Doc: PCD 25 (27228) WC
January 2025

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भारतीय मानक मसौदा

एल्युमीनियम कोल्ड रोलिंग तेल – फॉयल और शीट रोलिंग – विशिष्टि

(IS 14385 *का पहला पुनरीक्षण*)

Draft Indian Standard

ALUMINIUM COLD ROLLING OILS —FOIL AND SHEET ROLLING — SPECIFICATION

(*First Revision of* IS 14385)

(ICS 75.100)

Lubricants and their related products Sectional Committee, PCD 25	Last date for receipt of comment is **8 March 2025**

FOREWORD

(*Formal clauses will be added later*)

Rolling is one of the industrial processes which uses the plastic behaviour of metals for producing shape change. The increasing demand of aluminium and its alloys has prompted the introduction of mass production rolling techniques similar to those practiced for the rolling of steel. Thus, development of Indian Standards for the oils used between the moving rolls during the deformation of aluminium into foil or sheets is essential on lines similar to those in practice for rolling of steel.

Rolling of aluminium is done in both hot and cold conditions. During the hot rolling operation, 20 cm to 30 cm thick cast aluminium slabs are converted into hot plate or strip of desired thickness, normally 4 mm to 12 mm, which after cooling to room temperature, are rolled in cold mill to produce sheets 0.25 mm to 5.0 mm approximately and foils of required finish gauge 0.12 mm to 0.006 mm and tempered.

In the case of hot rolling, cooling is done by soluble oil emulsion in water, where water acts as a coolant and the oil portion of the emulsion provides lubrication.

In the case of cold rolling, straight run light mineral oil distilled with low viscosity of 1.8 cSt to 4.0 cSt at 40 °C are generally used for sheet and foil production. Normally such special distillate cuts are used together with suitable dosage of film forming additives.

The first revision of this standard has been brought out to reflect the latest practices and specification in industry for aluminium cold rolling oils. The major changes in this revision are:

a) The title of the standard has been changed to reflect both sheet and foil rolling applications;
b) The grades have been modified to reflect end use application, i.e., sheet rolling and foil rolling;
c) As per end use application, the requirements for kinematic viscosity, flash point, distillation characteristics, total acid number, ageing properties, sulphur content, and water content have been modified; and
d) Requirement for minimum content of paraffins and naphthenes added.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirement and the methods of sampling and test for aluminium cold rolling oils, intended for use for both sheet and foil rolling of aluminium.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed in Annex A.

3 GRADES

The material shall be of the following two grades:

Grade I — Intended for aluminium foil rolling.
Grade II — Intended for aluminium sheet rolling.

4 REQUIREMENTS

4.1 General Requirement

The oil shall be clear, homogeneous and free from foreign matter, sediment and visible impurities. It shall not contain any ingredients injurious to persons using or handling it.

4.2 Specific Requirement

The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in col 5 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed is securely closed metal drums or other suitable containers of appropriate size and strength or tankers as agreed between the purchaser and the supplier.

5.2 Marking

5.2.1 Each container (except tanker) shall be marked with the following information:

a) Name and grade of the material;
b) Manufacturer’s name, initials or trade-mark, if any;
c) Quantity of the material; and
d) Batch number and year of manufacture.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 KEEPING PROPERTIES

The material when stored in original sealed containers under ambient temperature conditions in shade shall retain the properties described under **4** for a period of not less than one year from the date of manufacture.

7 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).

Table 1 Requirements of Aluminium Cold Rolling Oil - Sheet & Foil
(*Clause* 4.2)

Sl	Characteristic	Requirements	Methods of Test

No.		**Grade I Foil Rolling**	**Grade II Sheet Rolling**	
(1)	**(2)**	**(3)**	**(4)**	**(5)**
i)	Colour, ASTM, *Max*	0.5	0.5	IS 1448 (Part 12)
ii)	Kinematic viscosity, mm^2/s at 40 °C	1.8 – 2.0	2.0 – 2.6	IS 1448 (Part 25/Sec 1)
iii)	Flash point, COC, °C, *Min*	80	105	IS 1448 (Part 69)
iv)	Distillation characteristics:			IS 1448 (Part 18)
	a) Initial Boiling Point (IBP), °C, *Min*	200	240	
	b) Final Boiling Point (FBP), °C, *Max*	240	280	
v)	Acidity inorganic, mg KOH/g	Nil	Nil	IS 1448 (Part 2)
vi)	Total Acid Number (TAN) mg KOH/g, *Max*	0.02	0.02	
vii)	Ageing			Annex B
	a) Total Acid Number, mg KOH/g, *Max*, after ageing (at 110 ± 2 °C for 48 h)	0.03	0.03	
	b) Colour change after ageing	Unchanged		
viii)	Sulphur, percent by mass, *Max*	0.000 1	0.000 1	IS 1448 (Part 33)
ix)	Paraffins and Naphthene, percent by volume, *Min*	99.5	99.5	IS 1448 (Part 23)
x)	Bromine number by electrometric titration, *Max*	1	1	IS 1448 (Part 44)
xi)	Ultraviolet (UV) absorbance, *Max* at wavelength in millimicron			Annex C
	280 – 289	0.7	0.7	
	290 – 299	0.6	0.6	
	300 – 359	0.4	0.4	
	360 – 400	0.9	0.9	
xii)	Water Content, percent by mass, *Max*	0.01	0.01	IS 1448 (Part 182)

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No./ Other Standards	*Title*
IS 1447 (Part 1): 2021	Methods of sampling of Petroleum and its products : Part 1 Manual sampling (*second revision*)
IS 1448	Method of test for petroleum and its products
(Part 2): 2007 /ISO 6619: 1988	Petroleum products and lubricants — Neutralization number — Potentiometric titration method (*second revision*)
(Part 12): 2013/ ISO 2049 : 1996	Determination of colour (ASTM scale) (*second revision*)
(Part 18): 2020	Distillation of petroleum products (*third revision*)
(Part 23): 2004/ ISO 3837: 1993	Liquefied petroleum gases — Determination of hydrocarbon types — Fluorescent indicator adsorption method (*fourth revision*)
(Part 25/Sec 1) :2018/ISO 3104 : 1994	Transparent and opaque liquids Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
(Part 33) : 2021	Sulphur by high pressure decomposition device method (*third revision*)
(Part 44): 2013/ ISO 3839 : 1996	Determination of bromine number of distillates and Aliphatic olefins — Electrometric method (*third revision*)
(Part 69): 2019 / ISO 2592: 2017	Determination of flash and fire points — Cleveland open cup method (*second revision*)
(Part 182) : 2020 / ISO 12937 : 2000	Petroleum Products - Determination of Water - Coulometric Karl Fischer Titration Method

ANNEX B

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF TOTAL ACID NUMBER (TAN) AFTER AGEING

B-l PROCEDURE

Take 100 g of sample in 250 ml beaker and keep it in an oven at (110 ± 2) °C for 48 h. After that take out the beaker and test the sample for total acid number (TAN), mg KOH/g, as per method described in IS 1448 (Part 2).

ANNEX C

[*Table* 1, *Sl No*. (xi)]

DETERMINATION OF TEST METHOD FOR ULTRAVIOLET (UV) ABSORBANCE

C-l GENERAL

Because of sensitivity of the test, the possibility of errors arising from contamination, is great. It is of the greatest importance that all glassware be scrupulously cleaned to remove all organic matter such as oil, grease, detergent residues, etc. Examine all glassware, including stoppers and stopcocks, under ultraviolet light to detect any residual fluorescent contamination. As a precautionary measure it is recommended practice to rinse all glassware with purified iso-octane immediately before use. No grease is to be used on stopcocks or joints. Great care to avoid contamination of oil samples in handling and to assure absence of any extraneous material arising from inadequate packaging is essential, because some of the polynuclear hydrocarbons sought in this test are very susceptible to photo-oxidation, the entire procedure is to be carried out under subdued light.

C-2 APPARATUS

C-2.1 Separatory Funnels

250 ml, 500 ml, 1 l, and 2 l capacity, equipped with tetrafluoroethylene polymer stopcocks.

C-2.2 Reservoir

500 ml capacity, equipped with a 24/40 standard taper male fitting at the bottom and a suitable ball-joint at the top for connecting to the nitrogen supply. The male fitting should be equipped with glass hooks.

C-2.3 Chromatographic Tube

180 ml in length, inside diameter to be (15.7 ± 0.1) ml, equipped with a coarse, fitted-glass disc, a tetrafluoroethylene polymer stopcock, and a female 24/40 standard tapered fitting at the opposite end (overall length of the column with the female joint is 235 ml). The female fitting should be equipped with glass hooks.

C-2.4 Disc

Tetrafluoroethylene polymer 50 mm diameter disk approximately 3 mm thick with a hole bored in the centre to closely fit the stem of the chromatographic tube.

C-2.5 Suction Flask

250 ml or 500 ml filter flask

C-2.6 Condenser

24/40 joints, fitted with a drying tube, length optional.

C-2.7 Evaporation Flask (Optional)

250 ml or 500 ml capacity all glass flask equipped with standard taper stopper having inlet and outlet tubes to permit passage of nitrogen across the surface of contained liquid to be evaporated.

C-2.8 Spectrophotometric Cells

Fused quartz cells, optical path length in the range of (5 000 ± 0.005) cm, also for checking spectrophotometer performance only, optical path length in the range (1 000 ± 0.005) cm. With distilled water in the cells, determine any absorbance differences.

C-2.9 Spectrophotometer

Spectral range 250 millimicron to 400 millimicron with spectral width of 2 millimicron or less; under instrument operating conditions for these absorbance measurements, the spectrophotometer shall also meet the following performance requirements:

Absorbance repeatability, ± 0.01 at 0.4 absorbance

Absorbance accuracy, ± 0.05 at 0.4 absorbance

Wavelength accuracy, ± l.0 millimicron

C-2.10 Nitrogen Cylinder

Water pumped or equivalent purity nitrogen in cylinder equipped with regulator and valve to control flow at 34 KPa.

C-3 REAGENTS AND MATERIALS

C-3.1 Organic Solvents

All solvents used throughout the procedure shall meet the specifications and tests described in this standard. The iso-octane, benzene, acetone, and methyl alcohol designated in the list following this paragraph shall pass the following test:

C-3.1.1 To the specified quantity of solvent in a 250 ml Erlenmeyer flask, add 1 ml of purified *n*-hexadecane and evaporate on the steam bath under a stream of nitrogen (a loose aluminium foil jacket around the flask will speed evaporation). Discontinue evaporation when not over 1 ml of residue remains. To the residue from benzene add a 10 ml portion of purified iso-octane, re-evaporate, and repeat once to insure complete removal of benzene.

C-3.1.2 Alternatively, the evaporation time can be reduced by using the optional evaporation flask. In this case the solvent and *n*-hexadecane are placed in the flask on the steam bath, the tube assembly is inserted, and a stream of nitrogen is fed through the inlet tube while the outlet tube is connected to a solvent tap and vacuum line in such a way as to prevent any flow-back of condensate into the flask.

C-3.1.3 Dissolve the 1 ml of hexadecane residue in iso-octane and make to 25 ml volume. Determine the absorbance in the 5 cm path length cells compared to iso-octane as reference. The absorbance of the solution of the solvent residue (except for methyl alcohol) shall not exceed 0.01 cm path length between 280 µm and 400 µm. For methyl alcohol this absorbance value shall be 0.00.

C-3.2 Iso-octane (2,2,4-trimethylpentne)

Use 180 ml for the test described in the preceding paragraph. Purify, if necessary, by passage through a column of activated silica gel about 90 cm in length and 5 cm to 8 cm in diameter.

C-3.3 Benzene, A.C.S. Reagent Grade

Use 150 ml for the test. Purify, if necessary, by distillation or otherwise.

C-3.4 Acetone, A.C.S. Reagent Grade

Use 200 ml for the test. Purify, if necessary, by distillation:

Eluting mixtures:

a) 10 percent benzene in iso-octane — Pipette 50 ml of benzene into a 250 ml glass-stoppered volumetric flask and adjust to volume with iso-octane, with mixing.

b) 20 percent benzene in iso-octane — Pipette 50 ml of benzene into a 250 ml glass-stoppered volumetric flask and adjust to volume with iso-octane, with mixing.

c) Acetone-benzene-water mixture — Add 20 ml of water to 380 ml of acetone and 200 ml of benzene, and mix.

C-3.5 Hexadecane, 99 Percent Olefin-Free

Dilute 1.0 ml of *n*-hexadecane to 25 ml with iso-octane and determine the absorbance in a 5 cm cell compared to iso-octane as reference point between 280 µm to 400 µm. The absorbance per cm path length shall not exceed 0.00 in this range. Purify, if necessary, by percolation through activated silica gel or by distillation.

C-3.6 Methyl Alcohol, A.C.S. Reagent Grade

Use 10.0 ml of methyl alcohol. Purify, if necessary, by distillation.

C-3.7 Dimethyl Sulfoxide

Spectrophotometric grade (l cm cell, distilled water reference, sample completely saturated with nitrogen).

Wavelength	*Absorbance Max*
261.5	1.00
270	0.20
275	0.09
280	0.06
300	0.015

There shall be no irregularities in the absorbance curve within these wavelengths.

C-3.8 Phosphoric Acid

85 percent A.C.S. reagent grade

C-3.9 Sodium Borohydride, 98 percent

C-3.10 Magnesium Oxide

Place 100 g of the magnesium oxide in a large beaker, add 700 ml of distilled water to make a thin slurry, and heat on a steam bath for 30 min with intermittent stirring. Stir well initially to insure that all the absorbent is completely wetted. Using a Buchner funnel and a filter paper (Schleicher and Schuell No. 597, or equivalent) of suitable diameter, filter with suction. Continue suction until water no longer drips from the funnel. Transfer the absorbent to a glass trough lined with aluminium foil (free from rolling oil). Break up the magnesia with a clean spatula and spread out the absorbent on the aluminium foil in a layer about 1 cm to 2 cm thick. Dry for 24 h at (160 ± 1) °C. Pulverize the magnesia with mortar and pestle. Sieve the pulverized adsorbent between 60 mesh to 180 mesh. Use the magnesia retained on the 180 mesh sieve.

C-3.11 Magnesium Oxide - Celite 545 Mixture (2+1) by Weight

Place the magnesium oxide (60 mesh to180 mesh) and the Celite 545 in 2 to 1 proportions, respectively, by weight in a glass stoppered flask large enough for adequate mixing. Shake vigorously for 10 min. Transfer the mixture to a glass trough lined with aluminium foil (free from rolling oil) and spread it out on a layer about 1 cm to 2 cm thick. Reheat the mixture at (160 ± 1) °C for 2 h and store in a tightly closed flask.

C-3.12 Sodium Sulphate, Anhydrous, A.C.S. Reagent Grade Preferably in Granular Form

For each bottle of sodium sulphate reagent used, establish as follows the necessary sodium sulphate prewash to provide such filters required in the method. Place approximately 35 g of anhydrous sodium sulphate in a 30 ml course, fritted-glass funnel or in a 65 ml filter funnel with glass wool plug wash with successive l5 ml portions of the indicated solvent until a 15 ml portion of the wash shows 0.00 absorbance per cm path length between 280 μm and 400 μm when tested as prescribed under **C-3.1**. Usually three portions of wash solvent are sufficient.

C-4 PROCEDURE

C-4.1 Before proceeding with analysis of a sample, determine the absorbance in a 5 cm path cell between 250 millimicron and 400 millimicron for the reagent blank by carrying out the procedure, without an oil sample, recording the spectra after the extraction stage and after the complete procedure as prescribed. The absorbance per cm path length following the extraction stage should not exceed 0.02 in the wavelength range from 280 μm to 400 μm; the absorbance per cm path length following the complete procedure should not exceed 0.02 in the wavelength range from 280 μm to 400 μm. If in either spectrum the characteristic benzene peaks in the 250 μm to 260 μm region are present, remove the benzene by the procedure under **C-3.1** and record absorbance again.

C-4.2 Place 300 ml of dimethyl sulfoxide in a 1 l separatory funnel and add 75 ml of phosphoric acid. Mix the contents of the funnel and allow to stand for 10 min. The reaction between the sulfoxide and the acid is exothermic. Release pressure after mixing, then keep funnel stoppered. Add 150 ml of iso-octane and shake to pre-equilibrate the solvents. Draw off the individual layers and store in glass stoppered flasks.

C-4.3 Weigh a 20 g sample of the oil and transfer to a 500 ml separatory funnel containing 100 ml of pre-equilibrated sulfoxide phosphoric acid mixture. Complete the transfer of the sample with small portions of pre-equilibrated iso-octane to give a total volume of the oil and solvent of 75 ml. Shake the funnel vigorously for 2 min. Set up three 250 ml separatory funnels with each containing 30 ml of pre-equilibrated iso-octane. After separation of liquid phases, carefully draw off lower layer into the first 250 ml separatory funnel and wash in tandem with the 30 ml portions of iso-octane contained in the 250 ml separatory funnels, shaking time for each wash is 1 min. Repeat the extraction operation with two additional portions of the sulfoxide acid mixture and wash each extract in tandem through the same three portions of iso-octane.

C-4.4 Collect the successive extracts (300 ml total) in a separatory funnel (preferably 2 l) containing 480 ml of distilled water, mix and allow to cool for a few minutes after the last extract has been added, add 80 ml of iso-octane to the solution and extract by shaking the funnel vigorously for 1 min. Draw off the lower aqueous layer into a second separatory funnel (preferably 2 l) and repeat the extraction with 80 ml of iso-octane. Draw off and discard the aqueous layer. Wash each of the 80 ml extracts three times with 100 ml portions of distilled water. Shaking time for each wash is 1 min. Discard the aqueous layers. Filter the first extract through anhydrous sodium sulphate prewashed with iso-octane (*see* **C-3.2**) into a 250 ml Erlenmeyer flask (or optionally into the evaporation flask). Wash the first separatory funnel with second 80 ml iso-octane extract and pass through the sodium sulphate. Then wash the second and first separatory funnels successively with a 20 ml portion of iso-octane and pass the solvent through the sodium sulphate into the flask. Add 1 ml of *n*-hexadecane and evaporate the iso-octane on the steam bath under nitrogen. Discontinue evaporation when not over 1 ml of residue remains. To the residue, add 10 ml portion of iso-octane, re-evaporate to 1 ml of hexadecane, and repeat this operation once.

C-4.5 Quantitatively transfer the residue with isooctane to a 200 ml volumetric flask, make to volume, and mix. Determine the absorbance of the solution in the 1 cm path length cells compared to iso-octane as reference between 280 µm to 400 µm (take care to lose none of the solution in filling the sample cell). Correct the absorbance values for any absorbance derived from reagents as determined by carrying out the procedure without an oil sample. If the corrected absorbance does not exceed the limits prescribed in this paragraph, the oil meets the ultraviolet absorbance specifications. If the corrected absorbance per cm path length exceeds the limits prescribed in this paragraph, proceed as follows:

C-4.5.1 Quantitatively transfer the iso-octane solution to a 125 ml flask equipped with 24/40 joint, and evaporate the iso-octane on the steam bath under a stream of nitrogen to a volume of 1 ml of hexadecane.

C-4.5.2 Add 10 ml of methyl alcohol and approximately 0.3 g of sodium borohydride (minimize exposure of the borohydride to the atmosphere, a measuring dipper may be used). Immediately fit a water-cooled condenser equipped with a 24/40 joint and with a drying tube into the flask, mix until the borohydride is dissolved, and allow to stand for 30 min at room temperature, with intermittent swirling.

C-4.5.3 At the end of this period, disconnect the flask and evaporate the methyl alcohol on the steam bath under nitrogen until the sodium borohydride begins to come out of the solution. Then add 10 ml of iso-octane and evaporate to a volume of about 2 ml to 3 ml. Again add 10 ml of iso-octane and concentrate to a volume of approximately 5 ml. Swirl the flask repeatedly to assure adequate washing of the sodium borohydride residues.

C-4.6 Fit the tetrafluoroethylene polymer disc on the upper part of the stem of the chromatographic tube, then place the tube with the disc on the suction flask and apply with vacuum (approximately 135 mm Hg pressure). Weigh out 14 g of the 2 : l Magnesium oxide-Celite 545 mixture and pour the adsorbent mixture into the chromatographic tube in approximately 3 cm layers. After the addition of each layer, level off the top of the adsorbent with a flat glass rod or metal plunger by pressing down firmly until the adsorbent is well packed. Loosen the topmost few millimetres of each adsorbent layer with the end of a metal rod before the addition of the next layer. Continue packing in this manner until all the 14 g of the adsorbent is added to the tube. Level off the top of the adsorbent by pressing down firmly with a flat glass rod or metal plunger to make the depth of the adsorbent bed approximately 12.5 cm in depth. Turn off the vacuum and remove the suction flask. Fit the 500 ml reservoir onto the top of the chromatographic column and prewet the column by passing 100 ml of iso-octane through the column. Adjust the nitrogen pressure so that the rate of descent of the iso-octane coming off the column is between 2 ml to 3 ml per min. Discontinue pressure just before the last of the iso-octane reaches the level of the adsorbent. (Caution: Do not allow the liquid level to recede below the adsorbent level at any time.) Remove the reservoir and decant the 5 ml iso-octane concentrate solution onto the column and with slight pressure again allow the liquid level to recede to barely above the adsorbent level.

Rapidly complete the transfer similarly with two 5 ml portions of iso-octane, swirling the flask repeatedly each time to assure adequate washing of the residue. Just before the final 5 ml wash reaches the top of the adsorbent, add 100 ml of iso-octane to the reservoir and continue the percolation at the 2 ml to 3 ml per min rate. Just before the last of the iso-octane reaches the adsorbent level, add 100 ml of 10 percent benzene in iso-octane to the reservoir and continue the percolation at the aforementioned rate. Just before the solvent mixture reaches adsorbent level, add 25 ml of 20 percent benzene in iso-octane to the reservoir and continue the percolation at 2 ml to 3 ml per min, until all this solvent mixture has been removed from the column. Discard all the elution solvents collected up to this point. Add 300 ml of the acetone-benzene-water mixture to the reservoir and percolate through the column to eluate the polynuclear compounds. Collect the eluate in a clean 1 l separatory funnel. Allow the column to drain until most of the solvent mixture is removed. Wash the eluate three times with 300 ml portions of distilled water, shaking well for each wash (the addition of small amounts of sodium chloride facilitates separation). Discard the aqueous layer after each wash. After the final separation, filter, the residual benzene through anhydrous sodium sulphate pre-washed with benzene (*see* **C-3.3**) into a 250 ml Erlenmeyer flask (or optionally into the evaporation flask). Wash the separatory funnel with two additional 20 ml portions of benzene which are also filtered through the sodium sulphate. Add 1 ml of *n*-hexadecane and completely remove the benzene by evaporation under nitrogen, using the special procedure to eliminate benzene as previously described under **C-3.1**. Quantitatively transfer the residue with iso-octane to a 200 ml volumetric flask and adjust to volume. Determine the absorbance of the solution in the 1 cm path length cells compared to iso-octane as reference between 250 µm to 400 µm. Correct for any absorbance derived from the reagents as determined by carrying out the procedure without an oil sample. If either spectrum shows the characteristic benzene peaks in the 250 µm to 260 µm region; evaporate the solution to remove benzene by the procedure under **C-3.1.** Dissolve the residue, transfer quantitatively, and adjust to volume in iso-octane in a 200 ml volumetric flask. Record the absorbance again. If the corrected absorbance does not exceed the limits proposed in this paragraph, the oil meets the proposed ultraviolet absorbance specifications.