

NOTICE

OR CHANGES IN THIS TEXT BE INITIALED
AND DATED, so that those following on
the routing know whom to contact for
further clarification.

DATE:

November 13, 1980

IN TURN:

	DATE RECEIVED	APPROVED INITIALS	DATE
*J. G. Dickinson, C&P, Chestnut Run	11/13/80	JGD	11/2/81
J. G. Delker, C&P, Chestnut Run	1-5-81	JGD	1-5-81
L. W. Thomas, C&P, Chestnut Run	1-2-81	LWT	1-2-81
J. C. Watts, C&P, Chestnut Run	1/5	JCW	1/24/81
J. A. Lamiet, C&P, Chestnut Run	1/23	JAL	2/2/81
H. W. Ling, C&P, Chestnut Run	2/4/81	HWL	2/4/81
E. W. Stewart, C&P, Chestnut Run	2/5/81	ES	2/5/81
W. W. Greer, C&P, B-17274	2/9/81	WWG	2/9/81
J. D. Travis, C&P, B-17272	3/8/81	JDT	3/9/81
T. A. Bartolec, C&P, B-17276	3/10/81	TAB	3/12/81
R. F. Hedgepath, Legal, D-7063	4/10/81	RHC	4/13/81
C. W. Hoeffman, MCD, N-2468-A1	3/17/81	CH	3/17
F. W. Warwick, MCD, N-2537-1	3/17/81	FWW	3/17
W. H. Miller, MCD, N-2537-5			

APPROVAL REQUEST
TECHNICAL LITERATURE

Please approve the attached publication. Any comments and/or changes to this publication must be signed and dated. See note below.

TITLE: Rutile Pigment Slurries (TiO₂ Slurry Booklet)

PURPOSE: Revise, update & replenish stock.

Those whose name follow the asterisk () above certify that the product and performance claims contained in subject material are accurate and that the files of the Chemicals and Pigments Department contain data to support the claims. They further certify that any claims attributed to non-Du Pont sources are consistent with our knowledge and experience.

NOTES: Extra copy for J. G. Dickinson attached.

E-43017

~~TiO₂ Slurry Booklet~~

~~DRAFT~~

~~TiO₂ SLURRY BOOKLET~~

FOREWORD

As pioneers in the development of titanium dioxide slurry technology Du Pont is in a unique position to advise you in the most effective use of this modern distribution and storage method, and the efficiencies, economic^e, and ecological benefits to be derived from it. If, after reading this booklet, you wish to explore the possibilities of conversion to slurry, please contact your local Du Pont representative or our Wilmington office:

de Nemours & Co. (Inc.)
E. I. Du Pont, Company
Chemicals and Pigments Dept. *Department*
Wilmington, DE 19898
Attn: Manager, TiO₂ Products

INTRODUCTION

Commercial titanium dioxide production in the United States began at the end of World War I. For the next fifty years, the standard shipping package was the 50 lb multi-wall paper bag. In the late 1960's, an innovative departure from dry bagged pigment in the form of a high solids anatase TiO_2 slurry was offered to TiO_2 users in the paper industry. Enthusiastic and rapid acceptance prompted development of rutile TiO_2 slurries for the coatings industry. The first of these, designed for trade sales flat paints, was introduced in 1971. Since then, high solids slurries for durable exterior coatings and gloss wall and trim paints ^{have been} introduced. For paper, slurry offerings were broadened to include a specially developed rutile slurry ^(RPS) which offers improved optical performance both in wet end and paper and board coatings. JN

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SLURRY ECONOMICS

Demand for TiO_2 slurry has grown rapidly, exceeding 90,000 tons/yr in 1980. By 1988, U. S. consumption is projected to be 170,000 tons annually. Economics support this ready acceptance and rapid expansion. In most cases, savings accrued from reduced process energy (predispersed product), increased production capability, and improved handling and storage are sufficient to cover the cost of a slurry system when the investment is amortized over a two to five year period.

~~Many TiO_2 consumers can justify slurry usage.~~ Factors such as ^{you don't} actual savings generated, annual consumption, cost of conversion, mode of delivery and freight costs (control) the economics:

Contribute significantly to it

- Paint producers report savings of two to six cents per pound of TiO_2 ; paper mill savings ^{can exceed 40%} may reach ten percent of pigment consumption. Employing a checklist (Figure I) is a convenient way to ^{calculate} ~~tabulate~~ savings.
- No set amount of TiO_2 consumption has been established to justify slurry system installation. Under favorable conditions, as little as 300,000 lbs/yr ^{of a single grade} may be sufficient although twice that amount is usually necessary.
- Slurry system costs are influenced by design complexity, materials of construction, and degree of automation. Costs range from ~~a truckload scale, one grade system feeding one use point~~ for about \$25,000, to a multigrade railcar system for over \$200,000.

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- Mode of delivery can influence both conversion and operating costs. Railcars contain approximately 10,000 to 12,500 gallons (60 to 75 tons TiO_2 , dry basis), depending on grade. The minimum recommended storage tank capacity is one and a half carloads. Smaller tanks than this make it difficult for the customer to schedule deliveries and can lead to ~~demmorage~~^{detention} charges. Slurry systems designed for routine servicing by truck permit smaller storage, and recommended capacity is one and one half truck loads. Systems with under 5,000 gallons storage capacity are in service. Because less investment is required, the cost of this size system is within reach of many smaller TiO_2 consumers.

Is this needed?
Keep information general. Detail handled by sub. #.
If we go freight cost too high to cut 1 day.
300

~~While equipment and installation costs may favor truck receipt, the cost of slurry delivery to the plant may more than offset the reduced investment. This can occur if freight charges between shipping point and destination exceeds the amount prepaid and allowed.~~

SLURRY PROPERTIES

Slurry properties are carefully controlled in order to facilitate manufacture, shipment, storage and handling:

- Slurry rheology is maintained newtonian at least in the shear range of common pumps. At the same time, slight thixotropy is imparted at low shear to retard settling. These properties are not expected to change with age. Proper rheology is obtained without elaborate dispersant combinations or high dispersant levels.

- Solids level is kept high to allow use in high solids paint and coatings formulas and to avoid shipping large quantities of water. Currently marketed grades range from 62% to 76% solids.
- Slurry density is kept constant to insure accurate metering with commonly used flow meters.
- Slurry as shipped is sterile as are rail cars and trucks.

Since

→ Bacteria can live in slurry, biostats are included in the recipe.

Upon receipt, certain precautions must be taken by the customer to insure satisfactory storage and use.

- Settling can be expected from any practical slurry. If the cake is soft, easily reincorporated and forms slowly, ^{this} settling can be tolerated. Proper agitation, designed into the storage facilities is a necessity.
- Slurries are formulated at high solids; consequently, evaporation of even small amounts of water results in formulation of dried flakes on slurry wetted container walls. **I**t is therefore necessary to keep tank humidity at or near saturation by periodically injecting steam or atomized water.

Handwritten notes:
"This can be" (circled)
"1900"
"roughly?"

Slurry properties are strongly interrelated. The desire to achieve maximum percent solids and simple formulas with low dispersant levels is limited ^{principally} by dilatancy and increased tendency to form dry flakes. Formulation problems occur with higher levels of dispersant in slurry. Thixotropic slurries resist settling but do tend to thicken on aging and entrap air which causes density variations. An acceptable slurry is a careful balance of all properties.

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STORAGE AND HANDLING SYSTEMS

Design

Du Pont offers consulting and design assistance for installation and use of TiO_2 slurry facilities. Once the needs of a TiO_2 user in a given application are established, the design of facilities to use slurry is relatively straight-forward, but recognition of slurries' unique characteristics is essential. Extensive experience in the manufacture, distribution and use of TiO_2 enable us to recommend economical routes to using slurry effectively. Our design staff can prepare complete plans and estimates for a slurry system, and assist in procurement and installation of the equipment.

Since needs of the user vary widely we ~~have not prepared package systems,~~ preferring to ^{custom} design installations, ~~to suit each individual case.~~ We have designed ^a ~~one~~ package system for low volume cases (truck receipt) which can be installed in 3 to 5 months. Timing in these cases may take 7 to 10 months from project authorization to start-up.

In a well designed system, slurry can be pumped and stored as easily as other bulk fluids. Conversely, inadequate design can lead to problems such as pigment settling resulting in tank heels and plugged lines and valves.

A typical slurry ^{installation} ~~system~~ consists of an unloading pump station, storage tank, and distribution ^{network} ~~system~~ (Figure II). A ^{facility} ~~system~~ for two grades of slurry will of course need two storage tanks and two distribution systems, but one unloading ^{station} ~~system~~ can serve both grades.

The major cost of the system is the agitated storage tank. Piping becomes a large cost consideration for long or complex runs. Instrumentation can be relatively simple or can become a large cost item when many use points or extensive automation is involved.

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Unloading Station

The rail car unloading station (Figure III) consists of a pump, piping, and water and compressed air service. The pump is used both to transfer slurry to the storage tank and to recirculate and wash out car heels with a charge of water. Compressed air is used to clear the lines before and after unloading and to pressurize the car for more efficient unloading. Unloading can often be done with pressure alone (limited to 100 psig) but effective heel removal requires the pump for recirculation. (See Unloading ~~Procedures~~ ^{Practices}.)

The system is designed to unload at 150 gpm or more so that a 10,000+ gallon car can be emptied in about an hour. (See Pumps.)

Truck unloading stations can be simpler since trucks do not require recirculation and are usually unloaded with air pressure. The lift and run however is limited by the pressure rating of the tank truck, usually 25 psig. Most trucks are equipped with their own power-take-off air pumps.

Storage Tanks

A typical TiO_2 slurry storage tank arrangement is shown in Figure IV. Tanks are typically flat bottom cylinders and hold ^{a minimum of} about 1 1/2 carloads (or truckloads) or ~~more~~ ^{as} usage and inventory needs dictate.

Tanks may be made of ^{fiberglass reinforced polyester} (FRP), epoxy coated steel, concrete-tile, or stainless steel. Cost will be about \$1 to \$2 per gallon of capacity. Under 10,000 gal, FRP will generally be most economical, in the 20,000 gal range, epoxy coated steel is generally lowest cost, and in larger sizes concrete-tile is the economic choice. Stainless steel is an ideal material, but is usually more costly. For interior locations FRP tank size will be limited by building access openings, but steel or concrete-tile tanks can be erected in place.

Since slurry is ^{loaded} ~~delivered~~ warm and has a high heat capacity, there is little risk of its freezing if tanks are insulated except during long periods of severe cold weather. Normal intermittent agitation keeps the slurry mixed so that localized freezing will not occur, and continued agitation can provide enough heat input to keep the slurry fluid. Our rail cars are insulated and can deliver slurry in the coldest weather.

Agitation

TiO₂ slurry storage tanks must have agitation to prevent settling and maintain product uniformity. Slurry at full strength, or diluted to some constant solids level a percentage point or two lower, settles very slowly. Sediment formed in several days is reincorporated easily with agitation, but if left standing for several weeks can form heavy cakes that may resist reincorporation. Moderate agitation at about 1 hp per 1000 gallons is satisfactory, using turbine blades and wall baffles in the usual cylindrical flat bottom tanks. The agitator will cost up to half as much as the tank itself. Recirculation of tank contents with a pump is not adequate to keep the slurry suspended for long periods but can be used to reduce settling during periods of agitator outage. We recommend a timer on the agitator for periodic operation (15 minutes every two hours), to conserve power and avoid slurry heating.

Humidification

It is essential to humidify storage tanks to prevent drying of slurry on the walls. High solids content of TiO₂ slurry leaves little spare water and makes it quite vulnerable to drying and caking if tank humidity is not close to 100%. Dried TiO₂ wall scale is extremely hard and is likely to show up as grit in coatings. With adequate humidification the walls will remain wet and not show TiO₂ buildup as level changes and splashing coat the walls with a film of slurry.

Humidification is simply achieved by providing water fog spray in the tank periodically. The fog can be produced by a pressure water spray, air atomized water spray, or steam jet. Fog nozzles are sized to deliver three to five gallons of water a day when operated a few minutes per hour; this will not be enough water to materially affect slurry solids content.

Distribution Systems

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The simplest system delivers selected amounts of slurry to a batch tank. This can be accomplished with a pump at the storage tank, a transfer pipe, and a batch meter at the use point. The pipeline from storage to the meter may be left full of slurry between batches but it is desirable to provide a water flush through the meter after each batch delivery (Figure V). Normal intermittent use will avoid settling in the pipeline.

If widely separated use points are to be served, it is often advisable to use a header line past the use points looped back to the storage tank and maintained under pressure by a control valve at the tank return. Individual take-offs can be metered at each location *or with a main line meter as in Figure II.* (Figure VI). *LWT*

When more than one grade of slurry is used, it is advisable to duplicate the facilities up to the use point or a branching at adjoining use points. (Figure II.) It is generally unsatisfactory to use one supply system flushed between changes of grade.

In some applications, it is desired to deliver slurry to a continuous use point at a low TiO_2 rate that would be difficult to meter and control accurately (TiO_2 feed to the wet end of a paper machine). In this case, slurry is diluted to some low concentration (1 lb/gal) and then metered to the use point at a manageable flow rate. A typical dilution/feed system is shown in (Figure VI). Here slurry dilution is controlled by density measurement in a water/slurry mixing column which overflows to a holding tank from which it is circulated through a loop line to the use points. *ch* *Figure VI*

Since diluted slurry settles rapidly, it is necessary to keep it agitated continuously and moving constantly in the pipelines. In a dilute system, no dead pipe areas can be tolerated.

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Pumps

Either centrifugal pumps with open impellers, or air diaphragm pumps are preferred for TiO_2 slurry service.

Centrifugal pumps are generally chosen for higher pressure duty (above 80 psig) and/or continuous operation such as for a loop system; or where suitable air supply is not available for air diaphragm pump operation. Stainless steel is the preferred material of construction.

Diaphragm pumps are chosen for lower pressure duty and for intermittent operation such as car unloading or direct batching. The air diaphragm is excellent for moving slurry and is simpler to install and monitor than a centrifugal and costs much less. Aluminum is acceptable for slurry service in an air pump. It will eventually become eroded but will give long service and is economical to replace (cost is about $1/3$ that of a stainless unit or a centrifugal pump). For metered delivery a pulsation damper at the pump discharge is advisable. For the free flow of car unloading, however, the air pump pulsations are acceptable if piping is adequately supported.

Piping and Valves

Either FRP or stainless steel is commonly used for slurry piping. Choice between the two is often a case of customer preference. FRP pipe gives good service and costs less both to purchase and install than stainless, particularly where field welding may be hazardous. Unreinforced plastic pipe can be used in some cases. It is necessary to keep line velocity at about 5 ft/second in order to ^{OVERCOME} ~~avoid~~ settling in pipes. Minimum diameters recommended are $1\frac{1}{2}$ to 2 inches. Slurry piping should be run in as direct a routing as practical and the system should drain to one end or the other but this is not essential if the line can be blown clear. X
Lent

Valve selection is limited to ball, butterfly, and diaphragm types. Other valves will wear, plug, or bind, in slurry service. Ball valves are generally used for the best all around service, but particularly for frequent manual on/off operation and where complete shutoff is necessary. Butterfly valves are an economical alternative in less demanding locations such as in the unloading lines. For automatic control or regulation, diaphragm valves are preferred.

Instrumentation

Tank level indicators are a matter of individual choice. In some cases visual indication through the translucent wall of an FRP tank is acceptable. Local indication of level in opaque tanks may be obtained from a diaphragm pressure gauge installed near the tank bottom. If remote indication is desired, a differential pressure cell at the tank bottom may be used. Physical measurement with internal elements ^{is} ~~are~~ undesirable because any structure in the tank is subject to high stress from the agitated TiO_2 slurry flow, and pigment will tend to block or plug confined floats, etc.

We have found the magnetic flow meter to give the best all around service in slurry metering applications. It can reliably measure continuous or batch deliveries of full strength or diluted slurries over a wide range. It does not require flushing after use, but rather is best left full of process fluid at all times.

A lower cost alternative in the case of a batching duty where the meter may be flushed after use, is an oscillating piston meter. Although bushings require periodic replacement because of slurry abrasion, it is a simple job.

An effective pumping-metering arrangement can be made using the positive displacement feature of the air diaphragm pump. Control systems are available to count the pulses of the pump and convert them into volume readings which are then displayed and used to operate valves, pumps, etc. Cost is relatively low and reliability good since the pump itself is the metering element.

Low Cost Slurry System

One exception to the general practice of custom designing each slurry system is our low cost slurry system (LCSS). This is a 5,000 gallon system for small volume users designed to ~~replace the slurry "cube"~~. *begs the question? what is a cube? JAO* It will receive truck shipments and deliver metered batches of slurry to a single use point.

The LCSS system (Figure VII) is based on a conical bottom tank design without an agitator. Slurry suspension and mixing is achieved by periodic recirculation through a vertical internal header pipe. An air diaphragm pump provides recirculation and delivery. Batching is automatic with an oscillating piston meter, and includes automatic water flush. The tank and piping are FRP.

JAO Cost of the LCSS system is low, but will vary with the pipe runs required, inside or outside location, foundation availability, etc.

OPERATIONS

Unloading Practices

(Read Personal Safety Section before handling TiO₂ Slurry)

Railcars are unloaded as described in detail in our instruction manual "Railcar Unloading Procedures for Ti-Pure® TiO₂ Slurry". ~~A detailed piping diagram of a typical unloading station is shown in our drawing W-594621.~~

The basic unloading procedure is to pump the car empty, including a recirculation step to clear the heel, then blow the lines clear with air as follows:

- Clear the car outlet valve with brief air blow-back, *the ^{spurge air} recirculate* *through the recirculation header* for a few minutes. *X*
- Pressurize the car.
- Pump the car empty.
- Add 100 gal or more of water and recirculate for 1/2 hour
- Pump the car empty and blow lines clear. *LWT*



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Cars can be unloaded with air pressure alone but pumping/recirculation is advisable to completely empty the TiO_2 . Using air pressure on the car during pump unloading ~~both~~ speeds up the unloading and promotes more complete draining of slurry from the walls.

Trucks are usually unloaded with air pressure alone and most do not have recirculation headers. Settled pigment is not generally a problem with trucks since they are unloaded within a few days of filling. Most trucks are equipped with their own power-take-off air pump. ^{Air} Aid is used to blow the lines clear after unloading. The truck may be hosed down manually if desired.

Maintenance

Slurry tanks normally require a minimum of maintenance. Concrete-tile, FRP, and SS tanks are essentially maintenance free. The lining of a steel tank should be inspected periodically as it may require repair or replacement in 5 to 10 years; small defects in the lining can be patched with an air-dry epoxy.

Slurry tanks should be inspected frequently to assure that ^{the} humidification system is performing satisfactorily. Wall scale formation ~~on the walls~~ ⁱⁿ inadequately humidified tanks is a major source of quality problems.

Normal maintenance practices are satisfactory for slurry equipment, but the caking and settling properties of slurry must be considered during system shut down. A full strength slurry line may be left full for about three days if it is not open to atmosphere. However, a dilute slurry line should be flushed with water for any shutdown over 30 minutes.

Hygiene

A clean system is essential to prevent bacterial contamination of the slurry. Slurry is delivered in sterilized cars. Although TiO_2 pigment is inert, the dispersants are susceptible. Contaminated slurry may lose its dispersion stability and can pass bacteria along to the rest of the process.

We recommend sterilization of new systems by filling them with water containing 400 PPM of hypochlorite (such as HTH swimming pool formula). After the wash solution is drained, it is important to flush the system with water because small amounts of the chemical _q will severely flocculate pigment.

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PERSONAL SAFETY

Health Hazards

IF titanium dioxide slurries are allowed to dry down, dusts may form.

No. 17

In Du Pont tests, dusts of titanium dioxide produced a response in the lungs of rats comparable to the effects given by inert or nuisance dusts.

Titanium dioxide is regulated as an air contaminant by the U. S. Department of Labor (OSHA) under Title 29 CFR 1910.1000. OSHA has ruled that an employee's exposure to titanium dioxide in any eight-hour work shift of a forty-hour week shall not exceed a Time-Weighted Average of 15 mg/m³ in air.*

The American Conference of Governmental Industrial Hygienists (ACGIH) classifies titanium dioxide as a nuisance dust and recommends a Threshold Limit Value, TLV®, of 10 mg/m³ total dust or 5 mg/m³ respirable dust. Du Pont has elected to observe the ACGIH limit.

*Due to changing recommendations and governmental regulations, such as those of the Department of Labor, U. S. Environmental Protection Agency, and the Food and Drug Administration, references herein to governmental requirements may be superseded. You should consult and follow the current governmental regulations, such as Hazard Classification, Labeling, Food Use Clearances, Worker Exposure Limitations, and Waste Disposal Procedures for the up-to-date requirements.

*See next page.
JW.*

Insert A

PERSONAL SAFETY

If titanium dioxide slurries are allowed to dry down, dusts may form. Under some circumstances, exposure to these dusts are subject to regulation or control recommendation. Before using Du Pont LiPure[®] titanium dioxide slurries, review the Data Sheets and Material Safety Data Sheets for the appropriate LiPure[®] grades.

I changed the Personal Safety section to read as above since we do not have one write up which is applicable to all grades. This is because grades having $>5\%$ silica is treated differently from those with less silica.

HWatts
1/22/81