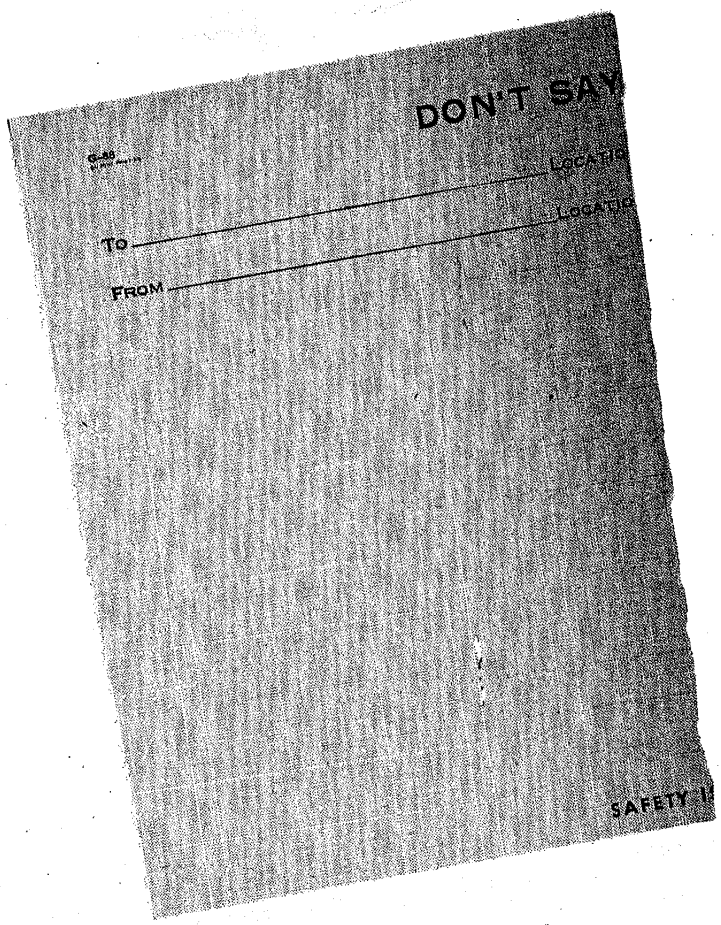
DON'T SAY
TO
FROM

F. H. BEADLES	- WILMINGTON	N. I. DRESS	- PHILADELPHIA
E. E. FURST	- WILMINGTON	R. E. DURFEE	- EVERETT
C. F. KALB	- WILMINGTON	D. L. HERNDON	- MARSHALL LAB.
E. T. BREUER	- WILMINGTON	R. J. KNAKE	- FORT MADISON
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E. I. DU PONT DE NEMOURS & CO., INC.

FABRICS AND FINISHES DEPARTMENT

FINISHES DIVISION

PARLIN PLANT LABORATORY REPORT NO. 29

FURFURYL ALCOHOL DETERGENTS FOR CLEANING CONTAINERS AND EQUIPMENT USED IN THE MANUFACTURE OF ACRYLIC ENAMELS

DATE ISSUED: MAY 15, 1961

PROJECT NO: JL-1014

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PARLIN PLANT LABORATORY REPORT NO. 29

SUBJECT: FURFURYL ALCOHOL DETERGENTS FOR CLEANING CONTAINERS AND EQUIPMENT USED IN THE MANUFACTURE OF ACRYLIC ENAMELS

INTRODUCTION:

Because of the Du Pont Company policy of extended use of reconditioned drums and the rapid demands for acrylic type finishes, the need for an effective cleaner for equipment and containers is apparent.

During the past two years, the cost of a reconditioned drum has increased from $1.15 to $3.15. The majority of this increase was attributed to the difficulty experienced by the reconditioner in cleaning Automotive "Lucite" drums. Experience to date has shown the quality of the reconditioner to be poor as evidenced by several Automotive complaints on contamination being a result of dirty reconditioned drums. It was believed that with a more efficient cleaning system the reconditioning cost might be substantially reduced and the quality of the drums improved.

Of equal importance is the need for a fast and efficient cleaning of equipment to meet production requirements and prevent contamination. Consideration must also be given to the cleaning of application equipment. For cleaning of this type, it would be desirable to have high flash point material as an added safety precaution.

OBJECTIVE:

1. To evaluate existing solvents, detergents and strippers as a cleaner for acrylis finishes currently used in Automotive, metal strip coating and wire enamel industries.

2. Investigate the different cleaning processes used in the trade.

3. Conduct practical tests of most promising candidates designed to simulate drum reconditioning systems.

SUMMARY AND CONCLUSIONS:

Drum Reconditioning

(1) Laboratory tests substantiated by practical tests conducted at the Bayonne Barrel and Drum Co., show that the best all-around cleaner for reconditioning "Lucite" drums is a composition consisting of 40% Furfuryl Alcohol, 10% Tri-Sodium Phosphate and 50% city water. The outstanding merits of this solution are:

1. Excellent solvent for "Lucite" which can be diluted with water and still retain sufficient solvent action.

2. Low toxicity which with proper handling presents no unusual hazards.

3. The relatively high open flash point of 168°F. lends itself to industrial handling.

The disadvantages to the use of Furfuryl Alcohol are:

1. The chief objection is the cost of approximately 93¢/gal. Compared to the straight caustic solution normally used by conditioners, this represents a substantial increase of 87¢ per drum. On the other hand, the furfuryl cleaner is cheaper by far than any of the stripper-type detergents offered by the general trade which range in price from $2.50-$5.00 per gallon.

2. Furfuryl Alcohol reacts with strong acids so that precautions must be taken to avoid contact of such materials with the alcohol.

Equipment Cleaner

Favorable laboratory results have been obtained from tests designed to clean equipment and tote tank cleaning procedures with a solvent blend formulated as follows:

Furfuryl Alcohol	20.0%
Methylene Chloride	20.0
Xylene	60.0
	100.0%

The advantage offered by this composition is that it provides the optimum in general cleaning efficiency with a fairly safe flash point. The composition removes the finish by lifting so that the cleaner can be filtered or allowed to settle and reused. The most efficient method of cleaning the tote tanks is by jet spray.

Its disadvantage is that an increase in methylene chloride content is frequently necessary for different type finishes. This is advisable in order to improve the general efficiency of the cleaner.

DISCUSSION:

In order to obtain some knowledge regarding the effectiveness of the common solvents in removing baked "Lucite" type finishes, a test was devised to evaluate this characteristic. The solvent, together with the results, appear in the following tabulation:

* SOLVENTS WHICH WERE EFFECTIVE IN REMOVING BAKED "LUCITE" FROM STEEL PLATES

Solvent Code	Chemical Name of Solvent or Material	Relative Cleaning Time-Mins.	Cost #/Lb.	Flash Point °F.	Boiling Range °C.	Toxicity
H-203	Methylene Chloride	1	0.168	-	39 - 42	Skin irritant, harmful vapor
H-46	Ethylene Dichloride	5	0.126	59	80 - 87	Skin irritant, harmful vapor
H-30	Acetone	6	0.067	2	34 - 58	Skin irritant, harmful vapor
H-203 / H-47	Methylene Chloride 25% / Toluene 75%	9	0.070		39 - 112	Skin irritant, harmful vapor
H-205	Trichloroethane	10	0.167	-	110 - 115	Harmful vapor
H-46 / H-583	Ethylene Dichloride 30% / Xylene 70%	20	0.070		110 - 115	Skin irritant, harmful vapor
H-203 / H-583	Methylene Chloride 25% / Xylene 75%	26	0.070		39 - 146	Skin irritant, harmful vapor
H-79	Cellosolve Acetate	45	0.156	137	130 - 175	None
H-47	Toluene	55	0.035	52	110 - 112	Harmful vapor, skin irritant
H-6	Butyl Acetate	70	0.121	93	110 - 135	Skin irritant,
-	Carbon Tetrachloride	120	-	-		Harmful vapor
H-39	Diacetone Alcohol	180	0.120	153	0 - 175	None
H-583	Xylene	300	0.041	80	135 - 146	None

*All tests made using metal coated with 885-050, White "Lucite" baked at 350°F. for 1 hour. Lacquer is removed at same speed on primed or unprimed metal plates.

Solvents tested which did not remove baked 885-050, White "Lucite".

H-29	Pet. Nap.	
H-69	Isopropyl Alcohol	
G-697	Phosphoric Acid	
H-30)50% / H-506)50%	Acetone-Water	
H-506	Water	
NaOH)15% / H-506)85%	Sodium Hydroxide-Water	
C_6H_5OH)10% / NaOH)15% / H-506)75%	Sodium Hydroxide-Water-Phenol	

A review of the results show that the most effective solvents for removing "Lucite" are methylene chloride, ethylene chloride and acetone. Because of the expense, toxicity or low flash point, these solvents are unsuitable for production scale drum reconditioning.

Evaluation of Detergents Used by Reconditioners on "Lucite" Drums

Several detergents used by local drum reconditioners were evaluated in the laboratory for general cleaning efficiency. It was found that usually the cleaners represented one of two distinct compositions, that is, they were either the chlorinated solvent or cresylic acid type. The chlorinated type is quite efficient but its use cannot be justified against the cost. The cresylic acid type cleaners did not appear to be very efficient since it was necessary to maintain a temperature of 180°F. in order to obtain any positive cleaning action. Furthermore, cresylic acid possesses a strong pungent odor and is corrosive. It is extremely doubtful if a reconditioner would consider the cresylic acid type cleaners for these reasons.

Furfuryl Alcohol Cleaners

Certain drum reconditioners on the West Coast were reported to be experiencing favorable results by the use of furfuryl alcohol in combination with water and caustic. Acting on this lead, a considerable amount of laboratory work was undertaken in order to completely evaluate the merits of furfuryl alcohol. Laboratory findings showed that the most efficient and economical formulations were essentially as follows:

No. 1		No. 2	
Furfuryl Alcohol	40.0%	Furfuryl Alcohol	40%
Tap Water	55.0	Water	60%
7% Caustic Solution	5.0		100%
	100.0%		

The No. 1 formulation was further tested by actually attempting to clean dirty returned "Lucite" drums. The standard procedure adopted was to place six bronze chains and two gallons of cleaner formulation into a drum. The drum was then placed on a slow drum roller (3 RPM per minute) and rolled approximately 25 minutes. In the initial tests, the drums were examined at 10 minute intervals to observe for gassing, solvent action, etc. For control purposes, a straight 7% caustic solution was used on both regular and metallic "Lucite" drums. Testing of the caustic and metallic had to be discontinued after 1 minute because of extreme gassing which caused the drum to bulge.

Examination of the drum cleaned with the No. 2 furfuryl alcohol cleaner did not reveal any appreciable evidence of real effective cleaning action, although the "Lucite" did appear to be"pot cheesy" in film integrity. The drums were then flushed with water by means of a hose. This action completely removed the "Lucite" and left the drum walls very clean. The straight caustic cleaning solution was found to cause a direct opposite effect in that the "Lucite" became extremely hard and tough and obviously could not be removed by a water wash. This observation proved to be a very important factor in successful reconditioning of "Lucite" drums in that it lead to the replacement of caustic with tri-sodium phosphate for use with furfuryl alcohol. The use of tri-sodium phosphate renders the "Lucite" soft and fluffy so that it can be easily removed by rinsing.

On the basis of laboratory findings, the No. 2 furfuryl alcohol cleaner, bolstered with tri-sodium phosphate, seemed far superior to other types tested to date. Although the cost per gallon would be approximately 90¢, the product possessed many desirable features such as, ease of formulation, practically no hazard from toxicity and a reasonably safe flash point.

In view of the possible results obtained with drums, it was decided to conduct a practical test at the Bayonne Barrel & Drum Company at Newark, N. J. This was done by charging 100-gallons of solution into a 250-gallon detergent tank as follows.

First Portion		
City Water	452 lbs.	Dissolve with heat
Tri-Sodium Phosphate	90 "	110-130°F.
Second Portion		
Furfuryl Alcohol	361 lbs.	Increase temperature to 140-160°F.
	903 lbs.	

The tank is equipped with three revolving nozzles and will accommodate three drums per cycle. The "Lucite" drums are inserted on end so that the nozzle extends into the drum through the end bung opening. The hot cleaning solution is sprayed into the drum for one minute at about 40 lbs. pressure at a temperature of 140-160°F.

Examination of a number pf drums before and after the cleaning operation showed the furfuryl solution to be very effective in removing the "Lucite". In most instances, all of the "Lucite" was removed. Following the jet spray cleaning, the drums were continued through the Bayonne cleaning schedule as follows:

1. Caustic chaining - 40 mins. total - three positions
2. Caustic flushing - 5% solution
3. Water rinsing - drying
4. Inspection

Inspection of the drums showed that in most cases the drum interiors were very bright and that the rejections were largely due to spot contamination or dirty chimes. The spots were usually confined to local areas and appeared to be due to caustic staining resulting from the rinsing operation. The amount of dirt in the chime in most cases was relatively small and situated in an area difficult to remove by a single chaining.

The inspection performed by Bayonne appeared to be very critical, comparable in this respect to that used by Parlin. As recommended, the inspection was made through the side bung. Rejected drums were candidates for rechaining.

In discussing the cleaning problem with Bayonne personnel, they considered the furfuryl to be very efficient in removing the "Lucite". It was the consensus that failure to completely remove the "Lucite" from the drum chime was largely due to the age of the dirty drum prior to reconditioning. In many instances, it was believed that the dirty chime was the result of poor previous reconditioning so that the material had become hard and insoluble. No apparent difficulty is generally experienced with drums that have been cleaned for the first time.

On further discussing the results of the first practical test with Bayonne personnel, considerable concern was expressed over the low percentage (22%) of drums that passed inspection. For satisfactory drum cleaning, a minimum of 80% is expected. Bayonne contended that inspection adopted is too critical and highly impractical.

On the basis of the test performance, the following changes in the reconditioning process were recommended:

1. Replace sodium hydroxide (caustic) with tri-sodium phosphate in the chaining and rinsing operation.

2. Increase the chaining time in the 45° angle position and, if necessary, reduce the time in the horizontal position.

3. Consider the possibility of using the furfuryl alcohol cleaning for the chaining operation in addition to the pre-drum cleaning.

4. Limit the cleaning of "Lucite" drums to a single day operation rather than intermixing "Lucite" drum cleaning in regular production line methods.

It was reported that Flint was enjoying good success with reconditioned "Lucite" drums. The reconditioner was the American Barrel Company of Detroit. To compare the cleaning procedure and the quality of the drums, a visit was made to Detroit and Flint to conduct an evaluation.

Investigation disclosed the fact that quality-wise, the drums reconditioned by American Barrel have but a slight edge over those cleaned by Bayonne. This slight improvement can be attributed to the fact that American Barrel re-chains the drums as many as three times. In practically every instance, rejection is due to dirty chimes. Flint examines every drum prior to use, maintaining a fairly stringent inspection for "Lucite" drums. However, Flint is in a good position with regard to drums rejected for "Lucite", in that they can be used for undercoats. Because of this, they have very few totally unusable drums that are returned to the reconditioner.

The cleaning solution used by the American Barrel is one which they developed. They claim that the success of their cleaning depends entirely on the composition of the cleaner. They contend that the investment required to develop the cleaner was fairly large and they propose to sell it to other reconditioners. In fact, they would like to interest the Du Pont Company in the purchase of this material for sales purposes. Since they would not divulge the composition nor provide a sample, the general efficiency could not be evaluated.

As a matter of general interest, the drum reconditioning processes used by the American and Bayonne Barrel Companies are outlined as follows:

Operations	American Barrel Co.,Detroit	Bayonne Barrel & Drum Co.,Bayonne
1	Flush drums with American Barrel cleaning solution, 10 mins., at 180°F. - intermittent jet nozzle flushing.	Flush drums with Furfuryl Alcohol trisodium phosphate 4 mins. at 180°F.,intermittent jet nozzle spray cleaning,
2	Chain drums 20 mins. with above solution - 40 lbs. chains - 180°F. temperature controlled by gas jets directed on bottom chime.	Chain drums with slight amount of cleaning solution - 2 oz. tri-sodium phosphate added. Chained 40 mins. temperature not controlled 50 lbs. chain.
3	Drums drained	Drums drained
4	Drums flushed with caustic 180°F.	Drums flushed with tri-sodium phosphate 180°F.
5	Hot water wash.	Hot water wash.
6	Drums dried by gas flame directed on drum while rotating.	Drums dried by warm air 150-180°F.
7	Inspection of drum - rejected send thru chaining process 1-4 times.	Inspected - does not want to rechain.

The reconditioning system presently employed by Bayonne Barrel is that outlined above. Aside from the cost factor, Bayonne is well satisfied with the general performance. The expense, of course, is due to the initial cost of furfuryl. Active attempts are being made to salvage the furfuryl alcohol. Bayonne Barrel is considering the use of furfuryl for all drum cleaning providing they can revise the present system to a more efficient basis. As of May 10, 1961, Bayonne Barrel & Drum has increased the efficiency of the above furfuryl alcohol cleaning system to 90%.

Equipment and Tote Tank Stripper

There are many conditions where the furfuryl alcohol drum cleaner can be effectively employed to strip equipment and tote tanks. However, the conditions are usually limited to direct contact or by submerging. The reason being that the furfuryl alcohol mixture does not provide vapor cleaning properties. For tote tank and mixer cleaning, the following composition appears most effective:

Furfuryl Alcohol	20.0%
Methylene Chloride	20.0
Xylene	60.0
	100.0%

Laboratory tests show that this composition jet sprayed and re-circulated in a closed container, is very effective as a stripper for "Dulux", "Lucite", etc. By varying the methylene chloride content, the effectiveness can be controlled to remove most types of formulas. When used in conjunction with a satisfactory filtering system, the detergent can probably be reused several times for satisfactory cleaning. Large scale practical or field tests have not been made with this stripper.

Precautions for Handling Furfuryl Alcohol

Furfuryl alcohol has a low vapor pressure (0.5 mm at 70°F.), hence it is difficult to build up a high vapor concentration in the air at room temperature.

It is important that individuals working with furfuryl alcohol observe precautions commonly practiced in working with other chemicals, such as, butyl cellosolve, di-n-propyl ketone and dipropylene glycol mono-methyl ether (Dowanol 50-B). Such normal precautions are:

1. Avoid prolonged or repeated contact with skin and clothing and wash the skin promptly with soap and water when it is contacted.

2. Wear protective gloves or clothing when spillage is a possibility.

3. In case of contact with eyes, immediately flush with plenty of water, and obtain medical attention.

4. Provide adequate ventilation in areas where furfuryl alcohol will be handled.

In the presence of concentrated mineral acids or strong organic acids, furfuryl alcohol reacts with explosive violence; hence, precaution must be exercised to prevent the contact of furfuryl alcohol and strong acids, except under controlled conditions. Actually, an acid catalyst is used in resinification of furfuryl alcohol and millions of pounds of furfuryl alcohol resins are made in this manner every year, demonstrating again that the proper handling poses no problem and that the danger exists only when the material is misused.

Physiological Data

The Quaker Oats Company reports that furfuryl alcohol shows no tendency to produce irritation and sensitization of the skin. In support of this, complete physiological report has been submitted for our information.

ROI:nk
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TENTATIVE SPECIFICATIONS FOR RECONDITIONED METAL DRUMS
TIGHT HEAT DRUMS - FINISHES

Prior Usage: Drums that have been in service for shipment of solvent, plasticizer, paint, varnish or lacquer are required. Drums previously containing lubricating grease or edible oils are specifically excluded.

Dimensions: In accordance with applicable Metal Drum Specification. Height shall not vary more than ± 1/2" in any one shipment.

Gauge: In accordance with applicable Metal Drum Specification.

Hoops: Shall conform to original contour. No sharp dents permitted.

Chimes: Shall be rolled to original contour and tight.

Heads: Smooth, free of dents and conforming to original contours.

Location of Fittings: In accordance with applicable Metal Drum Specification.

Plugs: Tri Sure or Rieke - Shall be cleaned, free of worn or damaged threads, equipped with new gaskets in accordance with applicable Metal Drum Specification, capable of providing a leak-proof closure. Die cast plugs unless otherwise specified. No coating permitted.

Flanges: Tri Sure or Rieke - Shall be free of worn or damaged threads. Threads shall be clean and face of flange shall be smooth, capable of providing a leak-proof closure.

Body: Cylindrical, slight dents not more than 1/4" in depth nor 3" in diameter, shall not exceed 6 in number, provided the overall effect does not detract from the appearance.

Welds: No welding permitted within 3" of chime or fitting.

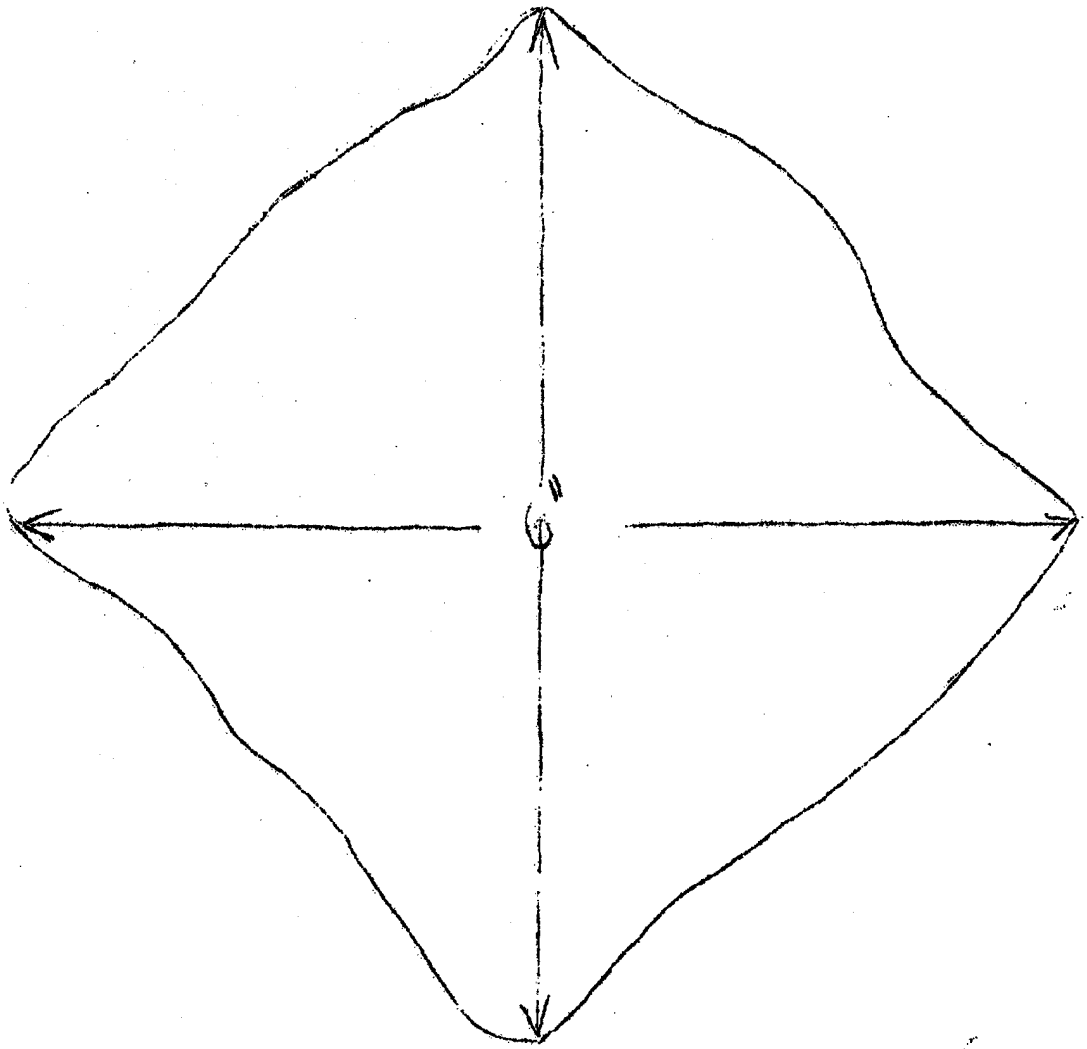
Leaks: All drums shall be structurally sound and free of leaks. (To be tested with air at 3 to 8 p.s.i. partly submerged).

Finish: INTERIOR

1. Shell - Clean and shiny, free from heavy red, rough rust.
Paint flakes, skins or chips are not permitted. Red, blue and green paint staining not allowed.

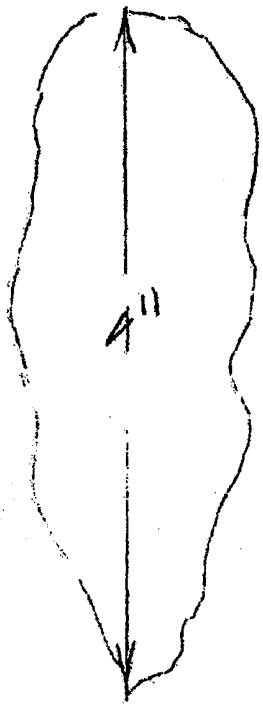
Exceptions: A. Rust Stains

Slight rust stains are permitted providing the chimes are clean and there are no more than 2-6" stained areas.



Exceptions (Contd.)

B. Paint Stains:

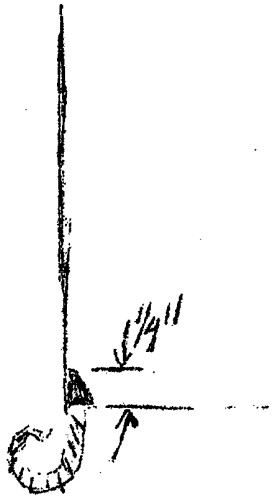


One area as shown allowed in any one drum if stain is very thin.
No red, blue or green stains allowed.

2. Chime:

Red, blue and green paint in chime is not permitted.

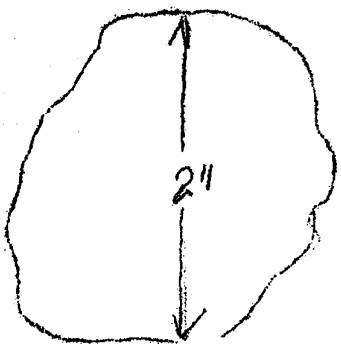
Exception:- Paint allowed in any one chime but not to exceed 1/4 inch.



3. Heads:

Clean and shiny, no evidence of red, blue or green paint stains or drippings.

Exception: If chime and shell is clean then staining on head 2 inches in area allowed.



EXTERIOR

Completely free of rust and foreign material. To be painted in accordance with applicable Metal Drum Specification after cleaning and blasting.

1 - Gaskets:

New gasket shall be provided in conformance with applicable Metal Drum Specification. Plugs shall be tightened to prevent breathing in storage.

2 - General:

The recommended drum must, in general, have the same appearance as a new drum. Conformance with specifications will be determined by inspection of drums as received.

3 - Testing:

Internal cleanliness may be checked by purchase, by solvent washing drum walls and testing purity of the solvent or by other methods as found necessary to safeguard product purity.

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