

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR
CHROME YELLOW (LEMON, MEDIUM, AND ORANGE; DRY,
PASTE IN OIL, AND PASTE IN JAPAN)

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 476

This specification was officially promulgated by the Federal Specifications Board on March 5, 1927, for the use of the departments and independent establishments of the Government in the purchase of chrome yellow (lemon, medium and orange; dry, paste in oil, and paste in japan).

[The latest date on which the technical requirements of this specification shall become mandatory for all departments and independent establishments of the Government is June 6, 1927. They may be put into effect, however, at any earlier date after promulgation.]

CONTENTS

	Page
I. General specifications.....	2
II. Grades.....	2
III. Material and workmanship.....	2
IV. General requirements.....	2
V. Detail requirements.....	2
1. Dry pigments.....	2
2. Paste in oil.....	2
3. Paste in japan.....	3
VI. Methods of sampling and testing.....	3
1. Sampling.....	3
2. Laboratory examination, dry pigment.....	4
3. Laboratory examination, paste in oil.....	5
4. Laboratory examination, paste in japan.....	8
5. Reagents.....	9
VII. Packing of shipments.....	10
VIII. Notes.....	10

I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. GRADES

This specification covers the grade of chrome yellow commonly known in the trade as "C. P." It covers a wide variety of lemon, medium, and orange yellows, both as dry pigments, as pastes in oil, and as pastes in japan.

III. MATERIAL AND WORKMANSHIP

See detail requirements.

IV. GENERAL REQUIREMENTS

See detail requirements.

V. DETAIL REQUIREMENTS

1. DRY PIGMENTS

The dry pigments shall be chemical precipitates consisting of normal or basic lead chromates or mixtures of these with or without admixtures of other insoluble compounds of lead, but without any other admixtures, and shall conform to the following requirements:

	Maximum per cent
Coarse particles (residue retained on No. 325 sieve).....	1.0
Total matter soluble in water.....	.5
Total of all substances other than compounds of lead.....	3.0
Organic colors or lakes.....	None.

The mass color and character of the tint shall be the same as and the tinting strength shall not be less than those of a sample mutually agreed upon by buyer and seller.

2. PASTE IN OIL

The paste color in oil shall consist of the pigment described above ground to a paste in linseed oil. The paste as received shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency and shall conform to the following requirements:

	Maximum	Minimum
	<i>Per cent</i>	<i>Per cent</i>
Pigment.....		75
Linseed oil.....	25	
Water and other volatile matter.....	.7	
Coarse particles and skins (total residue retained on No. 325 sieve).....	1.5	

The mass color and character of the tint shall be the same as and the tinting strength shall be not less than those of a sample mutually agreed upon by the buyer and seller.

3. PASTE IN JAPAN

The paste color in japan shall consist of the pigment described above thoroughly ground to a paste in high-grade grinding japan. The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within one hour to a hard, flat coat that can be varnished within five hours of time of application without streaking or bleeding. It shall also conform to the following requirements:

	Maximum	Minimum
	Per cent	Per cent
Pigment.....		70
Vehicle (containing not less than 40 per cent of nonvolatile matter).....	30	
Coarse particles and "skins" (total residue retained on No. 325 sieve).....	1.5	

The mass color and character of the tint shall be the same as and the tinting strength shall be not less than those of a sample mutually agreed upon by the buyer and seller.

VI. METHODS OF SAMPLING AND TESTING

Deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional information to ascertain whether the material meets the specification.

1. SAMPLING

It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1,000 packages shall be taken as representative of the whole.

With dry pigment, this package shall be opened by the inspector and a sample of not less than 1 pound taken at random from the contents. This shall be placed in a clean, dry metal or glass container closed with a tight cover, sealed, marked, and sent to the laboratory. With paste, whenever possible, an original unopened container shall be sent to the laboratory, and when this is for any reason not done, the inspector shall determine by thorough testing with a paddle or spatula whether the material meets the requirements regarding not caking in the container. See VI, 3, (a). He shall then thoroughly mix the contents of the container and draw a sample of not less than 1 pound. This sample shall be placed in a clean, dry metal or glass container, which it must nearly fill. The container shall be closed with a tight cover, sealed, and sent to the laboratory for test with the inspector's report on caking. The inspector should have a portion of the mutually agreed upon sample of the dry pigment, paste in oil, or paste in japan, as may be called for in the contract. Any delivery that is obviously different in color than the mutually agreed upon sample, or in case of paste that is badly caked in the container, shall be rejected by the inspector without sending a sample to the laboratory.

When requested, a duplicate sample may be taken from the same package and delivered to the seller, and the inspector may take a third sample to hold for test in case of dispute.

2. LABORATORY EXAMINATION, DRY PIGMENT

(a) MASS COLOR.—Weigh 1 g each of sample and the mutually agreed upon standard and rub up separately on a glass plate or stone slab, using the same amount of the same linseed oil in each case. Rubbing up (mixing with oil) is best done with a muller and should be such that no lumps remain and each lot receives the same amount of rubbing. Place portions of each side by side on a clear strip of glass, turn the glass over and compare the colors. If there is a difference in color the delivery does not meet the specification.

(b) CHARACTER OF TINT AND TINTING STRENGTH.—Weigh accurately on a counter-poised watch glass about 0.02 g of the sample and one hundred times as much dry zinc oxide and transfer to a large glass plate. Add 0.7 cc of clear raw linseed oil (measure from a burette) and mix with a clean steel (not nickel plated) spatula until the mass appears to be homogenous. Wipe off the spatula on the glass plate and on the rubbing surface of the glass muller. Grind the paste with a circular motion fifty times with the muller, using a uniform pressure. Gather up the paste with the spatula and grind again fifty times with the muller. Repeat the operation once more. Gather up the paste with the spatula and set aside. Using the standard mutually agreed upon in place of the sample, duplicate the above procedure. Stir each of the pastes with clean spatulas and transfer portions to a microscope slide, quite close together and draw a spatula across both so as to make them meet in a line. Compare the colors on both sides of the glass. If the sample differs from the standard in tone; that is, if it shows more reddish yellow or a more greenish yellow than the standard it does not meet the specification in character of tint. If it shows the same character of tint and the rub out is as dark or darker than the standard, it meets the specification in tinting strength. If it shows the same character of tint and the rub out is lighter than the standard it does not meet the specification in tinting strength.

(c) COARSE PARTICLES.—Dry in an oven at 105 to 110° C., a No. 325 sieve, cool, and weigh accurately. Weigh 10 g of the sample, transfer to a mortar, add 100 cc of water, thoroughly mix by gentle pressure with the finger to break up all the lumps, and wash through the sieve with a gentle stream of water, stirring gently with a camel's hair brush until nothing more passes through the sieve. Dry the sieve at 105 to 110° C. for one hour, cool, and weigh. From the increase in weight of the sieve and the weight of sample calculate the percentage of coarse particles.

(*d*) ORGANIC COLORS OR LAKES.—Test the pigment successively with hot water, 95 per cent alcohol and chloroform. If the solutions should remain colorless, organic colors are probably absent, but if organic colors resistant to the above reagent are suspected, other tests may be applied and it may be necessary to follow methods given in such books as Tests for Coal Tar Colors in Aniline Lake, by Zerr and Mayer; A Systematic Survey of Organic Coloring Matters, by Schultz and Julius; Identification of Pure Organic Compounds, by Mulliken.

(*e*) MATTER SOLUBLE IN WATER.—Transfer about 2.5 g (accurately weighed) to a 250 cc graduated flask. Add 100 cc distilled water and boil for five minutes. Allow to cool, fill to mark with distilled water, mix thoroughly and allow to settle. Decant through a dry filter and discard the first 25 cc of filtrate. Transfer 100 cc of the remaining clear filtrate to a weighed dish, evaporate to dryness, heat for one hour at 105 to 110° C., cool, weigh, and compute percentage of matter soluble in water.

(*f*) WATER AND OTHER VOLATILE MATTER.—Heat about 2 g, accurately weighed, in a tared dish or weighing bottle for three hours at 105 to 110° C., cool and weigh. Compute loss in weight as water and other volatile matter.

(*g*) INSOLUBLE MATTER.—Treat an accurately weighed portion of about 1 g with 25 cc of concentrated hydrochloric acid and boil for from 5 to 10 minutes in a covered beaker, adding about 6 drops of alcohol, 1 at a time, to the boiling liquid. Dilute to 100 cc with hot water and boil for from 5 to 10 minutes. Filter the hot solution (if insoluble matter is present) and wash with boiling water till washings are free from lead and chlorine. Ignite, weigh, and compute percentage of insoluble matter.

(*h*) OTHER IMPURITIES.—If careful qualitative analysis shows absence of all bases other than lead, as will generally be the case, no further examination will be necessary. If other bases are found, determine by appropriate methods.¹

(*i*) CALCULATION OF TOTAL OF ALL SUBSTANCES OTHER THAN COMPOUNDS OF LEAD.—Add together the percentages of matter soluble in water (VI, 2, (*e*)), water and other volatile matter (VI, 2, (*f*)), insoluble matter (VI, 2, (*g*)), and other impurities (VI, 2, (*h*)).

3. LABORATORY EXAMINATION, PASTE IN OIL

(*a*) CAKING IN CONTAINER.—When an original package is received in the laboratory, it shall be weighed, opened, and stirred with a stiff spatula or paddle. The paste must be no more difficult to break up than a normal good grade of similar material. The paste shall finally be thoroughly mixed, removed from the container, and the

¹ See A. S. T. M. standards D 126-23.

container wiped clean and weighed. This weight subtracted from the weight of the original package gives the net weight of the contents. A portion of thoroughly mixed paste shall be placed in a clean container and the portions for the remaining tests promptly weighed out.

(b) COLOR.—Place portions of the sample and of the standard mutually agreed upon side by side on a clean strip of glass. Turn the glass over and compare the colors. If there is a difference in color the delivery does not meet the specifications.

(c) CHARACTER OF TINT AND TINTING STRENGTH.—Weigh accurately about 0.03 g of the thoroughly stirred paste (weighing by difference and recording exact weight). Transfer to a large glass plate. Add to this 100 times as much dry zinc oxide (accurately weighed). Proceed as in VI, 2, (b).

(d) COARSE PARTICLES AND SKINS.—Dry in an oven at 105 to 110° a No. 325 sieve, cool, and weigh accurately. Weigh an amount of paste containing 10 g of pigment (see VI, 3, (g)), add 100 cc of kerosene, mix thoroughly, and wash through the sieve using kerosene, but otherwise follow VI, 2, (c).

(e) WATER AND OTHER VOLATILE MATTER.—Weigh accurately from 3 to 5 g of the paste into a tared flat-bottom dish about 8 cm in diameter, spreading the paste over the bottom, heat at 105 to 110° C. for three hours, cool and weigh.

(f) MIXING WITH LINSEED OIL.—Place 100 g of the paste in a cup. Add 50 cc raw linseed oil slowly with careful stirring and mix with a spatula or paddle. The resulting mixture must be smooth and of good brushing consistency.

(g) PERCENTAGE OF PIGMENT.—Weigh accurately about 15 g of the paste in a weighed centrifuge tube. Add 20 to 30 cc of "extraction mixture" (see Reagents), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add enough of the reagent to make a total of 60 cc in the tube. Place the tube in the container of a centrifuge, surround with water, and counterbalance the container of the opposite arm with a similar tube or a tube with water. Whirl at a moderate speed until well settled. Decant the clear supernatant liquid, repeat the extraction three times with 40 cc of extraction mixture.

After drawing off the extraction mixture, set the tube in a beaker of water at about 80° C. or on top of a warm oven for 10 minutes, then in an oven at 105 to 110° C. for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 sieve to remove any skins, and preserve in a stoppered bottle.

(h) PREPARATION OF FATTY ACIDS.—To about 25 g of the paste in a porcelain casserole, add 15 cc of aqueous sodium hydroxide (see

Reagents) and 75 cc of ethyl alcohol, mix and heat uncovered on a steam bath until saponification is complete (about an hour). Add 100 cc of water, boil, add sulphuric acid of specific gravity 1.2 (8 to 10 cc in excess), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer, wash once with water, then add 50 cc of water and 50 cc of ether. Shake very gently with a whirling action to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15 cc portion of water and then with 5 cc portions of water until free from sulphuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g of anhydrous sodium sulphate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sulphate. Decant this clear solution, if necessary, through a dry filter paper into a dry 100 cc Erlenmeyer flask. Pass a rapid current of dry air (pass through a CaCl₂ tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C. on a dry, hot plate until the ether is entirely driven off.

It is important to follow all of the details, since ether generally contains alcohol, and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulphate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(i) TEST FOR MINERAL OIL AND OTHER UNSAPONIFIABLE MATTER.—Place 10 drops of the fatty acid (*h*) in a 50 cc test tube, add 5 cc of alcoholic soda (see Reagents) boil vigorously for five minutes, add 40 cc of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present. If the solution is not clear, the oil is not pure linseed oil.

(j) IODINE NUMBER OF FATTY ACIDS.—Place a small quantity of the fatty acids (*h*) in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.09 to 0.15 g into a 500 cc bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add

10 cc of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 cc of the Wijs solution (see Reagents) and let stand with occasional shaking for one hour in a dark place at a temperature of from 21 to 23° C. Add 10 cc of the 15 per cent potassium iodide solution and 100 cc of water, and titrate with standard sodium thiosulphate, using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulphate solution, calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g of sample.) If the iodine number is less than 175 the oil does not meet the specification.

(*k*) EXAMINATION OF EXTRACTED PIGMENT.—Apply tests VI, 2, (*d*); VI, 2, (*e*); VI, 2, (*g*); VI, 2, (*h*); and VI, 2, (*i*).

4. LABORATORY EXAMINATION, PASTE IN JAPAN

(*a*) CAKING IN CONTAINER.—Apply test VI, 3, (*a*).

(*b*) COLOR.—Apply test VI, 3, (*b*).

(*c*) CHARACTER OF TINT AND TINTING STRENGTH.—Make test in a manner similar to VI, 3, (*c*), except that on account of the volatile matter in japan pastes it is not easy to weigh exact amounts. For weighing, the materials should be placed in a wide-mouth stoppered weighing tube along with a short spatula or spoon. After weighing the whole, transfer with the spatula or spoon about 0.03 g to the glass plate, return spatula or spoon to weighing bottle, insert stopper, and again weigh. (To avoid trouble due to drying while weighing the zinc oxide cover the paste with about half of the oil to be later used.) The difference in weight gives the weight of material taken. Then weigh one hundred times this weight of zinc oxide and proceed as in VI, 2, (*b*).

(*d*) COARSE PARTICLES AND SKINS.—Follow method gives in VI, 3, (*d*).

(*e*) VOLATILE MATTER.—Follow method VI, 3, (*e*) with the precautions regarding weight of sample given in VI, 4, (*c*).

(*f*) MIXING WITH TURPENTINE AND DRYING TESTS.—Place about 100 g of the paste in a cup and add turpentine slowly from a burette while thoroughly mixing with a spatula or paddle. The paste should readily break up and form a paint of brushing consistency. Note the volume of turpentine required. This will vary somewhat, but, in general, about 50 cc will suffice. Thoroughly stir this paint, strain through a No. 100 sieve and apply by brushing to a clean metal panel held in a vertical position. Apply the paint to only a

part of the panel, leaving a margin of at least 2.5 cm (1 inch) of unpainted metal around the painted portion. It is best to use a camel's-hair brush for this painting. Allow the panel to stand in a vertical position in a well-ventilated room at room temperature (70 to 90° F.) for 1 hour. The film should be flat, of extremely fine and smooth texture, uniform as regards color and finish and must be hard enough to stand light rapid rubbing with the finger without being removed. After a total of five hours drying, a brush coat of Damar varnish (Damar resin cut in turpentine) shall be applied over the entire surface of the panel. Any softening or raising of the flat coat or any separation of color by this varnishing operation is cause for rejection. In case of dispute on this drying test due to atmospheric conditions, and umpire tests are necessary such tests shall be made in a well-ventilated room maintained at a temperature of 70° F. and relative humidity of 65 per cent saturation.

(g) PERCENTAGE OF PIGMENT.—Using the precautions in weighing sample given in VI, 4, (c) follow method given in VI, 3, (g).

(h) EXAMINATION OF EXTRACTED PIGMENT.—Apply tests VI, 2, (d); VI, 2, (e); VI, 2, (g); VI, 2, (h); and VI, 2, (i).

5. REAGENTS

(a) EXTRACTION MIXTURE.—

10 volumes ether (ethyl ether).

6 volumes benzol.

4 volumes methyl alcohol.

1 volume acetone.

(b) AQUEOUS SODIUM HYDROXIDE.—Dissolve 100 g of sodium hydroxide in distilled water and dilute to 300 cc.

(c) ALCOHOLIC SODIUM HYDROXIDE SOLUTION.—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g per 1,000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g to 1,000 cc), kept at about 50° C. for 15 days and then distilled.

(d) POTASSIUM IODIDE SOLUTION.—Dissolve 150 g of potassium iodide free from iodate in distilled water and dilute to 1,000 cc.

(e) WIJS SOLUTION.—The preparation of the iodine monochloride solution presents no great difficulty, but it should be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This

condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using gentle heat to assist the solution, if it is found necessary. Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15° C. and is free from reducing impurities in the proportion so that 13 g of iodine will be present in 1,000 cc of solution. Set aside a small portion of this solution while pure, and pass dry chlorine into the remainder until the halogen content of the solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

(f) STANDARD SODIUM THIOSULPHATE SOLUTION.—Dissolve pure sodium thiosulphate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g of crystallized sodium thiosulphate to 1,000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine.² This solution will be approximately decinormal, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly decinormal. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(g) STARCH SOLUTION.—Stir up 3 g of potato starch or 5 g of soluble starch with 100 cc of 1 per cent salicylic acid solution, add 300 to 400 cc of boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

VII. PACKING OF SHIPMENTS

Packing shall be in accordance with commercial practice unless otherwise specified.

VIII. NOTES

There are very many different hues and shades that can with equal exactness be called chrome yellow. The selection of the one desired is entirely a matter of taste. There is no practical method of getting the color desired, except that of having it match in color, character of tint, and tinting strength the sample mutually agreed upon. The composition requirements of the specification are of only secondary importance.

² Treadwell-Hall, Analytical Chem. 2, 6th ed. p. 551.

All purchasers and users of specification paint materials should consult Bureau of Standards Technologic Paper No. 274, entitled "Use of United States Government Specification Paints and Paint Materials." Material covered by this specification is not discussed in that publication, but the information contained therein will nevertheless be of value.

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