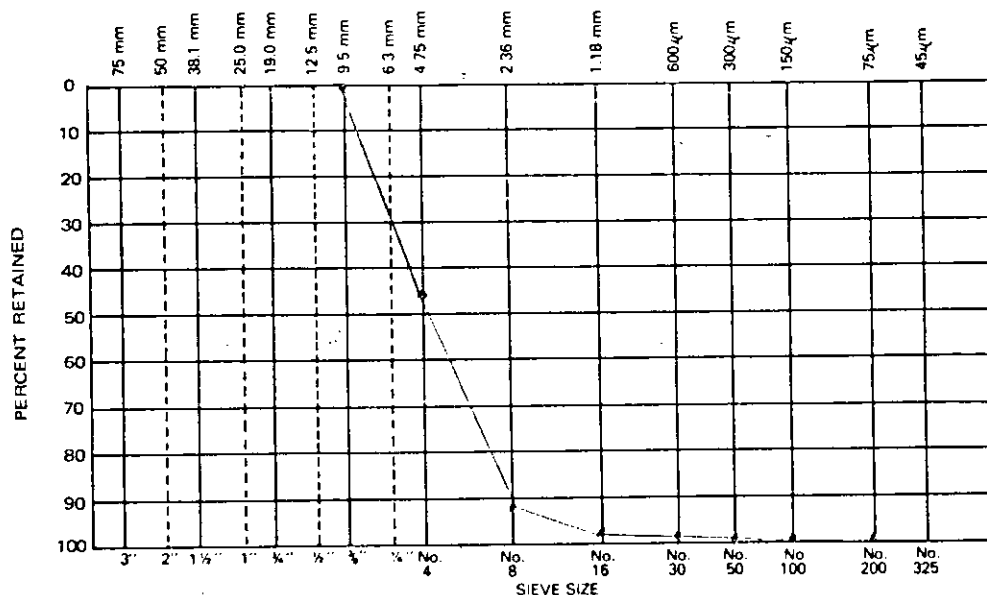
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 3P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83

1.4, AS PRODUCED PRILL, PER SUDIC 53-77
PARTICLE SIZE DISTRIBUTION
WASHED FINES

Sulphur Form: PRILLProducer: BURZA (FT. MCMURRY)ALBENTHA, CAN.)Gross Sample Size: 327.04 gramsDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 11/10/82Sieve Analysis: Wet WASHEDDry Fines Content (% Minus No. 50): 1.4%Coefficient of Uniformity: 2.1Comments: to 450 = 0.3%WASHED DOWN = 1.1%TOTAL = 1.4%

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	.73
No. 4 (4.75 mm)	45.5
No. 8 (2.36 mm)	46.4
No. 16 (1.18 mm)	5.7
No. 30 (600 μ m)	1.0
No. 50 (300 μ m)	0.4
Minus No. 50	1.4

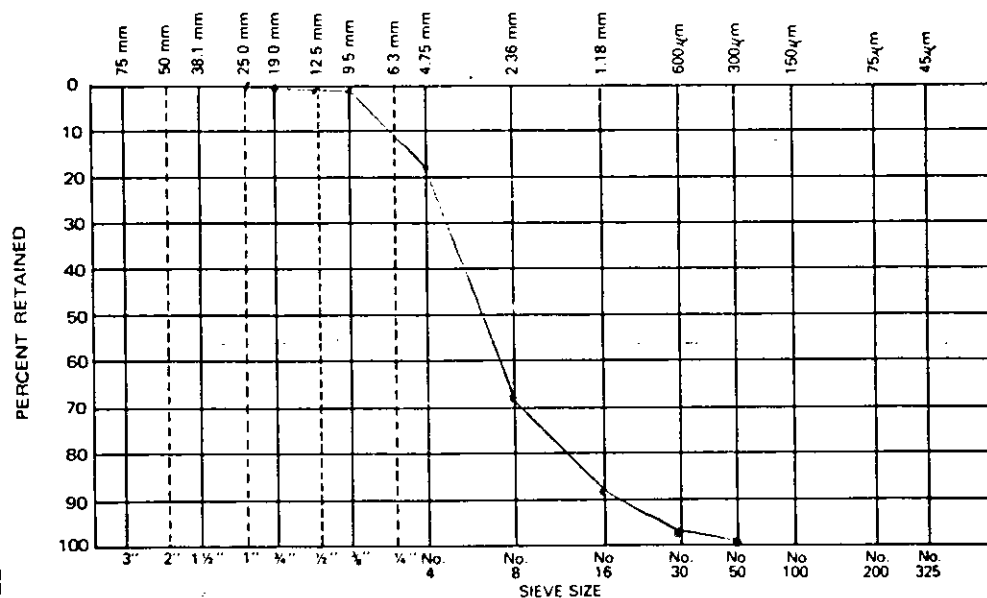
*To nearest
0.1%

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 4P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY ba DATE 1/25/832.2, AS PRODUCED DRILL PER SODIC
(2.2 E) PARTICLE SIZE DISTRIBUTION 53-77
WASHED FINESSulphur Form: PRILLProducer: BURZA (FT. McMURRY)

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	18.4
No. 8 (2.36 mm)	49.4
No. 16 (1.18 mm)	20.8
No. 30 (600 µm)	7.7
No. 50 (300 µm)	3.0
Minus No. 50	2.4

Gross Sample Size: 340.3 g.Date Formed: 10/28/82Date Received: 10/28/82Date Tested: 11/10/82Sieve Analysis: Wet ✓ WASHEDDry Fines Content (% Minus No. 50): 2.4Coefficient of Uniformity: 4.1Comments: washed (-50) = 1.7%sieved = 0.7%2.4%*To nearest
0.1%

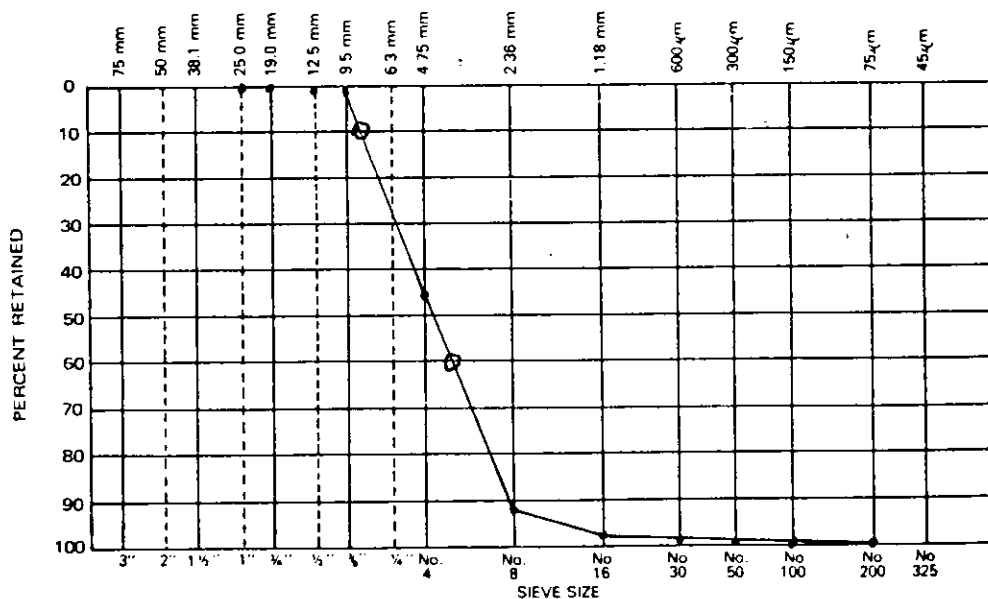
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 5P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/83**1.4.4.1A AS PRODUCED PRILL, SUDIC 53-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILLProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 325.9 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 11/10/82Sieve Analysis: Wet ☒Dry ☐Fines Content (% Minus No. 50): 1.1%Coefficient of Uniformity: 2.2

Comments:

1"	(25.0 mm)	-
3/4"	(19.0 mm)	-
1/2"	(12.5 mm)	-
3/8"	(9.5 mm)	0.7
No. 4	(4.75 mm)	45.5
No. 8	(2.36 mm)	46.4
No. 16	(1.18 mm)	5.7
No. 30	(600 μ m)	1.0
No. 50	(300 μ m)	0.4
Minus No. 50		1.1

* To nearest
0.1%

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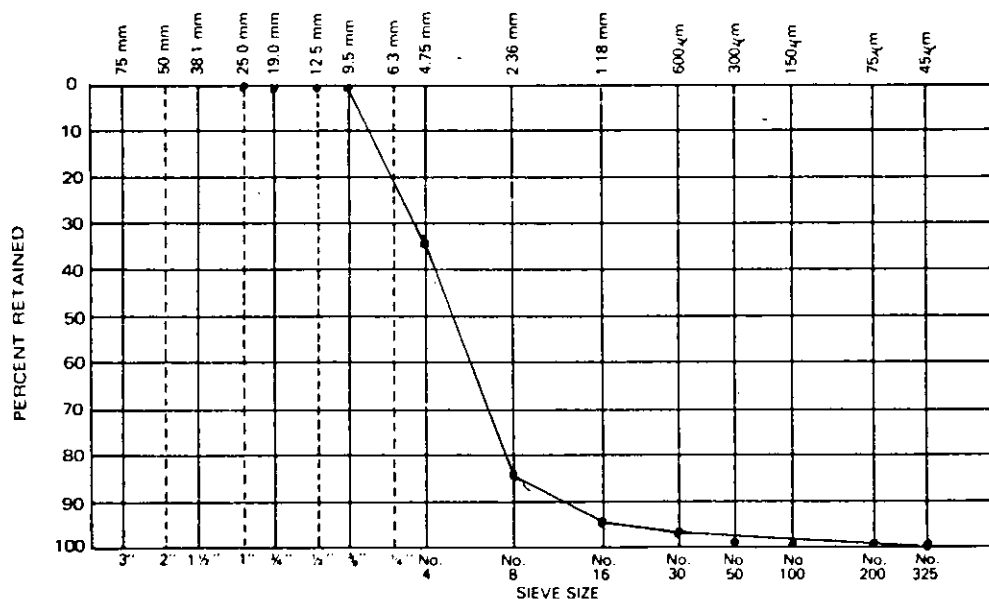
396 PLASTERS AVENUE N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 6P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY ba DATE 1/25/83**1.1/1.2 (A) AS PRODUCED PRILL, SUDIC 52-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 288.9 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/3/82

Sieve Analysis: Wet _____

Dry ✓Fines Content (% Minus No. 50): 1.0%Coefficient of Uniformity: 2.5

Comments:

1"	(25.0 mm)	-
3/4"	(19.0 mm)	-
1/2"	(12.5 mm)	-
3/8"	(9.5 mm)	0.1
No. 4	(4.75 mm)	34.2
No. 8	(2.36 mm)	49.4
No. 16	(1.18 mm)	10.9
No. 30	(600 μ m)	3.5
No. 50	(300 μ m)	1.0
Minus No. 50		1.0

* To nearest
0.1%

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 7P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/26/831.1/1.2(B) AS-RECEIVED PRILL, SUDIC S2-77
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 291.3 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/3/83Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 1.3 %Coefficient of Uniformity: 2.5

Comments:

1" (25.0 mm)

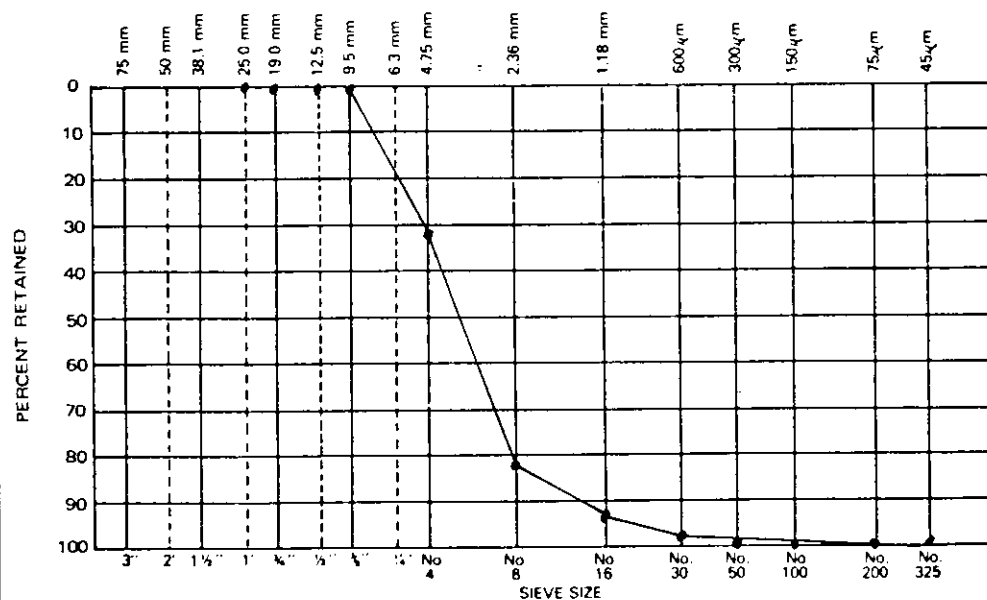
3/4" (19.0 mm)

1/2" (12.5 mm)

3/8" (9.5 mm)

No. 4 (4.75 mm) 31.8No. 8 (2.36 mm) 49.6No. 16 (1.18 mm) 12.4No. 30 (600 μ m) 4.0No. 50 (300 μ m) 1.0

Minus No. 50

1.3*To nearest
0.1%

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(404) 873-4761JOB NO. G-8657 SHEET 8P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/83**1.1/1.2 (C) AS-RECEIVED PRILL, 9001C 52-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CANGross Sample Size: 309 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/3/82Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 1.2%Coefficient of Uniformity: 2.4

Comments:

Sieve Size

Percent
Retained*

1" (25.0 mm)

3/4" (19.0 mm)

1/2" (12.5 mm)

3/8" (9.5 mm)

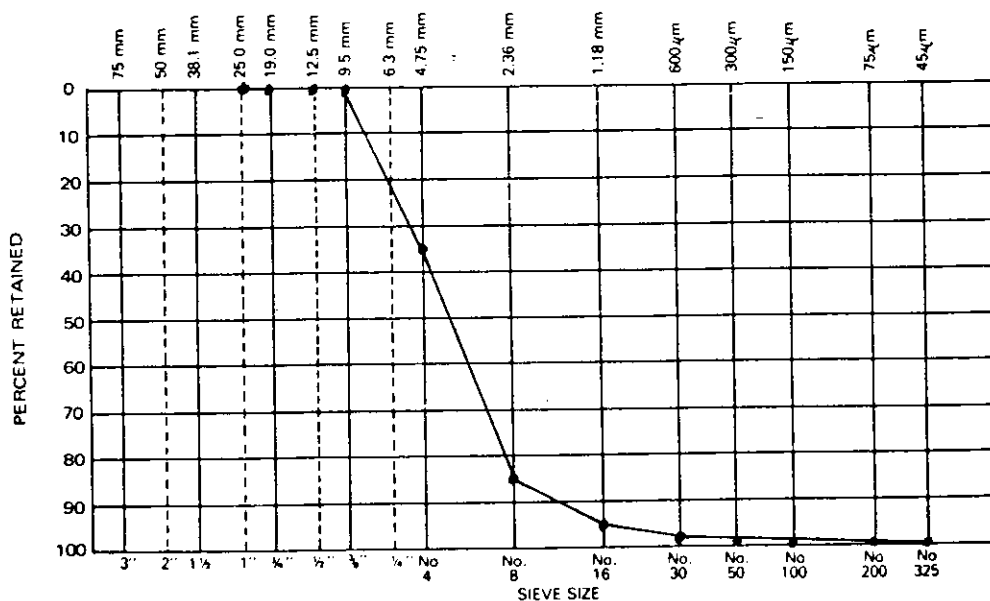
No. 4 (4.75 mm)

No. 8 (2.36 mm)

No. 16 (1.18 mm)

No. 30 (600 μ m)No. 50 (300 μ m)

Minus No. 50

35.649.69.43.40.91.2*To nearest
0.1%

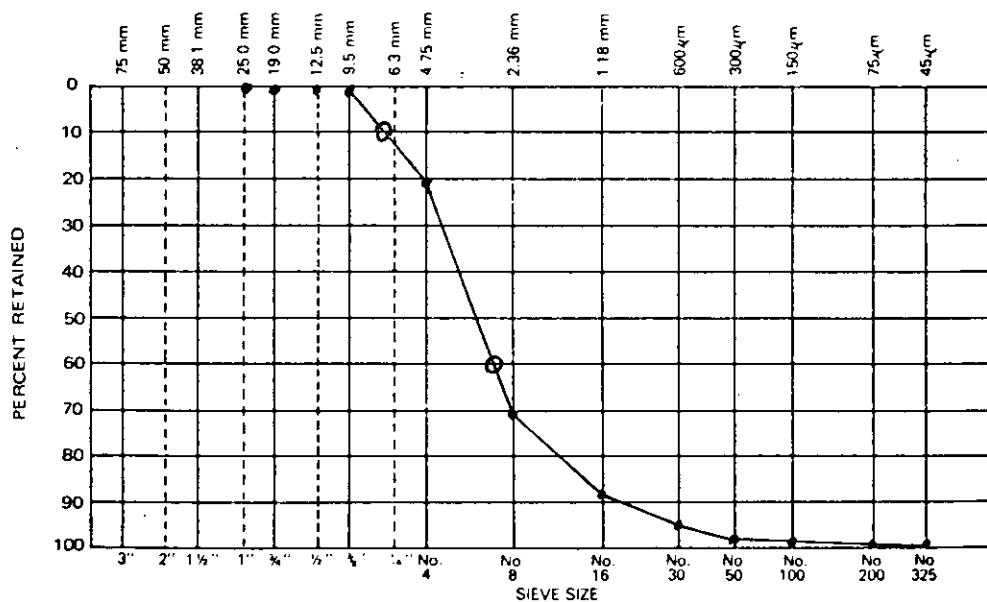
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 9P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY SA DATE 1/25/83**2.1(A) AS-RECEIVED PRILL, SUDIC 52-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN.Gross Sample Size: 322.7 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/3/82Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 2.7%Coefficient of Uniformity: 3.5

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	0.2
No. 4 (4.75 mm)	20.7
No. 8 (2.36 mm)	49.6
No. 16 (1.18 mm)	17.4
No. 30 (600 μ m)	6.7
No. 50 (300 μ m)	2.9
Minus No. 50	2.7

*To nearest
0.1%

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 10P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**2.1(B) AS-RECEIVED PRILL, SUDIC 52-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 283.5 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/13/82Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 2.3%Coefficient of Uniformity: 3.5

Comments:

1" (25.0 mm)

3/4" (19.0 mm)

1/2" (12.5 mm)

3/8" (9.5 mm)

No. 4 (4.75 mm)

No. 8 (2.36 mm)

No. 16 (1.18 mm)

No. 30 (600 μ m)No. 50 (300 μ m)

Minus No. 50

0.3

10.9

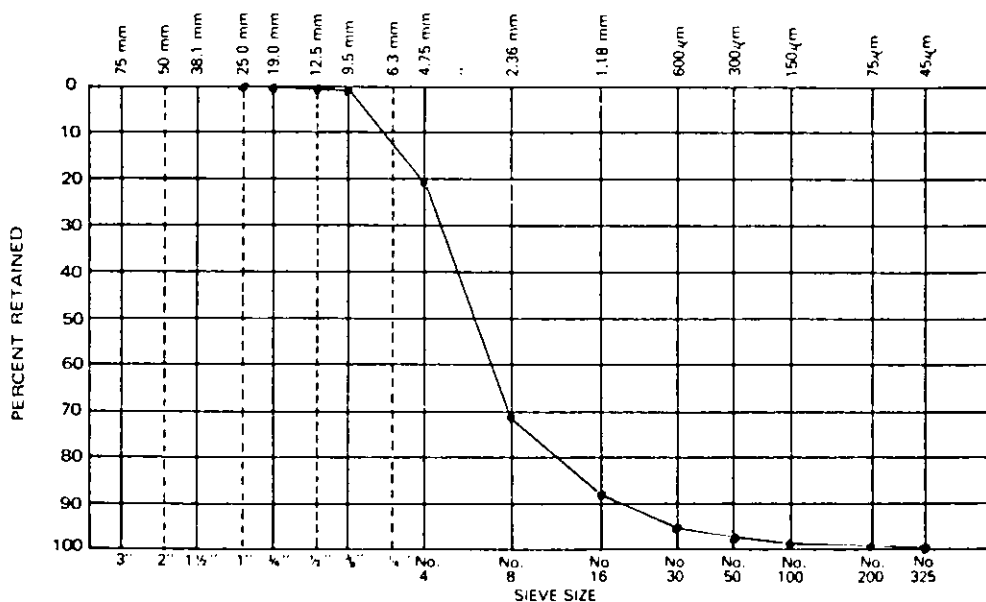
49.2

17.6

6.9

2.9

2.3

* To nearest
0.1%

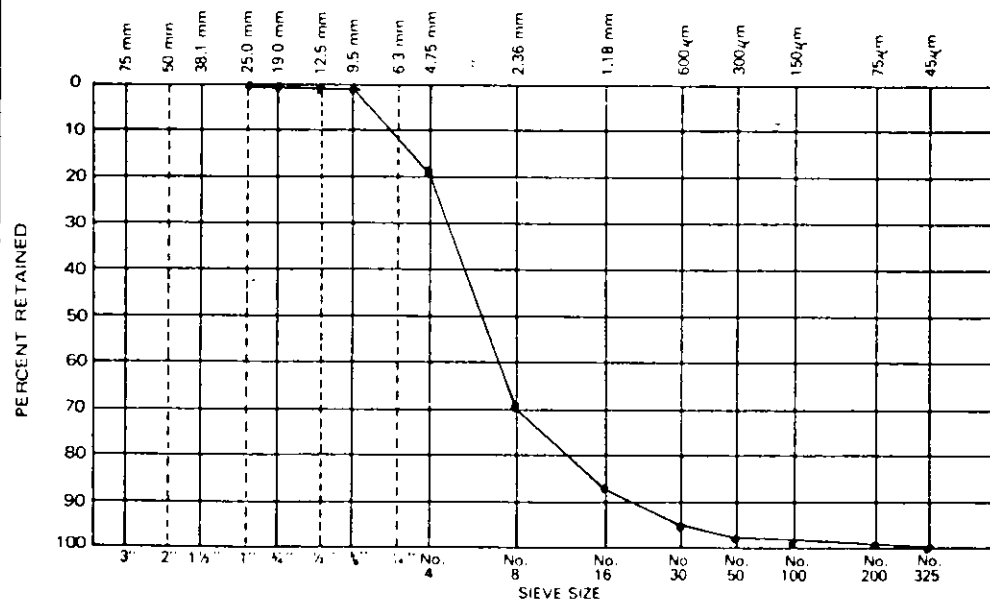
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 11P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**2.1 (C) AS-RECEIVED PRILL, SUDIC 52-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 276.1 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/3/82Sieve Analysis: Wet Dry ✓Fines Content (% Minus No. 50): 2.0%Coefficient of Uniformity: 2.7

Comments:

1"	(25.0 mm)	—
3/4"	(19.0 mm)	—
1/2"	(12.5 mm)	—
3/8"	(9.5 mm)	0.1
No. 4	(4.75 mm)	19.1
No. 8	(2.36 mm)	50.1
No. 16	(1.18 mm)	18.6
No. 30	(600 μ m)	7.0
No. 50	(300 μ m)	2.9
Minus No. 50		2.0

*To nearest
0.1%

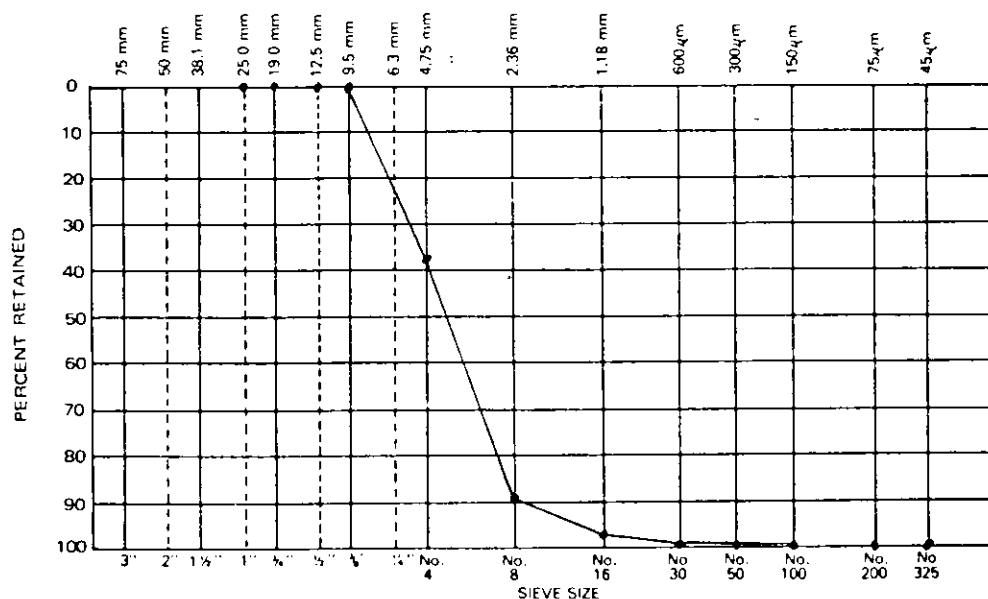
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 12P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**1.1/1.2 S2A WASHED OFF FINES, SUDIC 53-77**
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN.Gross Sample Size: 251.7 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/6/82Sieve Analysis: Wet VDry Fines Content (% Minus No. 50): 1.1%Coefficient of Uniformity: 2.0Comments: WASHED FINES 0.9SIEVED FINES 0.2TOTAL 1.1%

Sieve Size	Percent Retained*
1" (25.0 mm)	—
3/4" (19.0 mm)	—
1/2" (12.5 mm)	—
3/8" (9.5 mm)	—
No. 4 (4.75 mm)	38.0
No. 8 (2.36 mm)	50.1
No. 16 (1.18 mm)	8.0
No. 30 (600 µm)	2.3
No. 50 (300 µm)	0.8
Minus No. 50	1.1

* To nearest 0.1%

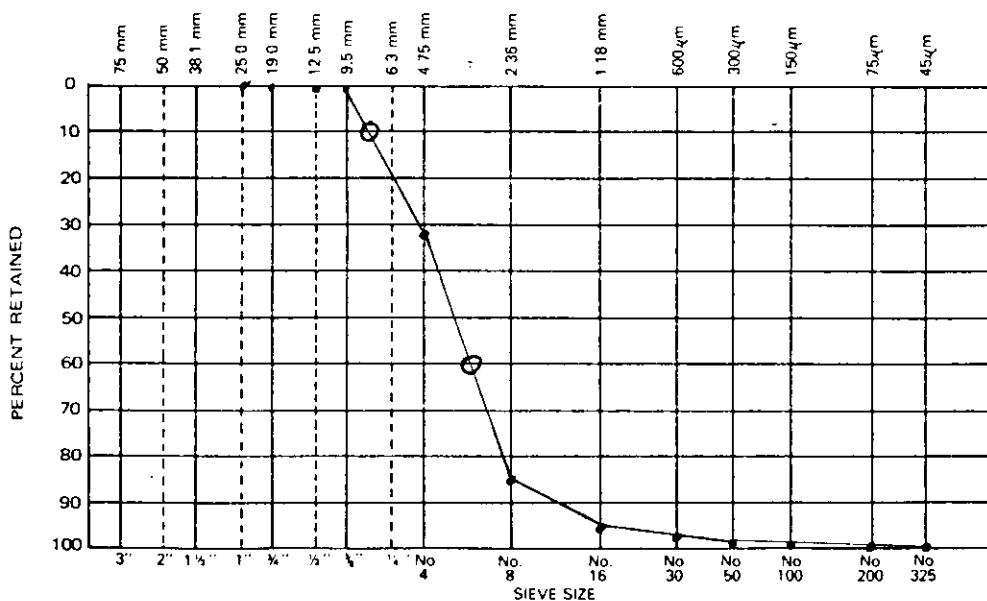


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(404) 873-4761JOB NO. G-8657 SHEET 13P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 S2B, WASHED OFF FINES, SUDIC 53-77
PARTICLE SIZE DISTRIBUTIONSulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN Sieve SizePercent
Retained*Gross Sample Size: 284.9 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/6/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 1.2 %Coefficient of Uniformity: 2.5Comments: WASHED FINES 0.7SIEVED FINES 0.5TOTAL 1.2 %

1"	(25.0 mm)	—
3/4"	(19.0 mm)	—
1/2"	(12.5 mm)	—
3/8"	(9.5 mm)	—
No. 4	(4.75 mm)	32.1
No. 8	(2.36 mm)	54.0
No. 16	(1.18 mm)	9.0
No. 30	(600 μ m)	2.7
No. 50	(300 μ m)	1.0
Minus No. 50		1.2

*To nearest
0.1%

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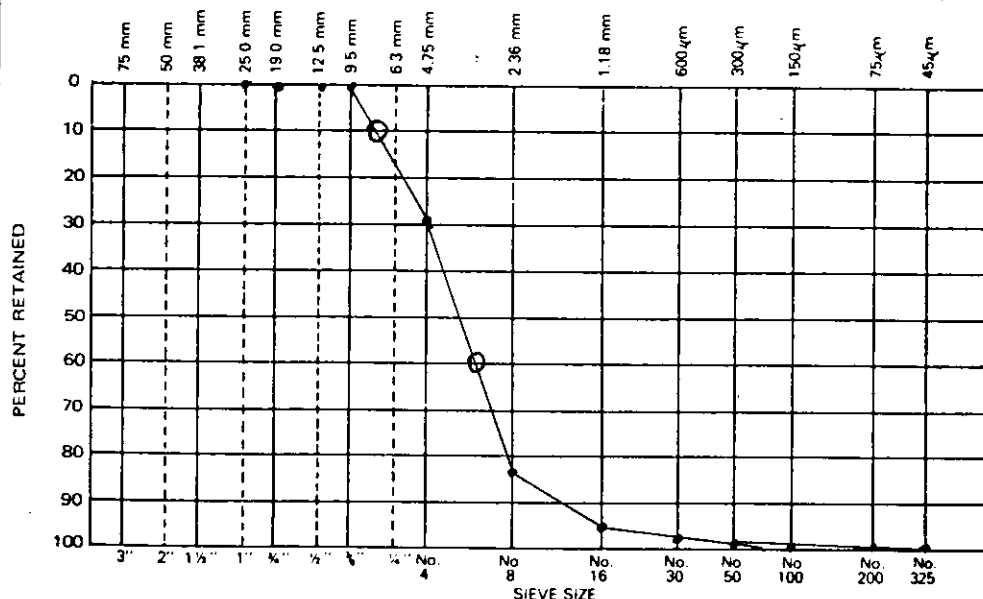
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 14P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 4/20/831.1/1.2 S2C WASHED OFF FINES, SUDIC S3-77
PARTICLE SIZE DISTRIBUTION REF. P58Sulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 30 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/6/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 1.7%Coefficient of Uniformity: 2.5

Comments:

WASHED FINES 1.3SIEVED FINES 0.4TOTAL 1.7%

1"	(25.0 mm)	—
3/4"	(19.0 mm)	—
1/2"	(12.5 mm)	—
3/8"	(9.5 mm)	—
No. 4	(4.75 mm)	28.5
No. 8	(2.36 mm)	53.3
No. 16	(1.18 mm)	11.8
No. 30	(600 μ m)	3.6
No. 50	(300 μ m)	1.1
Minus No. 50		1.7

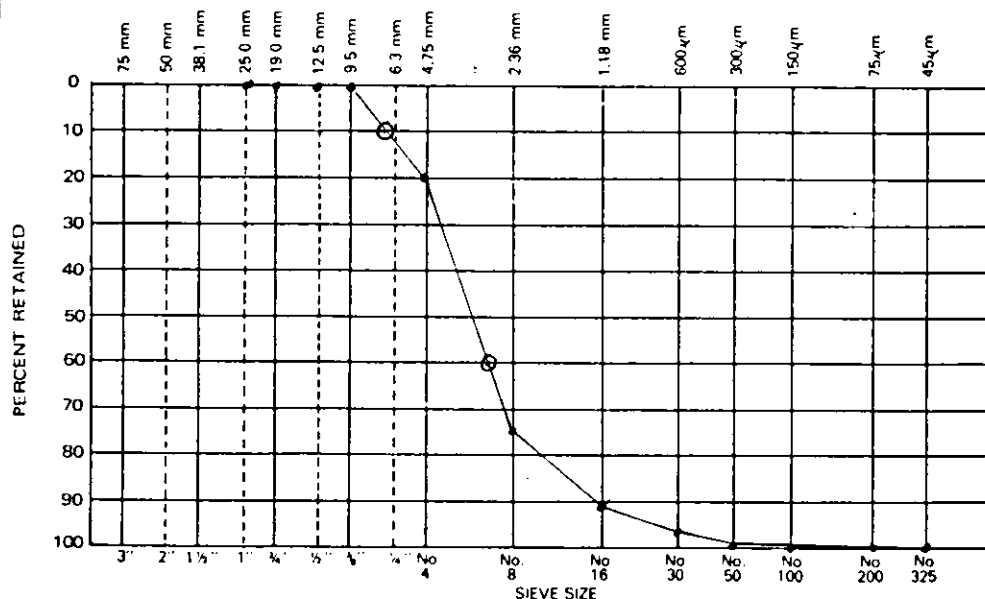
*To nearest
0.1%

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO G-8657 SHEET 15P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**2.1 S3A WASHED OFF FINES, SUDIC 53-77**
PARTICLE SIZE DISTRIBUTION REF. P59Sulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 2.41.2 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/17/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 2.5%Coefficient of Uniformity: 3.3Comments: WASHED FINES 1.8SIEVED FINES 0.7TOTAL 2.5%

1"	(25.0 mm)	
3/4"	(19.0 mm)	
1/2"	(12.5 mm)	
3/8"	(9.5 mm)	
No. 4	(4.75 mm)	19.6
No. 8	(2.36 mm)	53.3
No. 16	(1.18 mm)	16.5
No. 30	(600 μ m)	5.7
No. 50	(300 μ m)	2.4
Minus No. 50		2.5

*To nearest
0.1%

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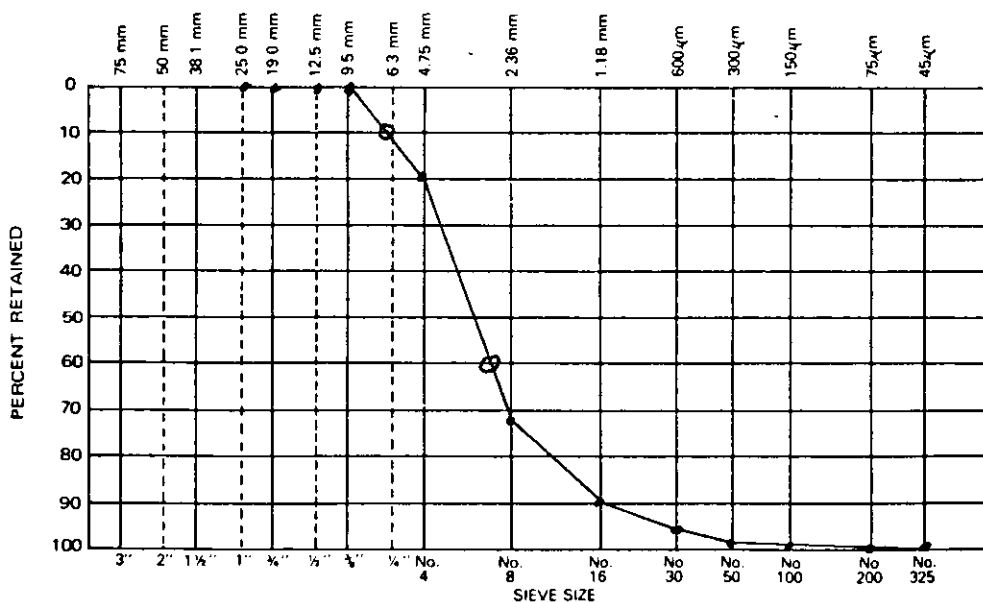
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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 16P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY SA DATE 1/25/83**2.1 S3B WASHED OFF FINES, SUDIC S3-77**
PARTICLE SIZE DISTRIBUTION REF. P. 60Sulphur Form: PRILL, FLETCHERProducer: BURZA, ALBERTA, CANGross Sample Size: 226.0 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry —Fines Content (% Minus No. 50): 2.7%Coefficient of Uniformity: 3.3

Comments:

WASHED FINES 1.9SIEVED FINES 0.8TOTAL 2.7%

Sieve Size	Percent Retained*
1" (25.0 mm)	—
3/4" (19.0 mm)	—
1/2" (12.5 mm)	—
3/8" (9.5 mm)	—
No. 4 (4.75 mm)	18.8
No. 8 (2.36 mm)	51.4
No. 16 (1.18 mm)	17.9
No. 30 (600 μ m)	6.5
No. 50 (300 μ m)	2.7
Minus No. 50	2.7

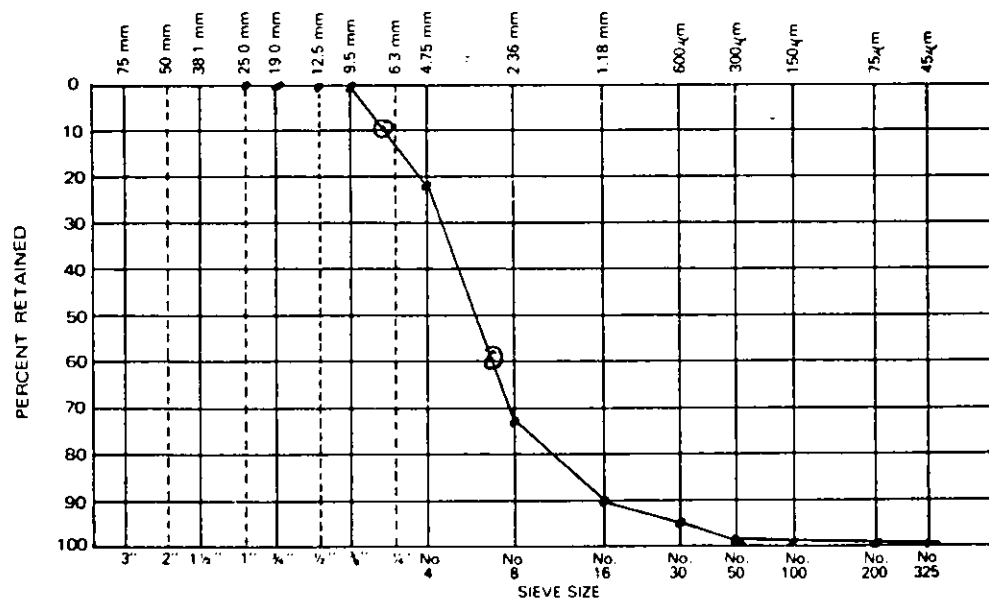
*To nearest
0.1%

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 17P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY SA DATE 1/25/83**2.1 S3C WASHED OFF FINES, SUDIC 53-77**
PARTICLE SIZE DISTRIBUTION REF P61Sulphur Form: DRILL, FLETCHERProducer: BURZA, ALBERTA, CAN. Sieve SizePercent
Retained*Gross Sample Size: 230.4 GRAMSDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 2.7%Coefficient of Uniformity: 3.3Comments: WASHED FINES 2.1SIEVED FINES 0.6TOTAL 2.7%

1"	(25.0 mm)	—
3/4"	(19.0 mm)	—
1/2"	(12.5 mm)	—
3/8"	(9.5 mm)	—
No. 4	(4.75 mm)	21.5
No. 8	(2.36 mm)	49.4
No. 16	(1.18 mm)	16.8
No. 30	(600 µm)	6.6
No. 50	(300 µm)	2.9
Minus No. 50		2.7

*To nearest
0.1%

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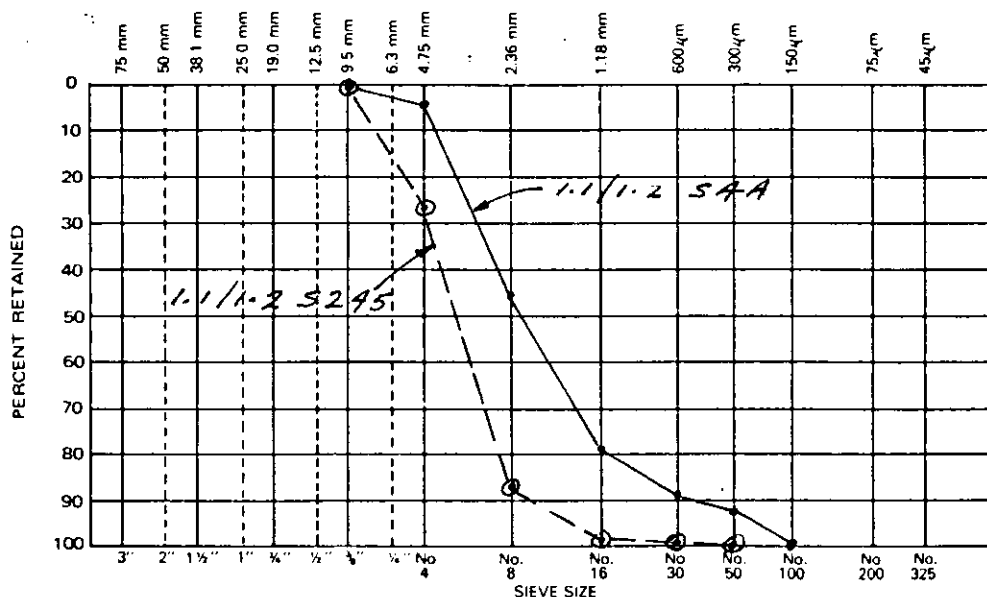
geotechnical, environmental & construction materials consultants

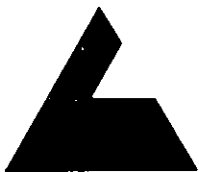
396 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 18P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83

1.1/1.2 S4A (TUMBLED, 10" DRUM)
PARTICLE SIZE DISTRIBUTION
1.1/1.2 S245 (REFERENCE, NOT TUMBLED)

Sulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 319.39Date Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 7.2%Coefficient of Uniformity: 3.1 (S4A)Comments: 2.0 (S245)

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	4.0
No. 8 (2.36 mm)	41.9
No. 16 (1.18 mm)	33.1
No. 30 (600 μ m)	10.1
No. 50 (300 μ m)	3.8
Minus No. 50	7.2

*To nearest
0.1%

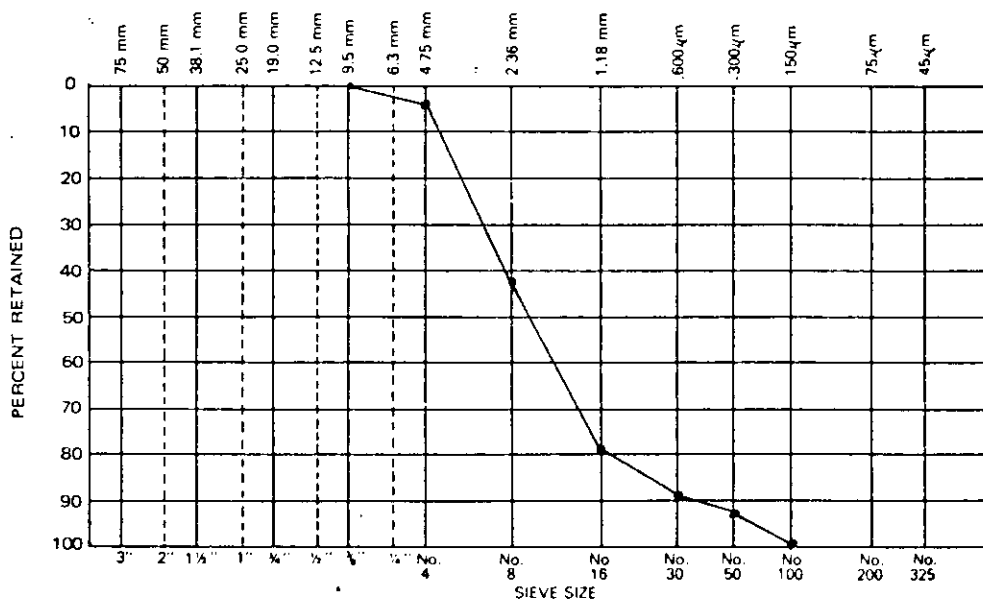
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395 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 19P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 548 PARTICLE SIZE DISTRIBUTIONSulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 281.8gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 7.8%Coefficient of Uniformity: 5.7

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	3.2
No. 8 (2.36 mm)	38.7
No. 16 (1.18 mm)	36.8
No. 30 (600 μ m)	9.8
No. 50 (300 μ m)	3.7
Minus No. 50	7.8

*To nearest
0.1%

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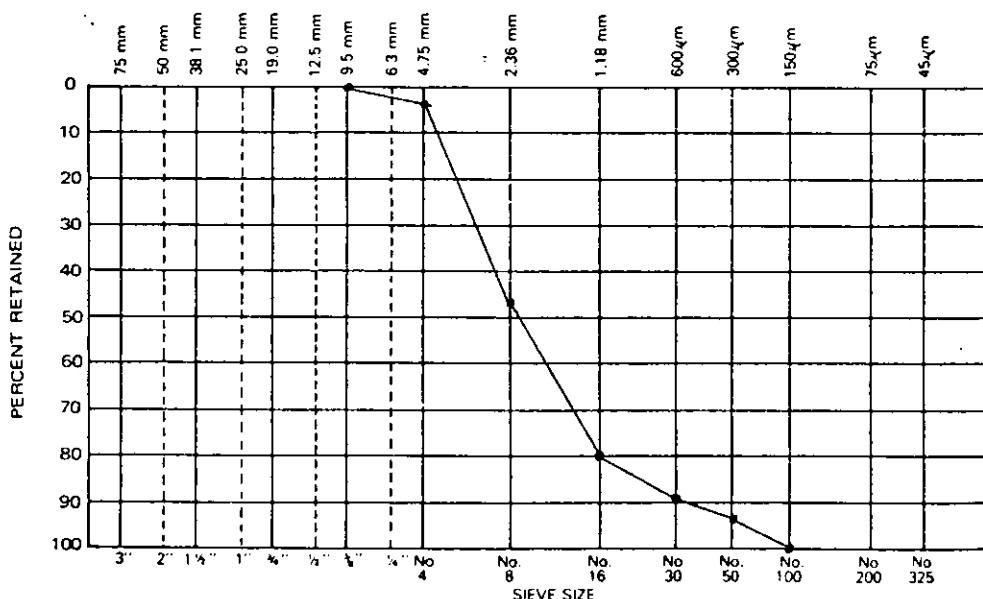
396 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 20P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 54C PARTICLE SIZE DISTRIBUTIONSulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 305.3gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 9.6 %Coefficient of Uniformity: 5.1

Comments:

Sieve Size

Percent Retained*

1"	(25.0 mm)
3/4"	(19.0 mm)
1/2"	(12.5 mm)
3/8"	(9.5 mm)
No. 4	(4.75 mm)
No. 8	(2.36 mm)
No. 16	(1.18 mm)
No. 30	(600 μ m)
No. 50	(300 μ m)
Minus No. 50	

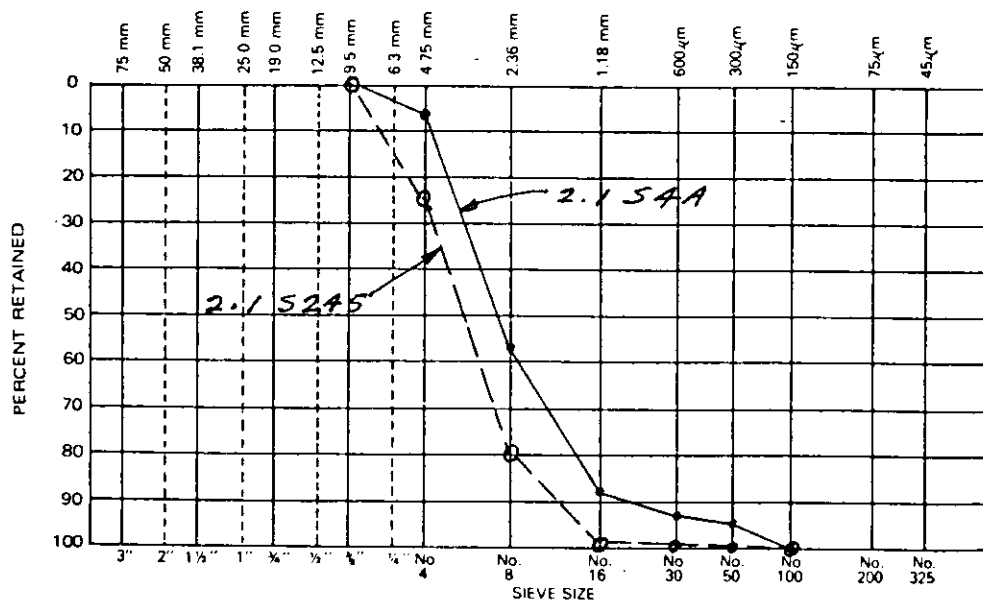
3.341.932.49.43.59.6*To nearest
0.1%

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 21P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**2.1 54A PARTICLE SIZE DISTRIBUTION****2.1 5245 (REFERENCE, NOT TUMBLED)**Sulphur Form: FLETCHER, PRILLProducer: BURZAGross Sample Size: 291.0 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 4.7%Coefficient of Uniformity: 3.0 (54A)Comments: 2.5 (5245)

Sieve Size		Percent Retained*
		<u>54A</u>
1"	(25.0 mm)	
3/4"	(19.0 mm)	
1/2"	(12.5 mm)	
3/8"	(9.5 mm)	
No. 4	(4.75 mm)	<u>6.5</u>
No. 8	(2.36 mm)	<u>51.2</u>
No. 16	(1.18 mm)	<u>30.0</u>
No. 30	(600 μ m)	<u>5.6</u>
No. 50	(300 μ m)	<u>2.0</u>
Minus No. 50		<u>4.7</u>

* To nearest
0.1%

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(404) 873-4761JOB NO. G-8657 SHEET 22P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/26/83**2.1 54B****PARTICLE SIZE DISTRIBUTION**Sulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 250.5Date Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet KDry Fines Content (% Minus No. 50): 5.4%Coefficient of Uniformity: 3.4

Comments:

Sieve Size

Percent
Retained*

1" (25.0 mm)

3/4" (19.0 mm)

1/2" (12.5 mm)

3/8" (9.5 mm)

No. 4 (4.75 mm)

No. 8 (2.36 mm)

No. 16 (1.18 mm)

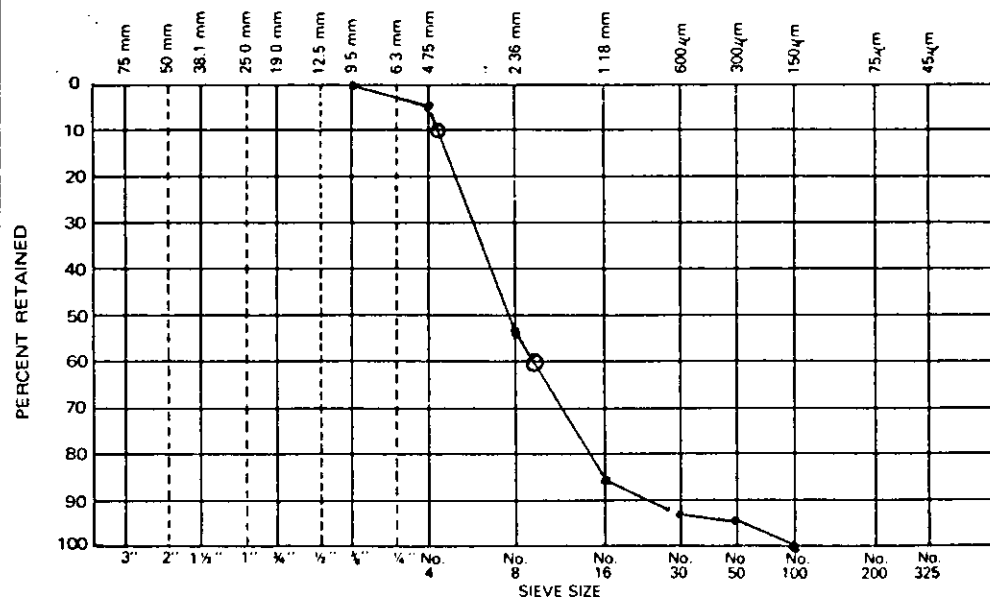
No. 30 (600 μ m)No. 50 (300 μ m)

Minus No. 50

5.147.433.66.32.15.4

* To nearest

0.1%



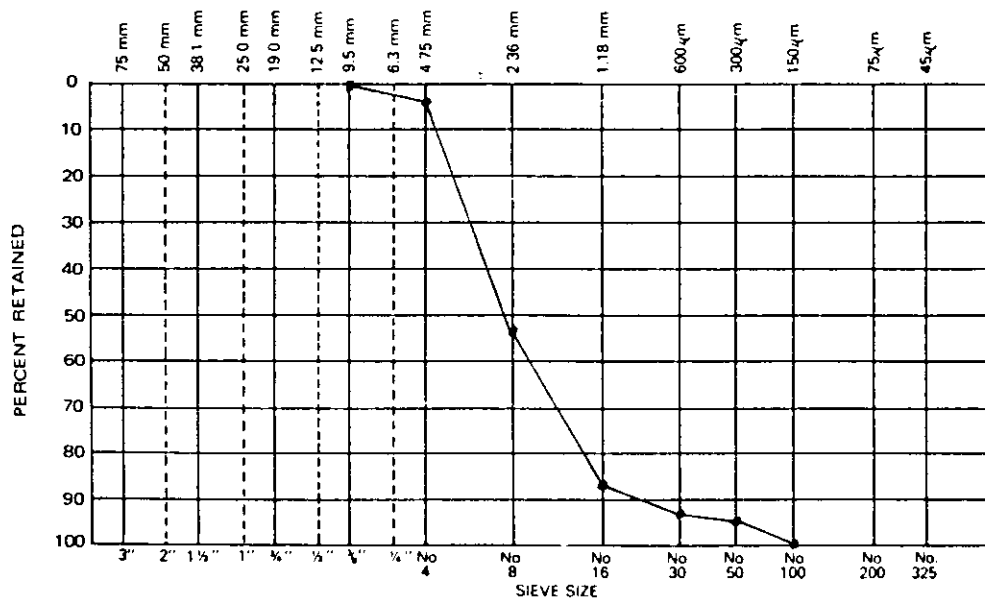
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(404) 873-4761JOB NO. G-8657 SHEET 23P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**2.1 SAC****PARTICLE SIZE DISTRIBUTION**Sulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 235.6 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/17/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 5.0%Coefficient of Uniformity: 3.4

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	4.1
No. 8 (2.36 mm)	49.4
No. 16 (1.18 mm)	34.1
No. 30 (600 μ m)	5.5
No. 50 (300 μ m)	1.9
Minus No. 50	5.0

*To nearest
0.1%

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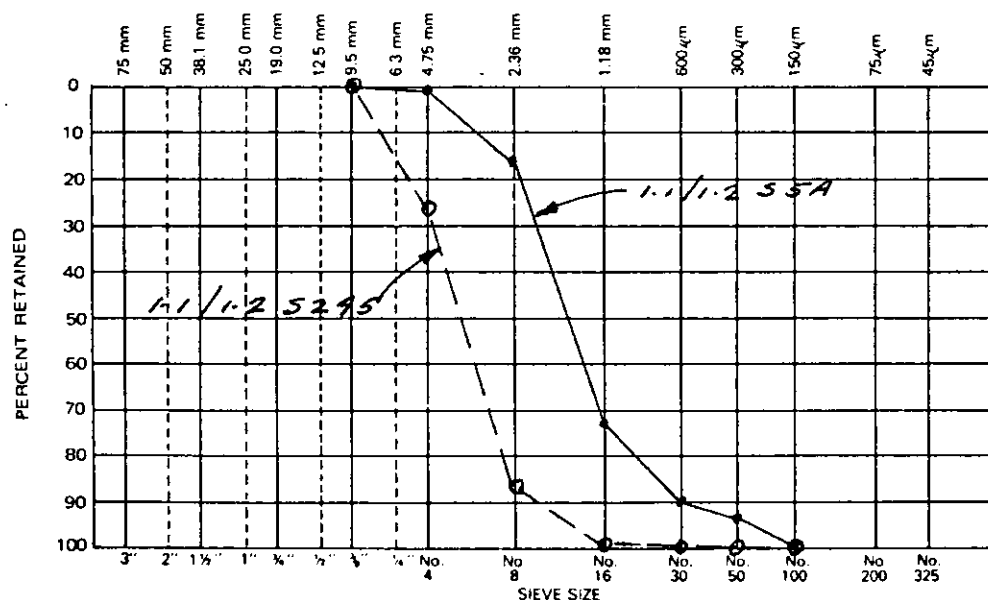
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(404) 873-4761JOB NO. G-8657 SHEET 24P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 55A

PARTICLE SIZE DISTRIBUTION

1.1/1.2 5245 (REFERENCE, NOT TUMBLED)Sulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 5-61.3 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 5.7%Coefficient of Uniformity: 3.3 (55A)Comments: 2.1 (5245)

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	0.13
No. 8 (2.36 mm)	17.6
No. 16 (1.18 mm)	55.3
No. 30 (600 µm)	16.2
No. 50 (300 µm)	5.1
Minus No. 50	5.7

*To nearest
0.1%

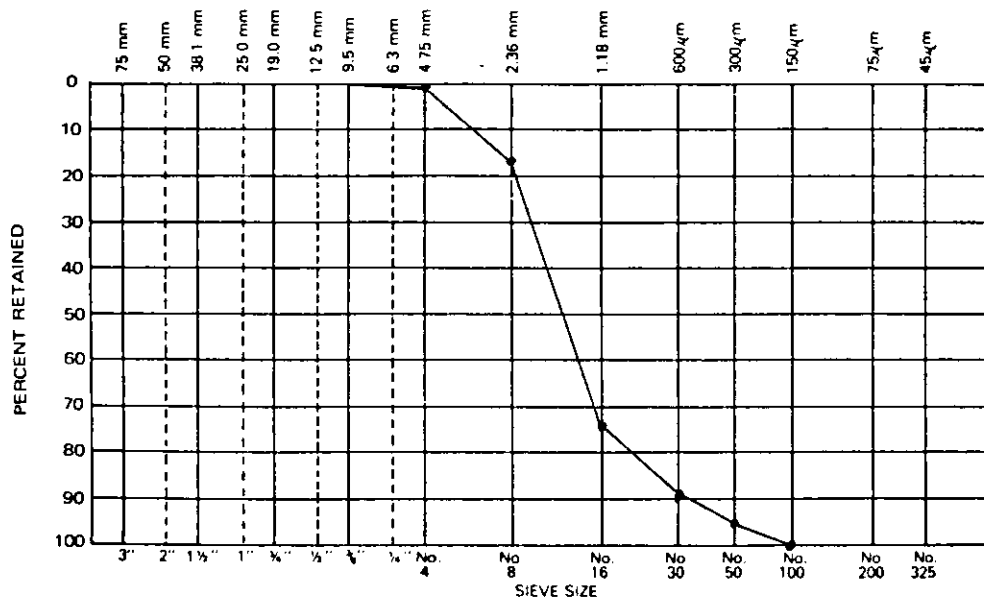
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(404) 873-4761JOB NO. G-8657 SHEET 25P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 55B PARTICLE SIZE DISTRIBUTIONSulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 556.68Date Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ☒Dry ☐Fines Content (% Minus No. 50): 5.5%Coefficient of Uniformity: 3.3

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	0.2
No. 8 (2.36 mm)	17.7
No. 16 (1.18 mm)	56.1
No. 30 (600 μ m)	15.5
No. 50 (300 μ m)	5.5
Minus No. 50	5.5

*To nearest
0.1%

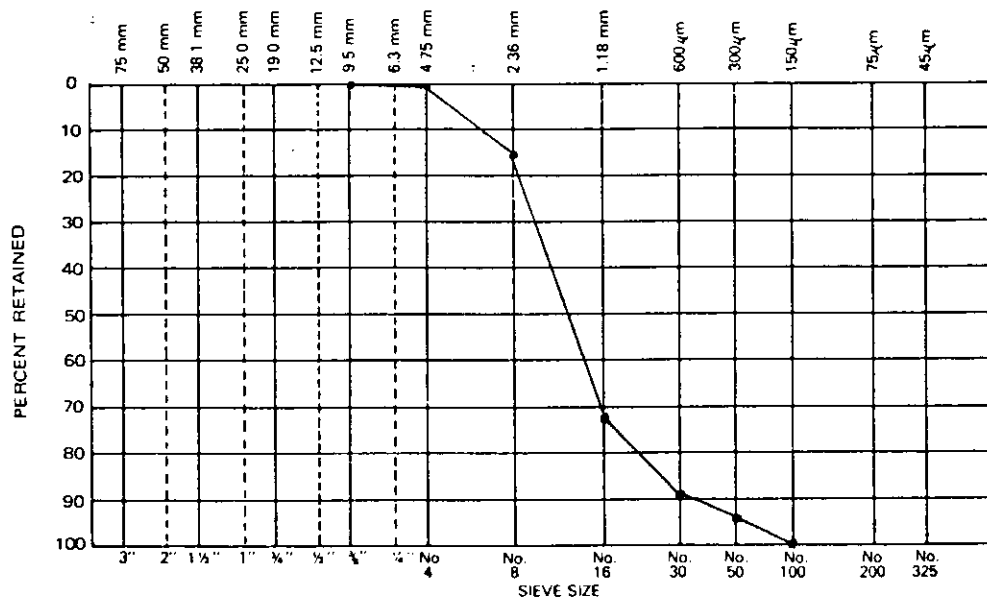
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(404) 873-4761JOB NO. G-8657 SHEET 26P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/831.1/1.2 55C PARTICLE SIZE DISTRIBUTIONSulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 491.5 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/7/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 5.8%Coefficient of Uniformity: 3.6

Comments:

Sieve Size	Percent Retained*
1" (25.0 mm)	
3/4" (19.0 mm)	
1/2" (12.5 mm)	
3/8" (9.5 mm)	
No. 4 (4.75 mm)	0.1
No. 8 (2.36 mm)	15.8
No. 16 (1.18 mm)	56.6
No. 30 (600 μ m)	16.6
No. 50 (300 μ m)	5.1
Minus No. 50	5.8

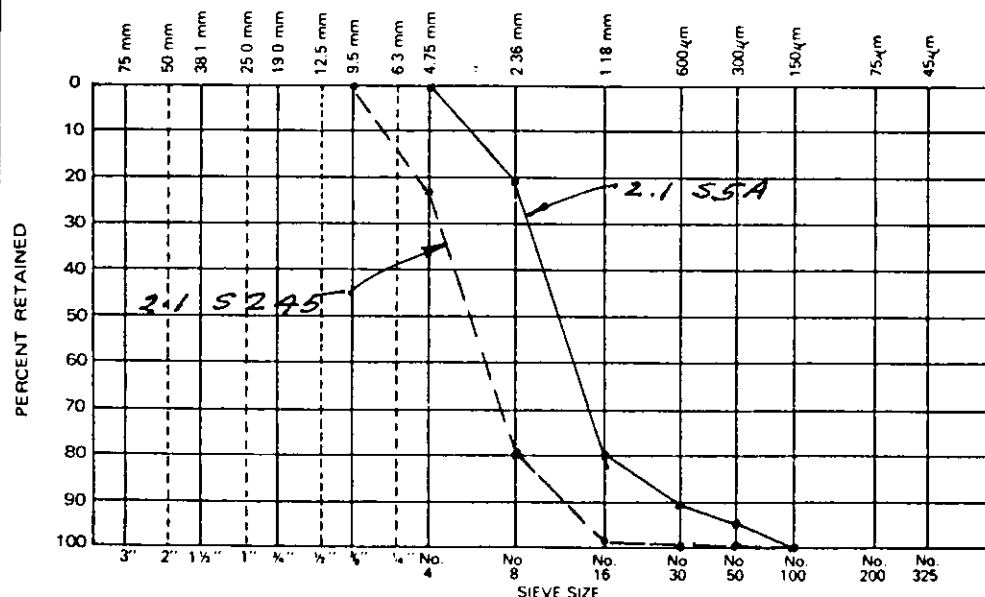
*To nearest
0.1%

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(404) 873-4761JOB NO. G-8657 SHEET 27P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY ga DATE 1/25/83**2.1 55A****PARTICLE SIZE DISTRIBUTION****2.1 5245 (REFERENCE, NOT TUMBLED**Sulphur Form; **FLETCHER PRILL**Producer: **BURZA**Gross Sample Size: **564.7g**Date Formed: **10/28/82**Date Received: **10/28/82**Date Tested: **12/9/82**Sieve Analysis: Wet ☒Dry ☐Fines Content (% Minus No. 50): **5.1%**Coefficient of Uniformity: **3.4 (55A)** Minus No. 50Comments: **2.4 (5245)**

Sieve Size		Percent Retained*
1"	(25.0 mm)	55A
3/4"	(19.0 mm)	
1/2"	(12.5 mm)	
3/8"	(9.5 mm)	
No. 4	(4.75 mm)	0.2
No. 8	(2.36 mm)	21.1
No. 16	(1.18 mm)	58.3
No. 30	(600 μ m)	11.8
No. 50	(300 μ m)	3.5
		5.1

*To nearest
0.1%

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(404) 873-4761JOB NO. G-8657 SHEET 28P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/832.155B**PARTICLE SIZE DISTRIBUTION**Sulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 514.8 gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/9/82Sieve Analysis: Wet ☒Dry ☐Fines Content (% Minus No. 50): 4.9%Coefficient of Uniformity: 3.4

Comments:

Sieve Size

Percent
Retained*

1" (25.0 mm)

3/4" (19.0 mm)

1/2" (12.5 mm)

3/8" (9.5 mm)

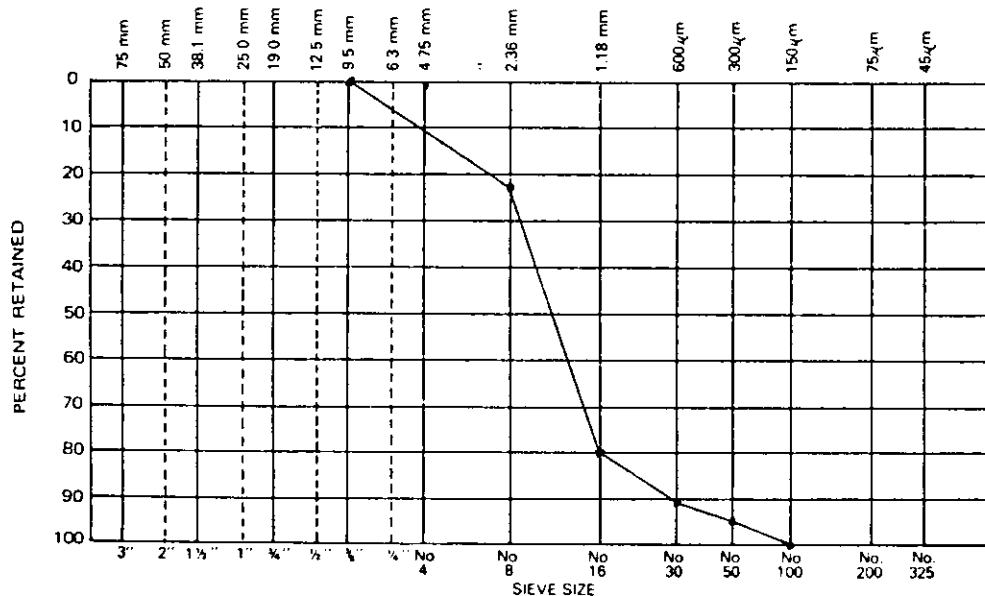
No. 4 (4.75 mm)

No. 8 (2.36 mm)

No. 16 (1.18 mm)

No. 30 (600 μ m)No. 50 (300 μ m)

Minus No. 50

0.121.758.611.23.54.9* To nearest
0.1%

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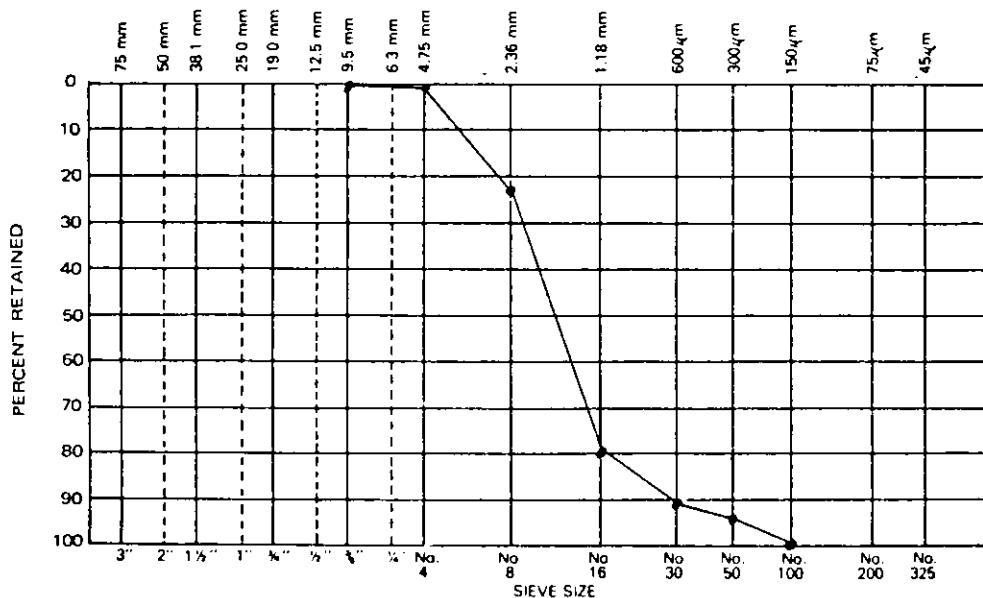
396 PLASTERS AVENUE, N.E.
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(404) 873-4761JOB NO. G-8657 SHEET 29P OF 29PJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/83**2.1 55C PARTICLE SIZE DISTRIBUTION**Sulphur Form: FLETCHER PRILLProducer: BURZAGross Sample Size: 570.7gDate Formed: 10/28/82Date Received: 10/28/82Date Tested: 12/9/82Sieve Analysis: Wet ✓Dry Fines Content (% Minus No. 50): 5.3%Coefficient of Uniformity: 3.4

Comments:

Sieve Size**Percent Retained***

1"	(25.0 mm)
3/4"	(19.0 mm)
1/2"	(12.5 mm)
3/8"	(9.5 mm)
No. 4	(4.75 mm)
No. 8	(2.36 mm)
No. 16	(1.18 mm)
No. 30	(600 μ m)
No. 50	(300 μ m)
Minus No. 50	

0.1
21.8
58.1
11.3
3.5
5.3

*To nearest
0.1%

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(404) 873-4761JOB NO. G-8657 SHEET 1F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 54A**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 319.3 gDate Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test ✓S5-77, 28 in Diameter Tumbler Test

Sieve Size	<u>1.1/1.2 5245</u> Lab Prepared Sample		<u>1.1/1.2 54A</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)	28.35	28.35	3.96	3.96
No. 8 (2.36 mm)	59.09	87.44	41.90	45.86
No. 16 (1.18 mm)	12.11	99.55	33.07	78.93
No. 30 (600 µm)	0.28	99.83	10.08	89.01
No. 50 (300 µm)	0.08	99.91	3.75	92.80
Minus No. 50	0.08	99.99	7.20	100.00
Total		515.07		410.46

Fineness Factor, $FF_0 = 515.07/100 = 5.15$ $FF_t = 410.46/100 = 4.10$ Fines Production (% Minus No. 50) = 6.8%Particle Breakdown Modulus, "PBM" = $\frac{5.15 - 4.10}{5.15} \times 100 = 20.4\%$

Comments:

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(404) 873-4761JOB NO. G-8657 SHEET 2F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY ga DATE 1/25/831-1/1-2 SAB**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 281.8 gDate Tested: 12/17/82Test: S4-77, 10 in Diameter Tumbler Test ☒S5-77, 28 in Diameter Tumbler Test

Sieve Size	<u>1-1/1-2 5245</u> Lab Prepared Sample		<u>1-1/1-2 SAB</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			3.16	3.16
No. 8 (2.36 mm)			38.68	41.84
No. 16 (1.18 mm)			31.83	78.67
No. 30 (600 μ m)			9.83	88.50
No. 50 (300 μ m)			3.73	92.23
Minus No. 50			7.77	100.00
Total		515.07		404.40

Fineness Factor, $FF_0 = 515.07/100 = 5.15$ $FF_t = 404.40/100 = 4.04$ Fines Production (% Minus No. 50) = 7.77%Particle Breakdown Modulus, "PBM" = $\frac{5.15 - 4.04}{5.15} \times 100 = 21.55\%$

Comments:

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(404) 873-4761JOB NO. G-8657 SHEET 3F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 54C FRIABILITY TESTINGSulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 305.3 gDate Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test ☒S5-77, 28 in Diameter Tumbler Test ☐

<u>1.1/1.2 5245</u> Lab Prepared Sample			<u>1.1/1.2 54C</u> Tumbled Sample		
Sieve Size	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained	
1" (25.0 mm)					
3/4" (19.0 mm)					
1/2" (12.5 mm)					
3/8" (9.5 mm)					
No. 4 (4.75 mm)			3.30	3.30	
No. 8 (2.36 mm)			41.86	45.16	
No. 16 (1.18 mm)			32.35	77.51	
No. 30 (600 µm)			9.42	86.93	
No. 50 (300 µm)			3.46	90.39	
Minus No. 50			9.61	100.00	
Total		515.07		403.29	

Fineness Factor, $FF_0 = 515.07/100 = 5.15$ $FF_t = 403.29/100 = 4.03$ Fines Production (% Minus No. 50) = 6.65%Particle Breakdown Modulus, "PBM" = $\frac{5.15 - 4.03}{5.15} \times 100 = 21.75$

Comments:

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(404) 873-4761JOB NO. G-8657 SHEET 4F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY ba DATE 1/25/832.1 54A**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 291.0 gDate Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test ☒S5-77, 28 in Diameter Tumbler Test ☐

Sieve Size	<u>2.1 5245</u> Lab Prepared Sample		<u>2.1 54A</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)	<u>24.15</u>	<u>24.15</u>	<u>6.46</u>	<u>6.46</u>
No. 8 (2.36 mm)	<u>55.55</u>	<u>79.70</u>	<u>51.24</u>	<u>57.70</u>
No. 16 (1.18 mm)	<u>19.84</u>	<u>99.54</u>	<u>30.03</u>	<u>87.73</u>
No. 30 (600 μ m)	<u>0.31</u>	<u>99.84</u>	<u>5.57</u>	<u>93.30</u>
No. 50 (300 μ m)	<u>0.08</u>	<u>99.92</u>	<u>1.96</u>	<u>95.26</u>
Minus No. 50	<u>0.08</u>	<u>100.00</u>	<u>4.74</u>	<u>100.00</u>
Total		<u>503.15</u>		<u>440.45</u>

Fineness Factor, $FF_0 = 503.15/100 = 5.03$ $FF_t = 440.45/100 = 4.40$ Fines Production (% Minus No. 50) = 4.74%Particle Breakdown Modulus, "PBM" = $\frac{5.03 - 4.40}{5.03} \times 100 = 12.51\%$

Comments:

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(404) 873-4761JOB NO. G-8657 SHEET 5F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/832.154B**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 250.5gDate Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test ☒S5-77, 28 in Diameter Tumbler Test ☐

Sieve Size	<u>2.15245</u> Lab Prepared Sample		<u>2.154B</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			5.11	5.11
No. 8 (2.36 mm)			47.43	52.54
No. 16 (1.18 mm)			33.61	86.15
No. 30 (600 µm)			6.31	92.46
No. 50 (300 µm)			2.12	94.58
Minus No. 50			5.43	100.01
Total		503.15		430.85

Fineness Factor, $FF_0 = 503.15/100 = 5.03$ $FF_t = 430.85/100 = 4.31$ Fines Production (% Minus No. 50) = 5.43 %Particle Breakdown Modulus, "PBM" = $\frac{5.03 - 4.31}{5.03} \times 100 = 14.31\%$

Comments:

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(404) 873-4761JOB NO. G-8657 SHEET 6F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/832.1 S4C FRIABILITY TESTING

Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82
Producer: BURZA Date Received: 10/28/82
Gross Sample Size: 235.6 g Date Tested: 12/7/82
Test: S4-77, 10 in Diameter Tumbler Test ✓
S5-77, 28 in Diameter Tumbler Test

Sieve Size	<u>2.1 S245</u> Lab Prepared Sample		<u>2.1 S4C</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			4.12	4.12
No. 8 (2.36 mm)			49.36	53.48
No. 16 (1.18 mm)			34.13	87.61
No. 30 (600 μ m)			5.52	93.13
No. 50 (300 μ m)			1.87	95.00
Minus No. 50			5.01	100.01
Total		503.15		433.35

Fineness Factor, $FF_0 = 503.15/100 = 5.03$ $FF_t = 433.35/100 = 4.33$ Fines Production (% Minus No. 50) = 5.01%Particle Breakdown Modulus, "PBM" = $\frac{5.03 - 4.33}{5.03} \times 100 = 13.92\%$

Comments:

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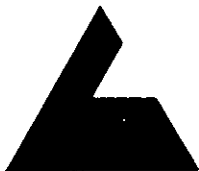
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(404) 873-4761JOB NO. G-8657 SHEET 7F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 55A FRIABILITY TESTINGSulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZA Date Received: 10/28/82Gross Sample Size: 561.3 g Date Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test ✓S5-77, 28 in Diameter Tumbler Test ✓

Sieve Size	<u>1.1/1.2 5245</u> Lab Prepared Sample		<u>1.1/1.2 55A</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)	<u>28.35</u>	<u>28.35</u>	<u>0.13</u>	<u>0.13</u>
No. 8 (2.36 mm)	<u>59.09</u>	<u>87.44</u>	<u>17.62</u>	<u>17.75</u>
No. 16 (1.18 mm)	<u>12.11</u>	<u>99.55</u>	<u>55.30</u>	<u>73.05</u>
No. 30 (600 μ m)	<u>0.28</u>	<u>99.83</u>	<u>16.16</u>	<u>89.21</u>
No. 50 (300 μ m)	<u>0.08</u>	<u>99.91</u>	<u>5.11</u>	<u>94.32</u>
Minus No. 50	<u>0.08</u>	<u>99.99</u>	<u>5.68</u>	<u>100.00</u>
Total		<u>515.07</u>		<u>374.46</u>

Fineness Factor, $FF_0 = 515.07/100 = 5.15$ $FF_t = 374.46/100 = 3.74$ Fines Production (% Minus No. 50) = 5.68 %Particle Breakdown Modulus, "PBM" = $\frac{5.15 - 3.74}{5.15} \times 100 = 25.44\%$

Comments:

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

396 PLASTERS AVENUE, N.E.
P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 8F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY sa DATE 1/25/831-1/1.2 55B FRIABILITY TESTINGSulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZA Date Received: 10/28/82Gross Sample Size: 556.6 g Date Tested: 12/1/82Test: S4-77, 10 in Diameter Tumbler Test S5-77, 28 in Diameter Tumbler Test ✓

Sieve Size	<u>1-1/1.2 5245</u> Lab Prepared Sample		<u>1-1/1.2 55B</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			0.18	0.18
No. 8 (2.36 mm)			17.71	17.89
No. 16 (1.18 mm)			56.14	74.02
No. 30 (600 μ m)			15.54	89.56
No. 50 (300 μ m)			4.96	94.52
Minus No. 50			5.46	99.99
Total		515.07		376.16

Fineness Factor, $FF_0 = 515.07/100 = 5.15$ $FF_t = 376.16/100 = 3.76$ Fines Production (% Minus No. 50) = 5.46%Particle Breakdown Modulus, "PBM" = $\frac{5.15 - 3.76}{5.15} \times 100 = 25.05\%$

Comments:

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 9F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/831.1/1.2 55C**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/15/82Producer: BURZA Date Received: 10/28/82Gross Sample Size: 491.5 g Date Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test S5-77, 28 in Diameter Tumbler Test ✓

Sieve Size	<u>1.1/1.2 5245</u> Lab Prepared Sample		<u>1.1/1.2 55C</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			<u>0.10</u>	<u>0.10</u>
No. 8 (2.36 mm)			<u>15.77</u>	<u>15.87</u>
No. 16 (1.18 mm)			<u>56.60</u>	<u>72.47</u>
No. 30 (600 µm)			<u>16.56</u>	<u>89.03</u>
No. 50 (300 µm)			<u>5.13</u>	<u>94.16</u>
Minus No. 50			<u>5.84</u>	<u>100.00</u>
Total		<u>515.07</u>		<u>371.63</u>

Fineness Factor, $FF_0 = 515.07/100 = 5.15$ $FF_t = 371.63/100 = 3.72$ Fines Production (% Minus No. 50) = 5.84%Particle Breakdown Modulus, "PBM" = $\frac{5.15 - 3.72}{5.15} \times 100 = 25.83\%$

Comments:

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 10F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/832.1 55A**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 564.7gDate Tested: 12/9/82Test: S4-77, 10 in Diameter Tumbler Test S5-77, 28 in Diameter Tumbler Test ✓

Sieve Size	<u>2.1 5245</u> Lab Prepared Sample		<u>2.1 55A</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)	<u>24.15</u>	<u>24.15</u>	<u>0.15</u>	<u>0.15</u>
No. 8 (2.36 mm)	<u>55.55</u>	<u>79.70</u>	<u>21.14</u>	<u>21.29</u>
No. 16 (1.18 mm)	<u>19.84</u>	<u>99.54</u>	<u>58.28</u>	<u>79.57</u>
No. 30 (600 μ m)	<u>0.31</u>	<u>99.84</u>	<u>11.79</u>	<u>91.36</u>
No. 50 (300 μ m)	<u>0.08</u>	<u>99.92</u>	<u>3.54</u>	<u>94.90</u>
Minus No. 50	<u>0.08</u>	<u>100.00</u>	<u>5.10</u>	<u>100.00</u>
Total		<u>503.15</u>		<u>387.27</u>

Fineness Factor, $FF_0 = 503.15/100 = 5.03$ $FF_t = 387.27/100 = 3.87$ Fines Production (% Minus No. 50) = 5.10 %Particle Breakdown Modulus, "PBM" = $\frac{5.03 - 3.87}{5.03} \times 100 = 23.06$

Comments:

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 11F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY GA DATE 1/25/832.155B**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/25/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 514.8gDate Tested: 12/19/82

Test: S4-77, 10 in Diameter Tumbler Test

S5-77, 28 in Diameter Tumbler Test ✓

Sieve Size	<u>2.15245</u> Lab Prepared Sample		<u>2.155B</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			0.09	0.09
No. 8 (2.36 mm)			21.72	21.81
No. 16 (1.18 mm)			58.64	80.45
No. 30 (600 μ m)			11.21	91.66
No. 50 (300 μ m)			3.46	95.12
Minus No. 50			4.88	100.00
Total		503.15		389.13

Fineness Factor, $FF_0 = 503.15/100 = 5.03$ $FF_t = 389.13/100 = 3.89$ Fines Production (% Minus No. 50) = 4.88Particle Breakdown Modulus, "PBM" = $\frac{5.03 - 3.89}{5.03} \times 100 = 22.66\%$

Comments:

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 12F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/832.1 55C**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZADate Received: 10/28/82Gross Sample Size: 570.7gDate Tested: 12/9/82Test: S4-77, 10 in Diameter Tumbler Test S5-77, 28 in Diameter Tumbler Test ✓

Sieve Size	<u>2.1 5245</u> Lab Prepared Sample		<u>2.1 55C</u> Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			0.12	0.12
No. 8 (2.36 mm)			21.80	21.92
No. 16 (1.18 mm)			58.07	79.99
No. 30 (600 µm)			11.25	91.24
No. 50 (300 µm)			3.49	94.73
Minus No. 50			5.27	100.00
Total		503.15		388.00

Fineness Factor, $FF_0 = 503.15/100 = 5.03$ $FF_t = 388.00/100 = 3.88$ Fines Production (% Minus No. 50) = 5.27%Particle Breakdown Modulus, "PBM" = $\frac{5.03 - 3.88}{5.03} \times 100 = 22.86\%$

Comments:

**LAW ENGINEERING TESTING COMPANY**

geotechnical, environmental & construction materials consultants

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 13F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/28/831-1/1.2 S 245
REFERENCE
NOT TUMBLED**FRIABILITY TESTING**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZA Date Received: 10/28/82Gross Sample Size: 246.9 g Date Tested: 12/19/82Test: S4-77, 10 in Diameter Tumbler Test NAS5-77, 28 in Diameter Tumbler Test NA

Sieve Size	Lab Prepared Sample		SIEVE Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			<u>28.35</u>	<u>28.35</u>
No. 8 (2.36 mm)	<u>28.1</u>	<u>28.1</u>	<u>59.09</u>	<u>87.44</u>
No. 16 (1.18 mm)	<u>11.9</u>	<u>100.0</u>	<u>12.11</u>	<u>99.55</u>
No. 30 (600 µm)		<u>100.0</u>	<u>0.28</u>	<u>99.84</u>
No. 50 (300 µm)		<u>100.0</u>	<u>0.08</u>	<u>99.92</u>
Minus No. 50		<u>100.0</u>	<u>0.08</u>	<u>100.00</u>
Total		<u>488.1</u>		<u>515.10</u>

Fineness Factor, $FF_o = 488.1/100 = 4.88$ $FF_t = 515.1/100 = 5.15$ Fines Production (% Minus No. 50) = NAParticle Breakdown Modulus, "PBM" = NA

Comments:

**LAW ENGINEERING TESTING COMPANY**

Geotechnical, environmental & construction materials consultants

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P.O. BOX 13260 • ATLANTA, GEORGIA 30324
(404) 873-4761JOB NO. G-8657 SHEET 14F OF 14FJOB NAME BURZA Sulfur PrillBY G.V. Aseff DATE 1/24/83CHECKED BY [Signature] DATE 1/25/83**2.1 5245 FRIABILITY TESTING****REFERENCE****NOT TUMBLED**Sulphur Form: FLETCHER PRILL Date Formed: 10/28/82Producer: BURZA Date Received: 10/28/82Gross Sample Size: 356.4 Date Tested: 12/7/82Test: S4-77, 10 in Diameter Tumbler Test NAS5-77, 28 in Diameter Tumbler Test NA

Sieve Size	Lab Prepared Sample		SIEVED Tumbled Sample	
	% Retained	Total Cumulative % Retained	% Retained	Total Cumulative % Retained
1" (25.0 mm)				
3/4" (19.0 mm)				
1/2" (12.5 mm)				
3/8" (9.5 mm)				
No. 4 (4.75 mm)			24.15	24.15
No. 8 (2.36 mm)	57.4	57.4	55.55	79.69
No. 16 (1.18 mm)	42.6	100.0	19.84	99.53
No. 30 (600 μ m)		100.0	0.31	99.84
No. 50 (300 μ m)		100.0	0.08	99.92
Minus No. 50		100.0	0.08	100.00
Total		457.4		

Fineness Factor, $FF_0 =$ $FF_t =$ Fines Production (% Minus No. 50) = NAParticle Breakdown Modulus, "PBM" = NA

Comments:

HALL, ESTILL, HARDWICK, GABLE, COLLINGSWORTH & NELSON

A PROFESSIONAL CORPORATION
4100 BANK OF OKLAHOMA TOWER
ONE WILLIAMS CENTER
TULSA, OKLAHOMA 74172
TELEPHONE (918) 588-2700

OF COUNSEL
AT TULSA, OKLAHOMA

WALTER B. HALL
RALPH L. ABERCROMBIE

February 8, 1983

WASHINGTON, D. C. OFFICE
1750 K STREET, N.W.
WASHINGTON, D. C. 20006
(202) 833-8990

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WRITER'S DIRECT DIAL NUMBER
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ROBERT P. LEGG
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JOHN E. ROONEY, JR.

Steve Smallwood, P.E.
Chief, Bureau of Air Quality Management
State of Florida
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32301

Dear Mr. Smallwood:

This letter will confirm our willingness to further assist the Department in resolving the technical issues remaining concerning regulation by rule of the handling of wet prilled sulfur. It is Agrico's position that concerns regarding the emission of sulfur dust from wet prilled sulfur are unfounded and that data allaying these concerns is presently on file. If an additional "mediation-type" process would significantly advance DER's assurance as to the positive environmental qualities of wet prilled sulfur, Agrico is willing to cooperate.

We suggest the following process of negotiation and testing:

1. The Department will provide a list of three consultants capable of performing the air quality analysis. Opponents of wet-prilled sulfur can strike one name, proponents one name, and DER will retain the remaining consultant. Proponents and opponents will split the consultant's cost evenly.
2. Proponents and opponents will meet with DER to negotiate the scope of question(s) to be answered and test(s) to be run. If agreement can not be achieved as to the proper question(s) and test(s), the parties will not proceed.

DER

FEB 09 1983

BAQM



PLEASE COMPLETE ALL INFORMATION IN THE 5 BLOCKS OUTLINED IN ORANGE
SEE BACK OF FORM SET FOR COMPLETE PREPARATION INSTRUCTIONS

AIRBILL NUMBER
241376214



YOUR FEDERAL EXPRESS ACCOUNT NUMBER
00741 **0121-0311-0**

DATE
2/8/83

FROM (Your Name)
Matthew G. Livingood

TO (Recipient's Name)
Steve Smallwood

If Hold For Pick-Up or Saturday Delivery
Recipient's Phone Number

COMPANY
FLA. Dept. of Environmental Regulation

COMPANY
FLA. Dept. of Environmental Regulation

STREET ADDRESS
2600 Blair Stone Road

STREET ADDRESS (P.O. BOX NUMBERS ARE NOT DELIVERABLE)
2600 Blair Stone Road

CITY
Tallahassee

CITY
Tallahassee

STATE
FL

STATE
FL

AIRBILL NO. **241376214**

ZIP ACCURATE ZIP CODE REQUIRED FOR OVERNIGHT DELIVERY
323102

YOUR NOTES/REFERENCE NUMBERS (FIRST 12 CHARACTERS WILL ALSO APPEAR ON INVOICE)

IN TENDERING THIS SHIPMENT SHIPPER AGREES THAT
F.E.C. SHALL NOT BE LIABLE FOR SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES ARISING FROM
CARRIAGE HEREOF. F.E.C. DIS-
CLAIMS ALL WARRANTIES, EXPRESS OR IMPLIED, WITH
RESPECT TO THIS SHIPMENT. THIS IS A NON-NEGOTIABLE
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FEDERAL EXPRESS USE
FREIGHT CHARGES
DECLARED VALUE CHARGE

SERVICES CHECK ONLY ONE BOX		DELIVERY AND SPECIAL HANDLING CHECK SERVICES REQUIRED
PRIORITY 1 1 <input type="checkbox"/> OVERNIGHT PACKAGES (up to 70 LBS.) COURIER PAK 2 <input type="checkbox"/> OVERNIGHT ENVELOPE (up to 2 LBS.) 3 <input type="checkbox"/> OVERNIGHT BOX (up to 5 LBS.) 4 <input type="checkbox"/> OVERNIGHT TUBE (up to 5 LBS.) STANDARD AIR 5 <input type="checkbox"/> DELIVERY 2ND BUSINESS DAY FOLLOWING PICK UP (up to 70 LBS.)	OVERNIGHT LETTER 1 <input checked="" type="checkbox"/> (up to 10 LBS.) 2 <input type="checkbox"/> (up to 2 LBS.) 3 <input type="checkbox"/> (up to 5 LBS.) 4 <input type="checkbox"/> (up to 5 LBS.) 5 <input type="checkbox"/> (up to 5 LBS.)	1 <input type="checkbox"/> HOLD-UP PICK UP AT DESTINATION FEDERAL EXPRESS LOCATION AND/OR IN SERVICE GUARD REC'D PHONE NUMBER REQUIRED 2 <input checked="" type="checkbox"/> DELIVER 3 <input type="checkbox"/> SATURDAY SERVICE REQUIRED See Reverse (Extra charge applies for delivery) 4 <input type="checkbox"/> RESTRICTED ARTICLES SERVICE (P.1 and Standard Air Packages only: extra charge) 5 <input type="checkbox"/> SSS (Signature Security Service required: extra charge applies) 6 <input type="checkbox"/> DRY ICE _____ LBS. 7 <input type="checkbox"/> OTHER SPECIAL SERVICE _____ 8 <input type="checkbox"/> _____ 9 <input type="checkbox"/> _____

PACKAGES	WEIGHT	DECLARED VALUE	O/S	EMP. NO.	DATE
				<input type="checkbox"/> CASH RECEIVED	AGT/PRO
				<input type="checkbox"/> RETURN SHIPMENT	AGT/PRO
				<input type="checkbox"/> THIRD PARTY	AGT/PRO
				<input type="checkbox"/> CHG TO DEL <input type="checkbox"/> CHG TO HOLD	
TOTAL				STREET ADDRESS	
TOTAL				CITY	
TOTAL				STATE	
TOTAL				ZIP	
RECEIVED BY SHIPPER'S DOOR OR REGULAR STOP <input type="checkbox"/> ON CALL STOP <input type="checkbox"/> F.E.C. LOC				RECEIVED BY: (Signature) X	
DATE/TIME For Federal Express Use 02-08-1715				DATE/TIME RECEIVED	
				F.E.C. EMPLOYEE NUMBER	

OVERNIGHT IS NEXT BUSINESS DAY (MONDAY THROUGH FRIDAY); TWO DAYS FROM ALASKA/HAWAII; SATURDAY DELIVERY AVAILABLE IN CONTINENTAL U.S. SEE "SPECIAL HANDLING."

PART
#2041730751
FEC-S-0751 D/O/B
REVISION DATE
7:82 NCR
PRINTED U.S.A.

RECIPIENT COPY (AFFIXED TO PACKAGE, GIVEN TO RECIPIENT AT DELIVERY)

Steve Smallwood
February 8, 1983
page 2

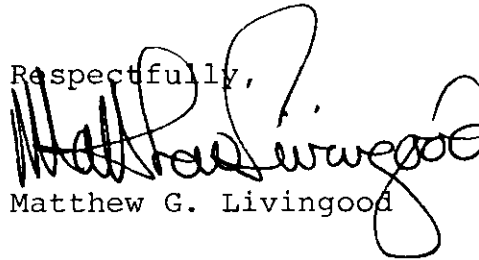
3. DER will arrange with the consultant to execute the tests.

4. Upon selection of the consultant, proponents and opponents will have no contact with the consultant. All contacts (except as to delivery of and access to the test material) will be through DER. Similarly, the consultant will be bound to refuse any and all contacts with anyone except DER's representative.

5. Upon submittal of test results to DER, neither proponents nor opponents will challenge the results nor submit any further technical material to the rulemaking record. Neither will any party supply any technical assistance or information to non-parties which could result in either challenge to the test results, or submission of further technical material to the rulemaking record by those non-parties.

If we can be of any further specific assistance, please do not hesitate to call.

Respectfully,



Matthew G. Livingood

MGL:ml

xc: Don Morrow
C. J. Head
Bobby Latham
Steve Savage
Michael Graves
Ed de la Parte
Hal Scott

MEMORANDUM

TO: Steve Smallwood
FROM: Judith S. Kavanaugh
DATE: February 1, 1983
RE: Questions Regarding Lundgren Test Method

As per our discussions, in order for our consultants to duplicate Dr. Lundgren's tests with the black box, in support of developing a reliable emission factor for solid sulphur, certain additional information not available from Dr. Lundgren's reports and testimony thus far is necessary. At your suggestion, I am submitting these questions to you, and would appreciate your forwarding them to Dr. Lundgren or otherwise providing the information we need. As you know, we believe it will be preferable for Freeport and Agrico to work together, and we continue to urge mutual cooperation. It is my understanding that Agrico has reservations about any contact between Agrico and Freeport because of a legal question as to whether or not this would be some sort of admission detrimental to Agrico. As I have indicated to you earlier, I do not agree with this legal concern, but Freeport stands willing to attempt to overcome any such reservations Agrico might have in order to accomplish the most objective technically correct analysis of the issues possible.

The following information is necessary and requested regarding the Lundgren testing:

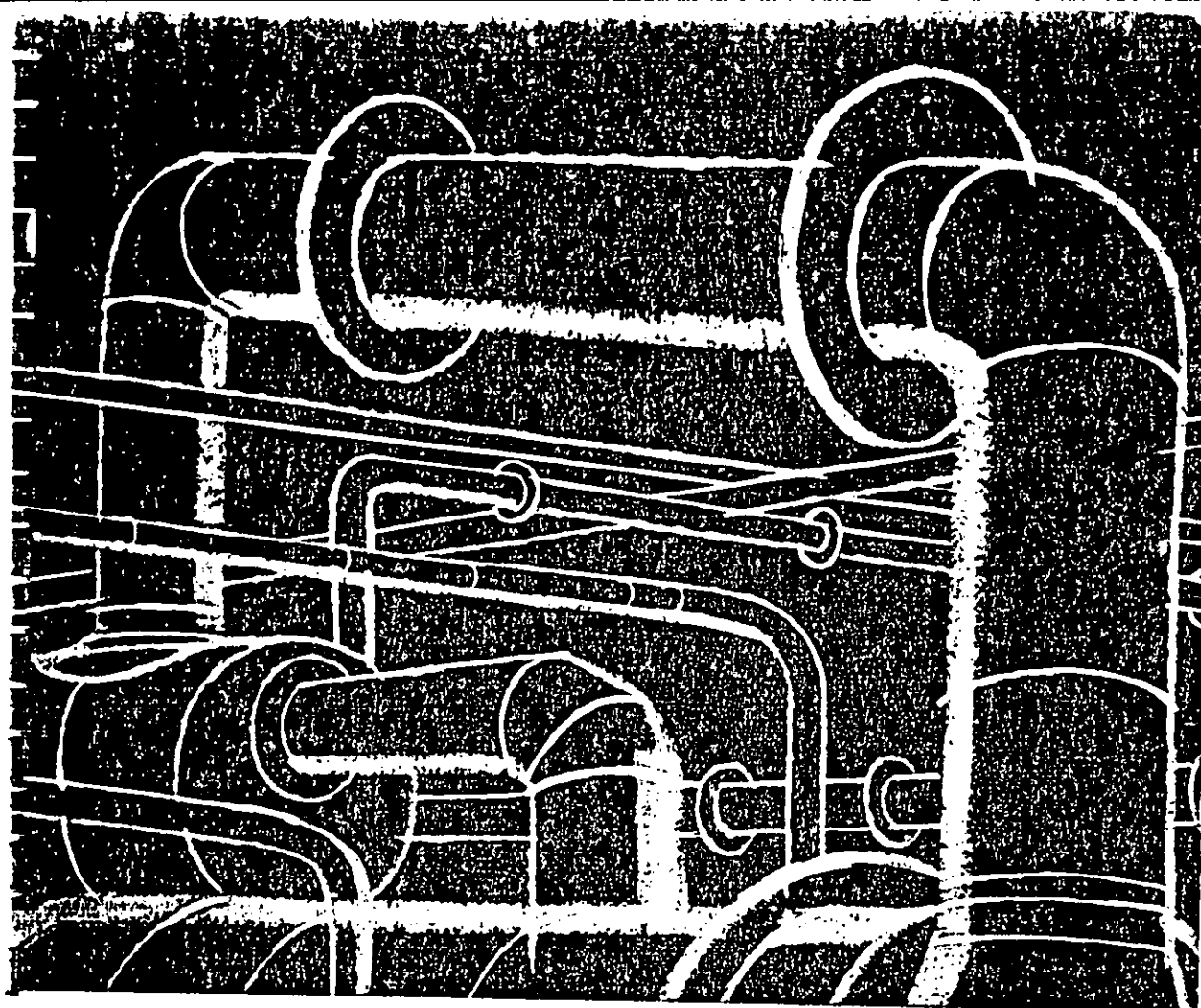
1. How long did Dr. Lundgren run the Hi-Vol during the test to collect the particles gathered?
2. Did Dr. Lundgren do any analysis of the background levels of particulate in the lab, not just sulphur, but any other particular which may have been suspended in ambient conditions?

3. How long did it take to pour the samples into the box?

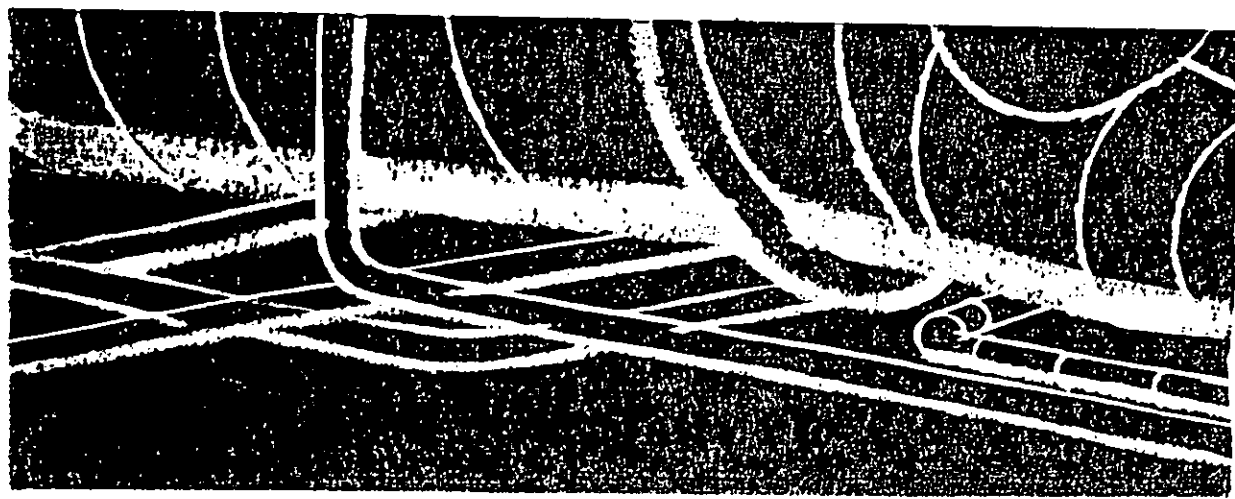
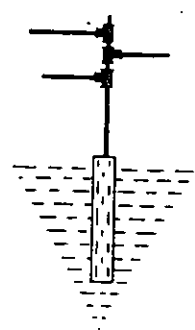
4. Please provide more details as to the wetting of the material, including time, any mechanical efforts to wet, or use of any wetting agents.

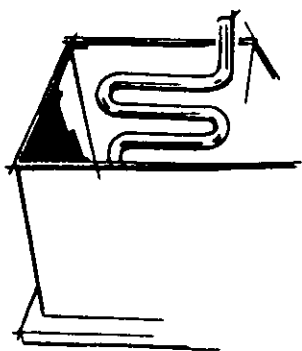
5. How did Dr. Lundgren measure the fines in the original material to calibrate to the 200 mesh particles?

I would appreciate receiving this information as soon as possible so that there will be no delay in further testing programs by Freeport. As usual I thank you for your assistance in this regard and look forward to hearing from you soon.



**FREEPORT
SULPHUR
HANDBOOK**





Sulphur Melting

Section Five

GENERAL OPERATING FACTORS

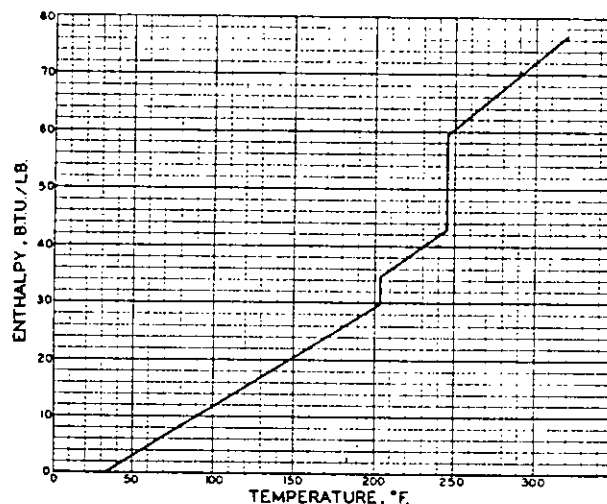
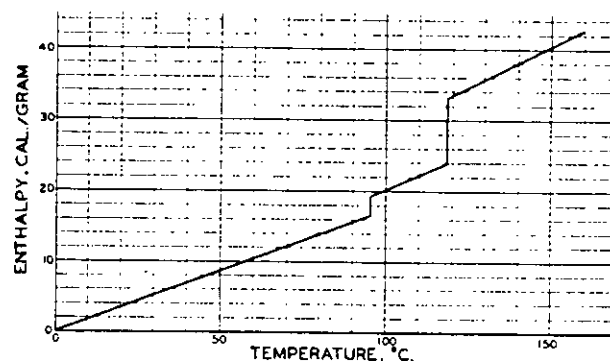
Liquid sulphur should be maintained at temperatures between 265° and 290°F. This range is high enough above the freezing point of about 240°F. to avoid local solidification. It is also sufficiently below the temperature at which a sudden increase in the viscosity of bright sulphur occurs, which is 320°F.

The fusion heat of rhombic sulphur is 21.4 B.t.u. per pound (144 B.t.u. per pound for water). About 65 B.t.u. per pound are required to convert dry solid sulphur at 70°F. to liquid sulphur at 285°F. The heat capacity of sulphur increases roughly from 0.18 B.t.u. per pound per °F. at 0°F. to 0.25 B.t.u. at 285°F. The illustration presents a convenient enthalpy curve for sulphur over the range of normal handling temperatures, and the following table gives similar information.

Enthalpy of Sulphur in Metric and English Units

Temp., °C.	15° g.-cal./g.	B.t.u./lb.
0	0	0
25	4.18	7.53
50	8.48	15.27
75	12.92	23.26
95.5	16.63	29.93
95.5	19.31	34.75
100	20.17	36.29
118.9	23.83	42.90
118.9	33.03	59.45
125	34.43	61.97
150	40.24	72.43
160	42.62	76.72

SULPHUR MELTING



Enthalpy of Sulphur in English and Metric Units.

Severe foaming can result when sulphur is melted with a moisture content exceeding about 1%. Foaming is dependent upon the rate of sulphur feed (and the included water), the rate at which the foam breaks, and the type and efficiency of agitation. With proper agitation and continuous controlled feed, Freeport has handled sulphur containing up to 9% moisture without prohibitive foaming. Continuous feeding of regular increments minimizes the amount of water present at any one time and decreases foaming.

A moisture content of 5% will approximately double the heat required to melt sulphur. This is because of the high latent heat of vaporization of water as compared to the low heat of fusion and low specific heat of sulphur.

Freeport's experience shows that effective agitation can raise melter heat transfer rates from a maximum of about 20 B.t.u. per hour per square foot per °F., to an average of 70 B.t.u. Transfer coefficients as high as 90 B.t.u. per hour per square foot per °F. temperature difference are possible. Locating the heating coils at the point of maximum agitation and, if possible, perpendicular to the direction of sulphur flow has been found to aid heat transfer. Adequate surface and subsurface agitation is needed to minimize foam formation, to rapidly disperse the solid sulphur into the liquid pool, and to pull the solid-liquid mixture down from the surface through the coils. Mechanical agitation is preferred to direct steam or air agitation because it provides smoother heat flow, minimizes channeling, and avoids corrosion problems introduced by moisture from steam or wet air.

Sulphur melting equipment can ordinarily be fabricated of mild steel and common red brick. Heating coils of black iron pipe in pans of mild steel, ordinary brick, or concrete have been used successfully. Some designers prefer to use acid-proof brick at the sulphur-air interface in the steel and concrete pan. Mortar for brick can be based on either common Portland, high-alumina, or acid-proof cement. The seams should be as thin as is practical to minimize erosion.

For the heating medium, steam is preferred to burner gas or flue gas. The operating temperatures are lower and formation of locally overheated, viscous sulphur coatings on heat exchange surfaces is prevented. When steam coils inside a pit are used, they should be located in the lower sections of the melter. This will keep them submerged and in contact with the liquid sulphur, which will facilitate heat transfer. It will also reduce the corrosion caused by alternate exposure of the coils to air and sulphur. Installation of a sacrificial outer pipe jacket in the sulphur-air interface zone on the steam supply and condensate return lines has been helpful in reducing coil corrosion. These jackets may be made of oversized pipes welded to the main pipes, with the annulus filled with cement. Noble alloys such as Type 316 stainless steel have been used successfully in this corrosive zone.

Saturated steam pressures from 50 to 100 p.s.i.g. (280° to 327°F.) are used in melting sulphur. The allowable steam temperature depends to some extent on the rate of heat transfer from coil to sulphur. This, in turn, is primarily a function of the amount and type of agitation in the melter. If sufficient agitation is provided to prevent the formation of a film of viscous sulphur on the coils, higher steam temperature can be employed in the melter. Superheated steam is not advised if the steam temperature is above the ignition temperature of the sulphur because of the fire hazard it

SULPHUR MELTING

introduces. It is preferable to use a liberal heat transfer area and lower steam temperatures.

When sulphur is melted in batches, steam pressures are usually reduced after melting has been completed to prevent the sulphur temperature from reaching the viscous range. Steam heat input after melting a batch should be reduced until it just balances radiation and other heat losses.

Temperature control and maintenance work can be facilitated by connecting the coils in parallel rather than in series. This provides equalized steam distribution and condensate drainage.

The steam system should be purged when starting an installation. It should also be well drained and trapped. All external lines should be insulated.

Unheated sections of pipe carrying sulphur from the melter should be avoided. A good rule to follow is to jacket or heat any pipe section greater in length than three pipe diameters. All valves and headers should also be heated and well insulated.

MELTERS

There are two basic types of sulphur melters. In the pit or tank type, solid sulphur is melted by receiving heat from liquid sulphur. In the vertical- and inclined-type melters, solid sulphur receives heat direct from a hot metal surface. The liquid drains off and more solid sulphur is continuously exposed to the hot surface. The pit-type melters may incorporate settling sections within themselves or be used directly with filtration systems. The vertical and inclined melters characteristically drain into settling pits located below or next to the melting unit.

Some pit- or tank-type melters are Freeport, Freeport-Berk, Monsanto, Simon-Carves, Titlestad, Chemico, and the classic unagitated pit melter.

The vertical and inclined types are represented by the Chemipulp, Hamilton, P.C.F., and Tegul melters.

AGITATED PIT-TYPE MELTERS

The Freeport Melter

The basic Freeport melter consists of an agitated liquid pool and employs continuous feeding of the solid sulphur. The unit is composed of a steel, brick, or concrete tank that contains a highly agitated pool of liquid sulphur heated by a series of submerged steam coils. The original Freeport melter design was rectangular, but through the cooperative efforts of Freeport and F. W. Berk

Company a circular design has been developed. This configuration, plus several other incorporated design changes, is referred to as the Freeport-Berk melter, which is described later.

Design and unit size of the basic melter are flexible. Experience has shown that the required heating surface ranges from 1 to 2 square feet per ton-day, depending upon the degree of agitation used and the amount of moisture present in the sulphur. In one large installation the unit consists of a sulphur pool approximately 5 feet in depth in a reinforced, Lumnite concrete pit, 15 feet square by 10 feet deep. This unit has a heating surface of 1,348 square feet and, with proper agitation, the melting capacity is in the order of 30 tons per hour of sulphur containing 2.5% moisture.

Agitation increases the rate of heat transfer, minimizes foam, and speeds dispersion of the solid sulphur particles as they are fed into the molten bath. There are no positive reliable data from which exact agitation design for sulphur melters can be made, although some pertinent information has been collected to give a general direction to be followed. In most cases, there is a tendency to under-design the agitator; this means that its true effectiveness and advantages are not used.

One installation from which data on power for agitation are available consists of a pool of sulphur approximately 40 inches deep in a rectangular steel tank 5 feet long by 3 feet wide by 6 feet deep. The sulphur is heated indirectly by steam flowing through three submerged coils of 4-inch standard pipe having a total heating surface of 39.4 square feet. This unit uses a vertical shaft turbine with eight 12-inch blades mounted at 45° angles. It consumes about 4 h.p. of energy when operated at 400 r.p.m. Following is a table presenting a comparison of power requirements for this unit when operating under various conditions.

Freeport Melter Power Consumption Data

	<i>Speed r.p.m.</i>	<i>Liquid head, in.</i>	<i>Current drawn, amps.</i>	<i>Power, h.p.</i>
Motor only, no load	1730	—	4.4	1.58
Motor and turbine agitator, no load	400	—	4.5	1.62
Motor and turbine agitator in water (80°F.)	400	34	7.3	2.62
Motor and turbine agitator in sulphur (265°F.)	400	34	11.3	4.06
Motor and turbine agitator in sulphur (266°F.)	144	34	4.5	1.62

SULPHUR MELTING

The surface and subsurface agitation provided by this system increased the over-all heat transfer coefficient from about 20 B.t.u. per hour per square foot per °F. for an unagitated bath to 80 B.t.u. per hour per square foot per °F. for an agitated bath.

Negligible foaming has been encountered in melting sulphur that contains average amounts of moisture (0.3% to 0.5%). Bright sulphur with about 4% moisture has developed maximum foam depths of about 22 inches. Foaming is not a limiting factor in this type of equipment if proper agitation is employed.

The surface agitation supplied by the turbine mixer adequately disperses all solid sulphur charged to the pool. Formation of crust or islands does not occur. It is important to introduce the sulphur near the center of the pit and have the liquid flow pattern directed downward from the point of sulphur introduction.

The large installation described previously provides agitation of the liquid sulphur pool by two impellers mounted on a common vertical drive shaft. The shaft is located in the center of the pit and rotates at 63 r.p.m. One large impeller is 5 feet in diameter and consists of ten flat blades set at angles of 45°. It is located 8 inches above the bottom of the pit. The other impeller is a three-bladed propeller-type unit, 30 inches in diameter. It is located about 1 foot below the minimum sulphur level or approximately 4 feet above the bottom of the pit. The large impeller carries the major part of the load. It rotates in such a direction that the sulphur flow is down through the center at the point of feed, across the bottom of the pit, and up through the coils around the sides. The top impeller supplies additional surface agitation and also forms a vortex which tends to pull the solid, wet, feed sulphur beneath the surface. A total of approximately 38 h.p. of agitation is provided by this system with both impellers submerged in the pool.

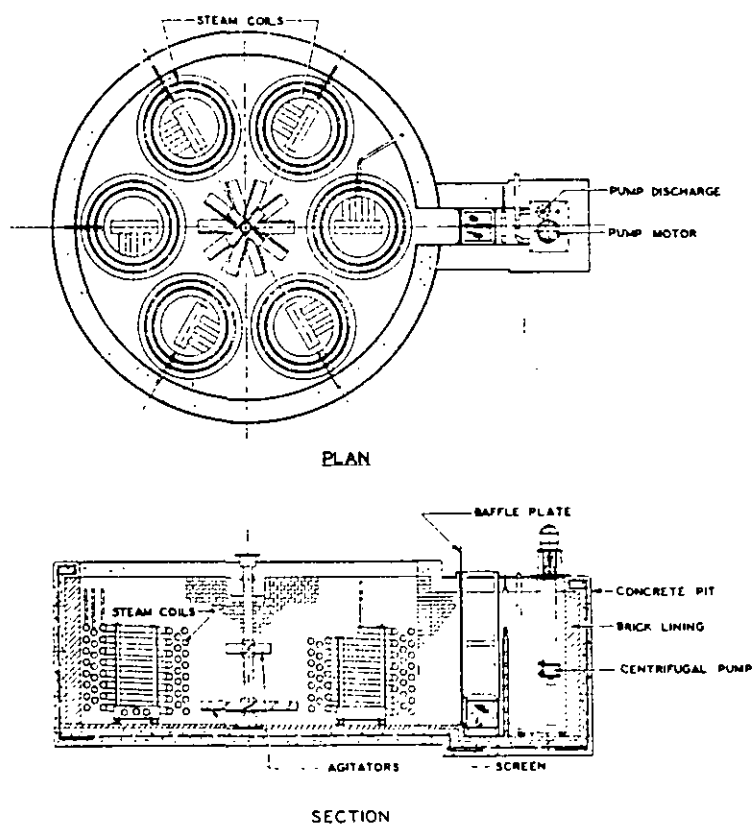
The melting tank and agitator may be fabricated of steel in small installations. Concrete pits with common brick linings are used in larger units. The steam coils are made of extra-heavy black pipe.

Freeport-Berk Melter

The Freeport-Berk melter is basically the same as the Freeport high-rate sulphur melter just described. However, it incorporates certain beneficial design changes which were developed cooperatively by Freeport and the F. W. Berk Company.

It consists of a circular melting pit that not only eliminates the possibility of dead spots where heavy contaminants might accumulate, but also offers simplified construction and thus lower installation costs. Several of these melters have been built for specific installations in the last two years.

To erect this type of melter fast, first pour a concrete floor and base for the melter in an appropriate excavation. On this base lay an inner brick lining of the final melter installation. Lay the lining in the same manner as any brick wall. Then pour the vertical concrete wall behind the brickwork to provide strength for the main structure. The brick wall acts as one side of the form. Make the outside form for casting the concrete wall from plywood. Hold the plywood in the proper circular shape and diameter with a simple bracing of light steel bands or wire. This is a slip wall type of construction. Then pour a layer of concrete to form a lip over the top of the brickwork so that the brick will not float when the pit is filled with liquid sulphur. It is a simple matter to remove the wooden form after the concrete has set. Fill the excavation behind the concrete with earth and grade it.

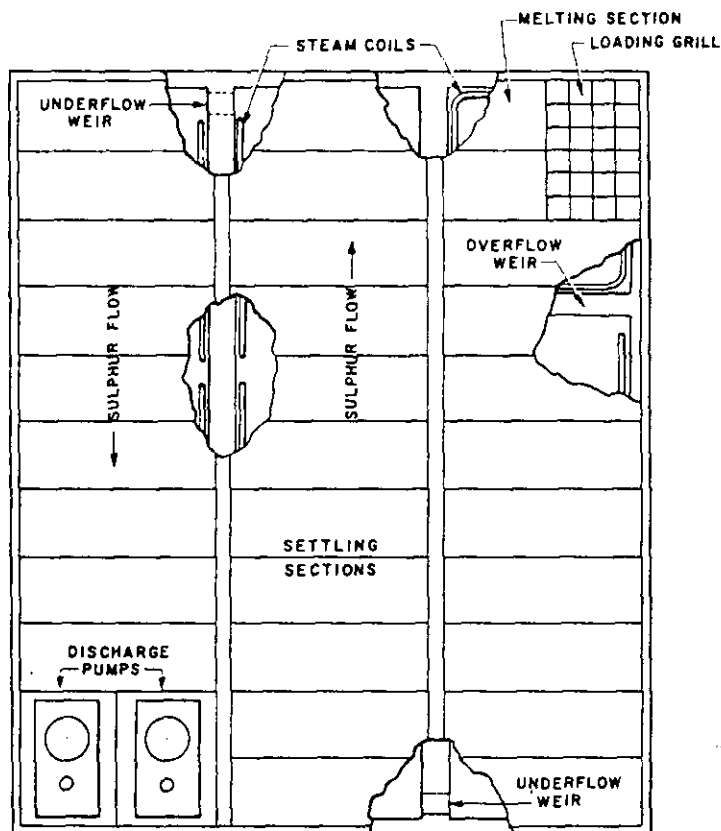


A new development has been made in the steam coil design. In the older Freeport-type units, a straight, multiple U-bend coil construction was used. In the newer melters the coils are formed by bending the pipe in a circular spiral. The spiral pipe coils were selected because they are economical to fabricate, can be divided into units that are self-supporting, and are light in weight. They can be easily removed from the pit for repair or replacement. Since the coils are formed by machine, there is a minimum of welding required. The particular unit shown in the illustration consists of three honeycombed concentric coils, and uses a common steam header and condensate drain system. The coil banks are usually designed and installed so that in case of failure of any one unit, it is a simple matter to remove the faulty unit without shutting down the entire melting system.

Agitation in this circular design is provided in much the same manner as in the original Freeport-type melter. The agitator employs a single vertical shaft with a large impeller at the bottom. Above this main impeller, but below the normal sulphur level, is an impeller of smaller diameter. This auxiliary impeller helps form a vortex near the surface that tends to bring the solid feed immediately below the liquid level. In designing the agitator, it is preferred that no bottom or foot bearing be used. This minimizes troublesome maintenance that would occur with a sleeve-type bearing that contains foreign solid contaminants. The preferred design is a vertically mounted motor and speed reducer with the necessary shaft-supporting bearings all mounted above the center of the pit on suitable channel supports. When the agitator is unsupported by a foot bearing, the top bearing assemblies must be substantially stronger. This is necessary because the top bearings must take all of the thrust. The top bearing support results in higher installation costs but normally yields much lower over-all maintenance costs. If periodic shutdown of the melter to repair the bottom bearing is not a limiting factor, it might prove advantageous to install a bottom bearing which would permit the use of lighter top bearing assemblies.

The heat transfer areas required for circular melters of this class are similar to the Freeport-type melter. For design purposes, it can be assumed that the over-all heat transfer rate from the steam to the liquid sulphur bath would be approximately 70 B.t.u. per hour per square foot per °F. temperature difference between the steam and the liquid sulphur bath.

For further information on this type of efficient sulphur melter, contact the Technical Service Department of the Freeport Sulphur Company.



Monsanto Melter Courtesy of Industrial and Engineering Chemistry.

Monsanto Design Melters

Most Monsanto melters consist of pits subdivided into melting, settling, and pump box sections. Sulphur is charged into a small melting section in which a motor-driven agitator mixes the solid with the liquid and facilitates melting. Molten sulphur overflows from the melting compartment into a rectangular settling pit, subdivided into several compartments by baffles. This permits more efficient settling and skimming of floating debris.

The melter capacity is determined by the requirements of the particular installation. Usually, a heating surface of $3\frac{1}{2}$ square feet per short ton of sulphur per 24 hours is provided when using 2-inch, Schedule 80, steel pipe steam coils in the melting compartment. This design is based upon mechanical agitation and is claimed to

include allowance for submergence of some of the coils in the dirt which may accumulate in the bottom of the pit.

In virtually all instances, Monsanto melters consist of a reinforced concrete pit, partly or wholly lined with 4-inch common red brick laid in acid-resisting cement. The lining is keyed into the concrete. Wholly unlined concrete pits have been found unsatisfactory because of the short life of the pit and contamination of the sulphur by particles of concrete. The entire pit is usually provided with removable steel or aluminum covers to exclude dust and reduce heat losses.

At the feed point, a steel grid with 6-inch-square openings is installed over one end of the melting compartment as a safety device and to screen out large lumps of tramp material that would damage the agitator. A small hopper can be built around the grid to permit accumulation of a sulphur pile above the grid and to provide additional free space to prevent foaming over. As the sulphur melts under the grid, the pile gradually sinks into the melter. An overhead hopper containing a 16- to 24-hour supply of sulphur can be installed with continuous or intermittent, gravity or mechanical feed.

The settling sections of the melting pit are normally sized to provide 72 hours retention time. If the settling sections are properly operated at this rate, 90% to 99% of the ash will be removed. It, of course, depends upon the particle size and density of the ash. Enough coils are installed in the settling sections to offset heat losses and thus keep the sulphur molten. These coils are placed along the external walls only. They are kept low in height, so they will remain submerged even though the normal operating level is not maintained. They are also installed so that they are removable. When sulphur filters are employed, the settling compartments are no longer needed for settling but are used as filtered sulphur storage tanks.

Agitation is confined to the melting section. Usually, the mixing is accomplished with a motor-driven mixer. Some companies use air or steam, but the operating cost of these may be substantial. Steam agitation is also likely to cause corrosion of the melting coils and overhead structures. Agitation, as practiced by Monsanto, is stated to reduce the necessary steam-coil surface by five-sixths.

Steam pressures used for melting are not usually in excess of 100 p.s.i.g. and superheated steam is avoided. Pressures are controlled manually to regulate the temperature of the sulphur in the pit, which is usually held at about 270°F. Steam pressure in jacketed sulphur pipe following the melter should be lower than melting steam to avoid overheating of the sulphur between pit and burner.

An external heating surface of $3\frac{1}{2}$ square feet per 2,000 pounds per day is used in the melter section. Steam consumption in melter and settler combined is about 160 to 230 pounds of 70 p.s.i.g. steam per 2,000 pounds melted, depending upon the amount of moisture present, atmospheric conditions, heat losses, etc.

An empty melter and settler with a capacity that provides a 72-hour retention time requires about 2 days to be completely filled. This required time is a function of the coil surface provided in the melter and of the capacity of the steam system. In case of a shut-down, no special precautions are necessary. The agitator is stopped as soon as all the solid sulphur either in or over the melting compartment is melted so as to save power. Steam should be kept in the coils but should be reduced in pressure to control the temperature if no melting is taking place. In case of a steam failure, the sulphur will remain molten, except for a crust on top, for at least 24 hours. If a long shutdown without steam is contemplated, it is desirable to empty both melter and settler.

The melting compartment during the melting operation should always be kept full to protect the coils from corrosion. Steam coil corrosion is increased whenever sulphur becomes wet and develops acid in storage. Corrosion of melter covers is more serious when melters are located outdoors than indoors. Such corrosion is caused by free acid in the sulphur and by the action of rain water on sulphur that has settled out or has been spilled on the cover. Condensation on the under surface of the covers may add to the corrosion, and steam-jacketed vents should be provided for the escape of moisture. Aluminum covers have been tried with considerable success.

Contamination of the sulphur increases the need for maintenance and cleaning of the pits. If sulphur is clean and dry, corrosion and repairs are slight, and cleaning may not be required for several years. When it becomes necessary to clean a pit, as much of the supernatant molten sulphur as can be reached by the vertical pump should be pumped out, and as much as possible of the remainder should be bailed out. After removing all of the molten sulphur possible, a limited amount of water can be added to the sulphur and dirt mixture while it is still hot. This often gives a "mushy" mixture which can be easily shoveled. The water should only be added in small areas as cleaning progresses. If the sediment cannot be shoveled out in this manner, jack hammers may have to be used to break it out. This sediment is similar to concrete after it solidifies.

Should the sulphur in the pit become ignited, the fire can be smothered with steam by using the 1-inch connection in the center of each pit compartment. A water line with hose connection should

also be provided for extinguishing any accidental fires that steam cannot handle. This includes fires on top of the melter or in the surrounding area.

Monsanto states that the operation of the sulphur melter and settler is simple and requires only a moderate amount of attention. Steady steam pressure and temperature, constant liquid sulphur level, and continuous addition of sulphur are desirable for good operation.

The melter described above is constructed as part of the Leonard-Monsanto contact sulphuric acid plant. The *Monsanto Chemical Company* of St. Louis, Missouri, and the *Leonard Construction Company*, 37 S. Washington Ave., Chicago, Ill., prefer to sell complete acid producing units.

UNAGITATED PIT-TYPE MELTERS

The unagitated pit-type melter is a pit or tank in which sulphur is melted by contact with submerged steam coils and previously melted sulphur. The pit is usually divided into three sections: a melting section, a settling section, and a pumping section. In most installations, the sulphur is charged to the melting section, passes through an underflow opening into the settling section, and over a baffle into the pump section. Periodically, floating debris is skimmed off of the melting section surface.

Melting pits are constructed in a variety of sizes. The size depends not only on the quantity of sulphur used, but also upon whether or not continuous or intermittent feed is employed. The pits are usually constructed of Portland or high-alumina concrete, brick, or steel plate. The steel and concrete pits are often partially or totally lined with common red brick. Brick lining is sometimes used only at the sulphur-air interface. Coils are usually fabricated of black iron pipe. Experience has shown that removable covers should be made of steel or aluminum alloys. This reduces heat losses, lessens contamination, and improves working and safety conditions. Vents should be provided in melter covers to allow the escape of moisture boiled off during the melting operation.

One typical installation melts 40 long tons of sulphur in 8 hours (11,200 pounds per hour) when operated as a batch melter. This unit contains about 650 square feet of heating surface (1,300 feet of Schedule 80, 1½-inch pipe), with 450 square feet in the melting section, 150 square feet in the settling section, and 50 square feet in the pumping section.

The melter described above will melt about 25 pounds of sulphur per hour per square foot of heating surface. It uses 85 p.s.i.g. steam and raises the sulphur temperature to 285°F. The over-all

heat transfer coefficient is about 13 to 14 B.t.u. per hour per square foot of heating surface per °F. temperature difference between the sulphur bath and the steam. Separate steam pressure control valves are provided for each section of the tank because of differing heat requirements. Coils within any one section should be connected in parallel to provide equal heat distribution and ease of maintenance.

Continuous melting has proved more satisfactory than intermittent or batch melting. Intermittent melting results in wide fluctuations in the level of molten sulphur in the pit. This causes increased steam coil corrosion because the coils are alternately exposed to sulphur and air. Although batch melting on one shift per day would reduce labor costs, the same end can be achieved by furnishing an elevated, solid storage bin with a continuous slow rate feeder conveying the sulphur from storage to the melting tank. Sulphur could also be transported from storage to the melting pit by labor using a scoop-type tractor. This should require only a minor amount of the operator's time on each shift.

VERTICAL AND INCLINED MELTERS

Chemipulp Melter

The Chemipulp melter consists of a steam-jacketed tank shaped like a section of an inverted cone. The solid sulphur is fed to a hopper over the melter. It drops into the melter, is melted by direct contact with the heated walls and flows into a collecting trough. The sulphur is then pumped to a sulphur filter or drained into settling tanks, which have capacity for 48 to 72 hours retention time.

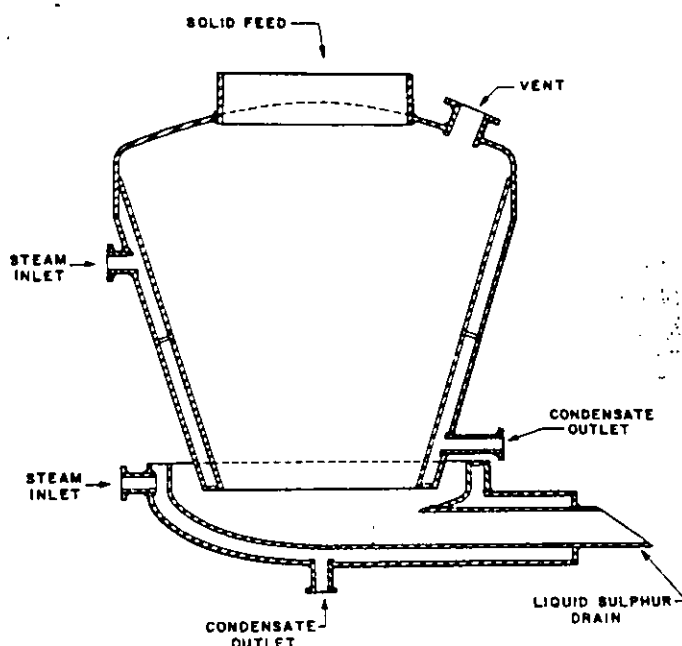
One Chemipulp melter installation consists of a conical section 4 feet high and is reported to melt 1,100 pounds of sulphur per hour with 50 p.s.i.g. steam.

The melter can be built in various sizes. It is fabricated of Type 316 stainless steel-clad mild steel.

The melter has a heating surface of 38.9 square feet. At an operating steam pressure of 50 p.s.i.g. the melting rate is 28.3 pounds of sulphur melted per square foot per hour. Higher rates are possible at increased steam pressures.

The designers state that serious cleaning and maintenance problems do not arise with the conical melter because there are no internal coils.

This melter is available from *Chemipulp Process, Inc.*, Watertown, N. Y., a firm that provides engineering service for design and startup and under certain conditions will procure materials for construction. Although Chemipulp will license the use of its melter for a nominal fee, it prefers to design the melter to fit specific requirements.



Chemipulp Sulphur Melter. Courtesy of Chemipulp Process, Inc

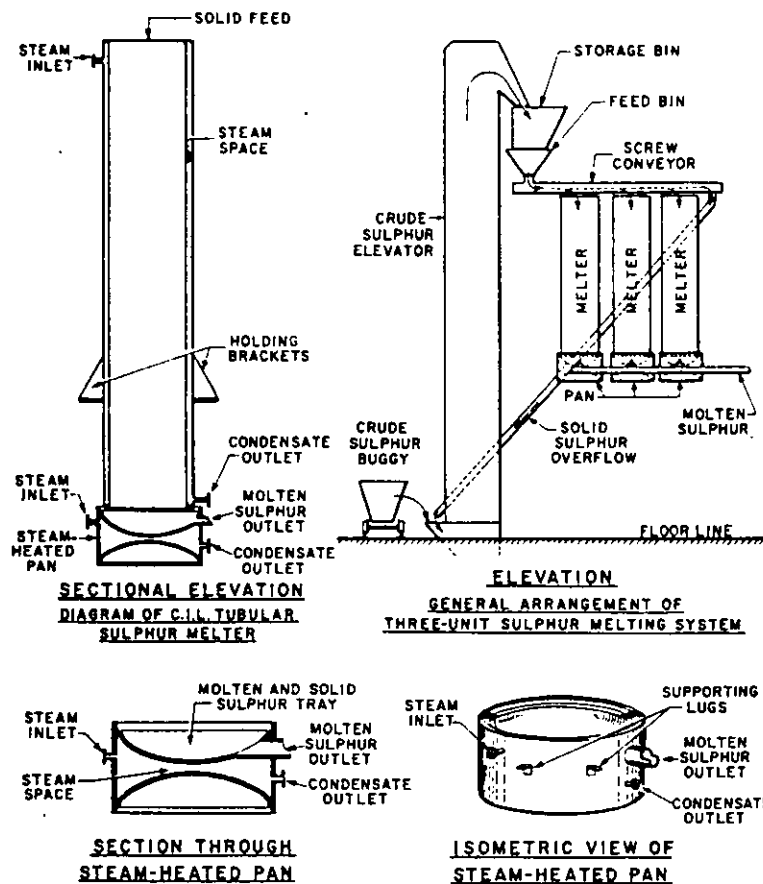
The Hamilton Tubular Melter

The Hamilton tubular sulphur melter consists of two vertical concentric steel pipes with an annular space of about 1 inch between them. The inner pipe is continuously fed with solid sulphur and steam is introduced into the annular section. The solid sulphur is melted by contact with the steam-heated, inner pipe wall. It then flows down the wall into a steam-heated collecting pan. The melted sulphur fills the pan until it overflows. The sulphur is then drained or pumped to a steam-heated settling tank having an average retention capacity for 48- to 72-hour operation.

The original unit consisted of pipes 10½ feet in length, with inner diameters of 20 and 23¾ inches, and wall thicknesses of ⅝ and ⅜ inch, respectively. The dimensions may be varied within reasonable limits.

The capacity of the melter is normally 2,100 pounds per hour. Under optimum conditions up to 2,900 pounds per hour have been melted. The melting rate can be varied by adjusting the steam pressure. The Hamilton melter can be operated singly or in batteries.

The entire melter is fabricated of mild steel and the unit operates on steam pressures from 50 to 100 p.s.i.g. It is claimed that the



C.I.L. (Hamilton) Tubular Sulphur Melter and Battery-Type Installation.
 Courtesy of Pulp and Paper Magazine of Canada.

melter can be brought up to full production in 10 to 15 minutes and shut down in approximately the same time. The melter described has a heating surface of 53 square feet and holds 2,000 pounds of bulk sulphur. It is stated that with 75 p.s.i.g. steam pressure, 10.7 pounds of sulphur are melted per pound of steam. Melting rates of 47.2 pounds of sulphur per square foot of heating surface per hour have been achieved. Its efficiency, as measured in pounds of sulphur melted per pound of steam, is nearly twice that of unagitated melting pits. Its capacity, as measured by production per square foot of heating surface, is nearly three times that of unagitated melting pits.

Repairs other than cleaning or minor replacements are negligible. The pan is the only part of the melter requiring cleaning

except for periodic washing. The cleaning is accomplished by disassembly. Many modifications have been made to these basic units at various installations. One popular modification has been to remove the pan and install an inclined flat or V-shaped bottom so that the melted sulphur drains directly into a separate vessel. The solid contaminants drain with the sulphur and are accumulated in the second vessel which is designed for easier and more frequent cleaning.

The patent covering this melter (U. S. No. 2,149,373) is assigned to *Canadian Industries Ltd.*, Hamilton, Ontario, Canada. Although C.I.L. does not build the melter, the design is available on a nominal royalty basis in the United States and elsewhere. One supplier of this type of unit is the *G. D. Jenssen Company*, P. O. Box 366, Massena, N. Y.

P.C.F. Sulphur Melter

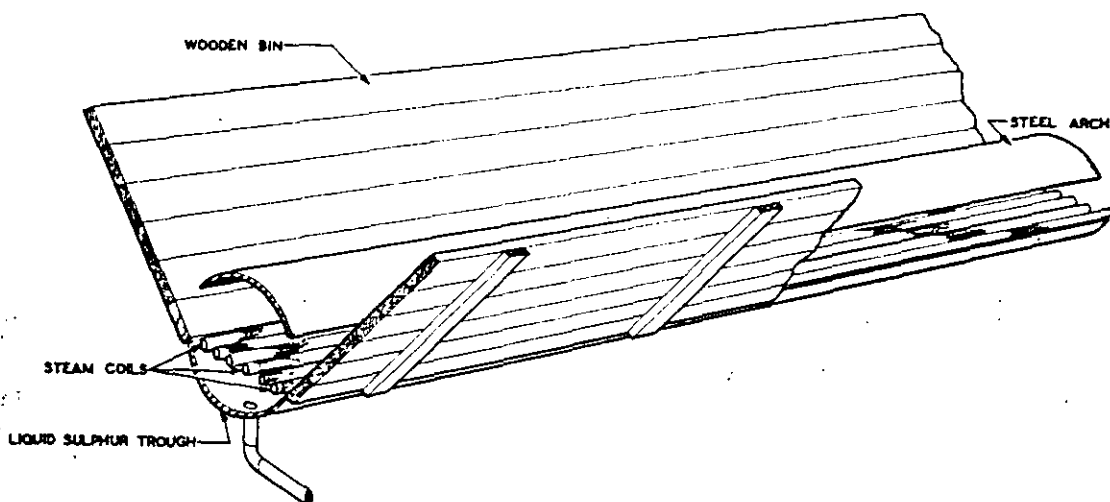
The P.C.F. sulphur melter is of the inclined type. It has been designed to eliminate additional handling of solid sulphur from the stockpile or storage bin to the sulphur melter. The letters P.C.F. are used to identify this unit because it was developed by Mr. Frank Harlow, Assistant Mill Manager of Penobscot Chemical Fibre Company.

It consists of a V-shaped storage bin combined with a heating unit. The original unit was constructed of wood but it can be constructed of other suitable materials. The sides are sloped so that the sulphur will feed to the bottom of the bin. In the bottom there are pipes running the full length and extending outside of the bin at each end. At one end, the pipes are connected to a steam supply header. At the other end, they are connected to a condensate drain header. These pipes form steam coils that melt sulphur in the bottom of the V. It is recommended that the coils be connected separately to the condensate and steam headers so that in case of failure, any one coil may be isolated without shutting down the entire melting system. The coils are usually constructed of wrought iron pipe but may be made of mild steel or higher alloy pipe.

This general type of melter has been tried in the past, but usually the melters were not too successful because extreme corrosion of the coils was experienced. It was found, however, that the corrosion usually resulted from acidic vapors and water released when the sulphur was melted. Vapors could not escape from the sulphur pile and the cold, solid sulphur acted as a reflux condenser that concentrated and held the acidic liquids. In the P.C.F. melter, a system was designed to provide for releasing the moisture and acid vapors. The coils are normally laid very closely together in the

SULPHUR MELTING

bottom of the trough. A fume hood is placed over the coils. The fume hood can be made from half sections of large diameter pipe or from a small, inverted V, wood trough. The size and position of the hood is set to allow for adequate space between the walls of the storage bin and the edges of the hood to permit solid sulphur to feed into the coil section. The fume hood forms a natural vent to conduct the moist vapors to a point outside of the stockpile.



P.C.F. Bin-Melter. Courtesy of Penobscot Chemical Fibre Company.

The sulphur received at the plant is unloaded from the carrier directly into the V-storage bin. The sulphur is melted by heat generated by the steam coils. The desired melting rate can be obtained by adjusting the steam pressure. The liquid sulphur flows by gravity to a settling tank or pump sump. From there, it is pumped into a larger liquid surge tank or directly to location for processing. Excellent results have been realized with this type of melting unit.

Texas Gulf Sulphur Company Melter (Tegul Melter)

The Tegul sulphur melter consists of a steam-heated rectangular shell. The bottom is made of inclined, steam-heated, V-shaped troughs. Additional steam coils are placed inside the tank and directly over the troughs. Sulphur is charged to a hopper over the melter and flows down against the steam-heated sides and coils. It is melted by direct contact with the hot surfaces and flows down the

SULPHUR MELTING

inclined troughs to a collecting pit below. The melted sulphur is then pumped to a filter or to a "48-to-72-hour" settling tank.

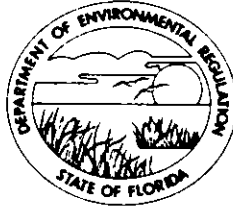
A Tegul melter with 154 square feet of heating surface is reported to melt 6,720 pounds of sulphur per hour using 100 p.s.i.g. steam. This is equivalent to 43.7 pounds per hour per square foot of heating surface. The designers claim that 10 pounds of sulphur can be melted per pound of 100 p.s.i.g. steam. Design size is flexible. The melter is usually fabricated of mild steel and insulated with 3 inches of rock wool. The steam coils are constructed of standard pipe.

Although the melter is designed for use with 100 p.s.i.g. steam, sulphur output can be varied by changing the steam pressure. The designers report wide flexibility of operation, including startup and shutdown times of 10 to 20 minutes. The melter has a bulk sulphur capacity of 5,700 pounds.

The designers claim a high efficiency because the molten sulphur runs off as soon as formed. This insures contact of the solid sulphur with the heating areas. It is also reported that the rapid run-off lessens chances of local overheating and formation of viscous sulphur layers on heat transfer surfaces.

The Tegul melter was designed by the *Texas Gulf Sulphur Company*, 75 E. 45 St., N. Y., N. Y. The firm does not manufacture the melter but will supply further information regarding its design, construction and operation so that it may be constructed in the user's own shop. *Chemipulp Process, Inc.*, Watertown, N. Y., may also be contacted for information concerning this unit.

STATE OF FLORIDA
DEPARTMENT OF ENVIRONMENTAL REGULATION



SOUTHWEST DISTRICT

7601 HIGHWAY 301 NORTH
TAMPA, FLORIDA 33610-9544

BOB GRAHAM
GOVERNOR

VICTORIA J. TSCHINKEL
SECRETARY

WILLIAM K. HENNESSEY
DISTRICT MANAGER

January 19, 1983

DER

JAN 24 1983

BAQM

Mr. H.W. Long, Jr.
Manager Environmental Control
Agrico Chemical Company
Post Office Box 1110
Mulberry, FL 33860

RE: Agrico Big Bend Sulfur Terminal - File No. IC29-55453

Dear Mr. Long:

We have reviewed the supplemental information supplied by you on December 20, 1982 in support of the above referenced permit application. This review finds that the application remains incomplete or otherwise deficient with regard to the following particulars; (these items are numbered to correspond to our initial review of June 2, 1982):

1. The water balance given in Attachment A is based on annual average conditions with the addition of a 25 year storm, which is given as 8.4 inches. This must be either corrected or substantiated. It would be more realistic to consider such a storm imposed on the pond system following an extended period of chronic precipitation, i.e., a condition where the system was already operating at design elevations. Also of interest is the situation where dry meteorological conditions have pertained for a lengthy period. Will sulfur dust be picked up from the ponds by winds under these conditions? Please provide water balances for both extreme circumstances.
- 2,3. The drawings CD201-206 do not provide sufficient detail as to pipe sizes at the various locations in the spray network. Please provide final drawings showing the spray piping as it is to be constructed.
- 4,6. Your cover letter indicates that it is intended to employ a Hypolon pond liner. Please provide a data specification sheet(s) on this material, together with a final design drawing(s). Indicate anticipated service life.
5. Please specify procedures to be used in sediment management, more particularly: What is common industry practice?

Mr. H.W. Long, Jr.
January 19, 1983
Page Two

7. The Law Engineering percolation test result is found insufficient in that there was only one test result and in that it was made at a location outside the proposed pond boundary. We ask that six (6) additional tests be performed in a rectangular pattern inside the boundary.
8. Drawing CD-201 does not indicate a sump in the vicinity of the southwest transfer point. Please explain. Also, provide sump designs.
9. Do "operating companies that handle and ship wet prill sulfur" have data indicating the concentrations of hydrogen sulfide, particulate elemental sulfur, sulfurous acid, sulfuric acid, and sodium sulfate commonly present in the recirculating spray water? Is sodium thiosulfate likely to be present?

What assurance can you provide that procedures practiced in "municipal drinking water treatment plants and sanitary sewer systems" will enable control of the products of microbial action? The only chemical control in the proposed design is sodium hydroxide addition. Are other control measures (besides aeration) contemplated? The application is silent on this point.

10. "The adherence of the fines to the sticky surface of wet prills" needs to be explained and quantified. It seems to be the generally held view that sulfur is not wettable by ordinary water of potable quality. If this is the case, under what conditions can this property (lack of wettability) be modified as a practical matter, or in other words how can the adherence of the fines to the prills be enhanced? How will this property be affected by the proposed pH control measures?
11. Characterize the allotropic forms of solid sulfur with regard to:
 - a. solubility
 - b. wettability
 - c. the generation of fines during handling
 - d. the susceptibility to microbial action.
- 12,13,14. The drawings do not show provisions for control measures to be employed in response to catastrophic rainfall events. Please explain the water management practices to be observed in such occurrences.

Supply information such as size distribution and any other soil properties that would support the high percolation rate reported.

Mr. H.W. Long, Jr.
January 19, 1983
Page Three

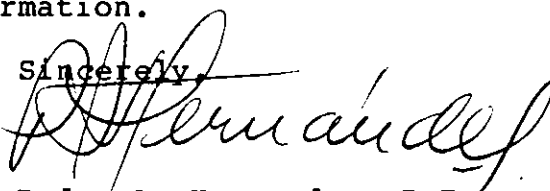
15. Describe the fate of the sodium sulfate and any other compounds that are expected to occur in the recirculating water.
16. It is agreed that the matter of the generator may be appropriately deferred until application for an operating permit.
17. Drawing #CD-203 is considered adequate.
18. It has not been established that sulfur particulates will be adequately contained by the pond system. Our concern was not with vegetation in the ponds, although algae control needs to be addressed, but with the marine environment nearby.
19. Corrosion of structures is of less concern than the anticipated corrosion of pumps, piping, and nozzles. Such corrosion would directly effect the efficiency of dust suppression. Please describe with particularity all provisions for control or abatement.
20. Considered adequate.

Our review of your December 20 response raises the following additional question:

21. Background quality of the ground water needs to be established.

Further processing of your application will be held in abeyance pending receipt of the requested information.

Sincerely,



Pedro A. Hernandez, P.E.
Industrial Waste Section

PAH/rb

cc: HCEPC

Judith S. Kavanaugh, Esquire
Martha M. Hall, Esquire
Steve Smallwood
Paul H. Amundsen, Esquire
Damon C. Glisson, Esquire
Edward P. de la Parte, Jr., Esquire

Larry Lukin - pls handle



January 14, 1983

Ms. Victoria J. Tschinkel, Secretary
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32301

RECEIVED
JAN 17 1983

Office of the Secretary

AND

Mr. L. D. Lukin, P.E., Director
Division of Environmental Programs
Department of Environmental Regulation
Twin Towers Office Building
2600 Blair Stone Road
Tallahassee, Florida 32301

RECEIVED
JAN 18 1983

DIRECTOR - PROGRAMS

Dear Secretary Tschinkel and Mr. Lukin:

RE: AGRICO CHEMICAL COMPANY'S SOUTH PIERCE
PLANT - PRILLED SULFUR HANDLING FACILITY,
POLK COUNTY - AC 53-55780

On November 19, 1982, the Department of Environmental Regulation informed Agrico, by certified letter, of its intent to deny the above-referenced Permit application. That letter stated that the Department's only apparent concern with respect to our application was with the particulate emission factor relied upon by Agrico for wet prill sulfur.

Since that date, Agrico and the Department have agreed to an extension of time during which Agrico will provide additional data concerning sulfur particulate emissions from wet prill. This letter seeks to confirm that the accuracy of the emission factor is the only barrier remaining to issuance of a Letter of Intent to grant our application. Our review of the Technical Evaluation

Secretary Tschinkel & Mr. Lukin
Page Two
January 14, 1983

and Preliminary Determination dated October 29, 1982,
and your letter of November 29, 1982, indicated no other
concerns.

Unless we hear from you to the contrary, therefore, Agrico
will assume that the emission factor is the only remain-
ing issue.

Thank you.

Sincerely,

AGRICO CHEMICAL COMPANY
SOUTH PIERCE CHEMICAL WORKS

A handwritten signature in cursive script that reads "L. C. Lahman".

L. C. Lahman
Plant Manager

LCL:des