U. S. DEPARTMENT OF COMMERCE BUREAU OF STANDARDS

# **FELDSPAR**

# **COMMERCIAL STANDARD CS23-30**



ELIMINATION OF WASTE Through SIMPLIFIED COMMERCIAL PRACTICE Below are described some of the series of publications of the Department of Commerce which deal with various phases of waste elimination.

#### Simplified Practice Recommendations.

These present in detail the development of programs to eliminate unnecessary variety in sizes, dimensions, styles, and types of over 100 commodities. They also contain lists of associations and individuals who have indicated their intention to adhere to the recommendations. These simplified schedules, as formulated and approved by the industries, are indorsed by the Department of Commerce.

#### Commercial Standards.

These are developed by various industries under a procedure similar to that of simplified practice recommendations. They are, however, primarily concerned with considerations of grade, quality, and such other characteristics as are outside the scope of dimensional simplification.

#### American Marine Standards.

These are promulgated by the American Marine Standards Committee, which is controlled by the marine industry and administered as a unit of the division of simplified practice. Their object is to promote economy in construction, equipment, maintenance, and operation of ships. In general, they provide for simplification and improvement of design, interchangeability of parts, and minimum requisites of quality for efficient and safe operation.

Lists of the publications in each of the above series can be obtained by applying to the National Bureau of Standards, Washington, D. C.

# U. S. DEPARTMENT OF COMMERCE R. P. LAMONT, Secretary BUREAU OF STANDARDS GEORGE K. BURGESS, Director

# **FELDSPAR**

# **COMMERCIAL STANDARD CS23-30**

(ISSUED OCTOBER 22, 1930)

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#### COMMERCIAL STANDARD CS23-30

#### ACCEPTORS

#### ASSOCIATIONS

Carborundum Co., The, Perth Amboy, N. J.

Carling Tile Co., Macon, Ga.

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- Robinson, Ill. Porcelain Co., Detroit, Champion Mich.
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American Ceramic Society, Columbus, Ohio (in principle).

- of American Institute Mining and Metallurgical Engineers, Washington, D. C. (in principle).
- New England Council, Boston, Mass. (in principle).

#### FIRMS

- Aetna Porcelain Enameling Co., Chicago, Ill.
- American Chinaware Corporation, Coshocton, Ohio. American Emery Wheel Works, Provi-
- dence, R. I.
- American Enameled Brick & Tile Co., South River, N. J.
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- Gladding, McBean & Co., Los Angeles, Calif.
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- Hall China Co., East Liverpool, Ohio. Hamilton Co., J. T. & A., Pittsburgh,
- Pa.
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- Heinz Co., H. J., Sharpsburg, Pa. Highland-Western Glass Co., Wash-
- Inginand-Western Grass Co., Wash-ington, Pa.
  Homer Laughlin China Co., The, Newell, W. Va.
  Hoyt, Philip S., Las Tablas, N. Mex.
  Hull Pottery Co., The A. E., Crooks-
- ville, Ohio.
- Humphryes Manufacturing Co., The, Mansfield, Ohio.
- Illinois Electric Porcelain Co., Ma-comb, Ill.
- Ingram-Richardson Manufacturing Co., Frankfort, Ind.
- International Portery Co., The, Trenton, N. J. Iron City Sanitary Manufacturing Co.,
- Pittsburgh, Pa. Iroquois China Co., Syracuse, N. Y. Ketcham, O. W., Crum Lynne, Pa. Knight, Maurice A., Akron, Ohio (in

- principle)
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- Knox Glass Bottle Co., Knox, Pa. Knox Porcelain Corporation, Knoxville,
  - Tenn.
  - Kohler Co., Kohler, Wis.
  - Kokomo Sanitary Pottery Co., Kokomo, Ind.
  - Lambertville Pottery Co., Lambert-
  - ville, N. J. Lapp Insulator Co. (Inc.), Le Roy, N. Y.
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  - Limoges China Co., Sebring, Ohio. Locke Insulator Corporation, Baltimore, Md. (in principle). Long Beach Glass Co., Long Beach,
  - Calif.
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  - Mayer China Co., The, Beaver Falls, Рa.

  - McKee Glass Co., Jeanette, Pa. Mercer Pottery Co., Trenton, N. J. Michigan Porcelain Tile Works, Ionia, Mich.
  - Mitchell-Bissell Co., Trenton, N. J
  - Montgomery Porcelain Products Co., Franklin, Ohio. Monument Pottery Co. (Inc.), The,
  - Trenton, N. J. Mosaic Tile Co., The, Zanesville, Ohio.

  - Nashville Pottery Co. (Inc.), Nashville, Tenn. (in principle).
  - National Enameling & Stamping Co. (Inc.), Granite City, Ill. National Plate Glass Co., Detroit,
  - Mich.
  - National Portland Cement Co., Dallas, Tex.
  - Navias, Louis, Schenectady, N. Y. (in principle).
  - New Kensington Sanitary Pottery (Inc.), New Kensington, Pa. New York State School of Clayworking
  - and Ceramics, Alfred, N. Y. (in principle).
  - North Carolina State College, Raleigh, N. C. (in principle). North State Pottery Co., Sanford, N. C.
  - Northwestern Terra Cotta Co., Chicago,
  - Ill.
  - Obear Nester Glass Co., East St. Louis, III.

  - Olean Tile Co., Olean, N. Y. Onondaga Pottery Co., Syracuse, N. Y. Owens-Illinois Glass Co., Alton, Ill. Pacific Clay Products, Los Angeles,

  - Calif.

  - Paige Co., M. B., Peabody, Mass. Panama Pottery Co. (Inc.), Sacramento, Calif.
  - Pardee Works, The C., Perth Amboy, N. J.
  - Pass & Seymour (Inc.), Syracuse, N.Y. Peerless Enamel Products Co., Belleville, Ill. Pence Tile Works, F. K., Paducah, Ky.

- Pennsylvania State College, State Col-

- Pennsylvania State Conege, State Con-lege, Pa. (in principle). Penn Tile Works Co., Aspers, Pa. Perfection Stove Co., Cleveland, Ohio. Pfaudler Co., The, Rochester, N. Y. Phoenix Glass Co., The, Monaca, Pa. Pit and Quarry (magazine), Chicago, Ill. (in principle). Platts Premier Porcelsin (Inc.) Santa
- Platts Premier Porcelain (Inc.), Santa Clara, Calif.
- Pope-Gosser China Co., The, Coshocton, Ohio. Porcelain Tile Co., Chicago, Ill.
- Potters Mining & Milling Co., The, East Liverpool, Ohio. Precision Grinding Wheel Co. (Inc.), Holmesburg, Philadelphia, Pa.
- Quakertown Stove Works, Quakertown, Pa.
- Radiant Glass Co., Fort Smith, Ark. Rawleigh Co., The W. T., Freeport, Ill.
- Republic Stamping & Enameling Co.,
- The, Canton, Ohio. Rock Island Stove Co., Rock Island,
- Ill.
- Rock Products Co., The, Toledo, Ohio. Roessler & Hasslacher Chemical Co.,
- The, New York, N. Y. Rookwood Pottery Co., The, Cincin-
- nati, Ohio.
- Rossman Corporation, Beaver Falls, Pa.
- Rundle Manufacturing Co., Milwaukee, Wis. Salem China Co., The, Salem, Ohio.
- Saxonburg Potteries, Saxonburg, Pa.
- Sharp-Schurtz Co., The, Lancaster, Ohio (in principle).
- Sherwood Bros. Co., New Brighton, Pa. Simonds Worden White Co., Dayton, Ohio.

- Smoot-Holman Co., Inglewood, Calif. Southern Glass Co., Los Angeles, Calif. Southern Potteries (Inc.), Erwin, Tenn. Standard & Specialty Porcelain Works,
- Sun Prairie, Wis. Standard Sanitary Manufacturing Co., Trenton, N. J.
- Stanley Insulating Co., Great Barring-ton, Mass.
- Star Porcelain Co., Trenton, N. J.
- Steel Sanitary Co., Alliance, Ohio.

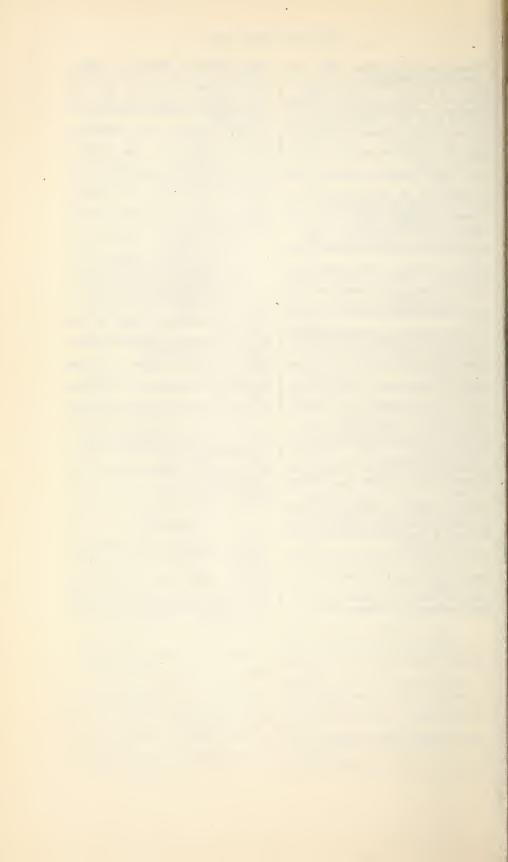
- Sterling Grinding Wheel Co., Tiffin, Ohio.
- Superior Enamel Products Co., St. Louis, Mo. Taylor Smith & Taylor Co., East Liver-
- pool, Ohio. Tidewater Feldspar Co., Higganum,
- Conn. Trenton Potteries Co., The, Trenton,
- N. J.
- Trumbull Electric Manufacturing Co., The, Trenton, N. J. Union Sanitary Works, Noblesville,
- Ind.
- United Feldspar Corporation, New York, N. Y. Oxford M
  - ford Mining & Milling Co., West Paris, Me.

Tennessee Mineral Products Corporation, Spruce Pine, N. C.

- United States Feldspar Corpora-tion, Cranberry Creek, N. Y. Universal Sanitary Manufacturing Co.,
- New Castle, Pa. University of Illinois, Urbana, Ill. (in principle).
- Virginia Polytechnic Institute, Blacks-burg, Va. (in principle).
- Waltham Grinding Wheel Co., Waltham, Mass.
- Washington Porcelain Co., Washington, N. J. Wells Co., The Floyd, Royersford, Pa.
- Western Electric Co. (Inc.), New York, N. Y.
- Western Pottery Co., Stockton, Calif. Westinghouse Electric & Manufactur-
- ing Co., Derry, Pa. Wheeling Sanitary Manufacturing Co.,
- Wheeling, W. Va. Wheeling Tile Co., Wheeling, W. Va. Wolff Co., Chicago, Ill.

#### GOVERNMENT

- Department, Commerce Bureau of Mines, Washington, D. C. (in principle).
- Treasury Department, Washington, D. C
- U. S. Shipping Board Merchant Fleet Corporation, Washington, D. C. (in principle).



# FELDSPAR

# COMMERCIAL STANDARD CS23-30

On January 14, 1930, a general conference of manufacturers, distributors, and users advocated the adoption of a commercial standard for feldspar. A committee was appointed with authority to make certain adjustments in the proposed standard to be referred to the industry for formal acceptance. This has been done and the standard has since been accepted and approved by the industry for promulgation by the Department of Commerce as shown herein. The standard is effective for new production September 1, 1930.

Promulgation recommended.

I. J. FAIRCHILD, Chief, Division of Trade Standards.

Promulgated.

GEORGE K. BURGESS, Director, Bureau of Standards.

APPROVED.

R. P. LAMONT, Secretary of Commerce.

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#### COMMERCIAL STANDARD CS23-30

#### I. SCOPE

This commercial standard classification covers ground feldspar used in the production of ceramic products, based on particle size and chemical composition. It is to be regarded as a classification rather than a definite purchase specification.

#### **II. GENERAL REQUIREMENTS**

All screen tests shall be made on standard screens (U. S. standard sieve series), the opening sizes of which are appended in Table 1. A standard method of screen testing is described on page 4.

A standard method of chemical analysis is described, beginning on page 4.

#### III. DETAIL REQUIREMENTS

#### A. PHYSICAL CLASSIFICATION BASED ON FINENESS OF GRINDINGS1

United States standard sieve series No.	Percentage remaining on No. 200 sieve	Maximum percentage on sieve designated	United States standard sieve series (opening in inches)
230 200 170 140 120	$\begin{array}{c} 0.\ 00-\ 0.\ 35\\ 0.\ 35-\ 1.\ 00\\ 1.\ 00-\ 2.\ 50\\ 2.\ 50-\ 5.\ 00\\ 5.\ 00-\ 9.\ 00 \end{array}$	1.0 1.0 1.0 1.0 1.0 1.0	$\begin{array}{c} 0.\ 0024\\ .\ 0029\\ .\ 0035\\ .\ 0041\\ .\ 0049 \end{array}$
100 80 60 40 20	$\begin{array}{c} 9,00{-}14,00\\ 14,00{-}21,00\\ 21,00{-}30,00\\ 30,00{-}42,00\\ 42,00{-}62,00 \end{array}$	1.0 1.0 0.6 0.3 None	. 0059 . 0070 . 0098 . 0165 . 0331

TABLE 1

<sup>1</sup> Fineness classification shall be made on a basis of the percentage remaining on the standard 200 sieve and that remaining on the sieve designated. Example: 140-sieve product will have 2.5 to 5.0 per cent remaining on the 200 sieve and less than 1 per cent on the 140 sieve.

#### B. CHEMICAL CLASSIFICATION BASED ON COMPOSITION AS IT INFLUENCES USE

The numbers designated herein are for the purpose of illustration and the various groups may be added to, up or down the scale, to provide for all commercial grades of feldspar.

#### GROUP 1

The first group includes the commonly accepted ceramic or body grades based on silica content and alkali ratio and containing less than 4 per cent soda (Na<sub>2</sub>O) content.

The silica number and ratio numbers are to be used in combination. For example: Grade No. 67-51 designates a spar of silica

 $\mathbf{2}$ 

content 66.00 up to 67.99 per cent and with 5 or more parts of potash  $(K_2O)$  to 1 part of soda  $(Na_2O)$ .

Number	Silica (SiO <sub>2</sub> ) content in per cent
65 67 69 71 73	$\begin{array}{c} 64.00{-}65.99\\ 66.00{-}67.99\\ 65.00{-}69.99\\ 70.00{-}71.99\\ 72.00{-}73.99\end{array}$
	Potash (K10)-soda (Na10) ratio.
61 51 41 81	6 or more potash to 1 soda. 5 potash to 1 soda up to 6 potash to 1 soda. More than 3 and less than 5 potash to 1 soda. 3 or less potash to 1 soda.

#### GROUP 2

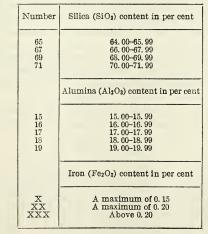
The second group includes the spars used chiefly for glazing purposes which are based on soda content and contain 4 per cent or more soda ( $Na_2O$ ).

Number	Soda (Na <sub>2</sub> O) content in per cent
4	4. 00-4. 99
5	5. 00-5. 99
6	6. 00-6. 99
7	7. 00-7. 99
8	8. 00-8. 99

#### GROUP 3

The third group includes the spars used for glass-making purposes and are based on silica, alumina, and iron content. The numbers are to be used in combination: For example, grade

The numbers are to be used in combination: For example, grade 69-17-X represents a grade of spar of 68.00 to 69.99 per cent silica, 17.00 to 17.99 per cent alumina, and with a maximum of 0.15 per cent Fe<sub>2</sub>O<sub>3</sub> content.



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#### IV. STANDARD METHODS OF TEST

#### A. PHYSICAL TEST

Mesh or fineness.-A 100 g portion of the dry sample is weighed out to an accuracy of 0.1 g. It is then transferred to a number 200 sieve and over a sieve pan which fits closely. The pan shall contain sufficient water to reach within not less than one-fourth inch or more than three-fourths inch from the top of the pan. The sieve and pan shall be vibrated or shaken in such a manner that water in the pan is splashed on the screen from below, so as to wash the powder about and cause the material that can pass through the sieve to pass into the pan below. This treatment shall be continued until no appreciable amount is passing through. The contents of the screen are then washed into a pan and thoroughly dried. The dried material is then placed on the coarsest sieve to be used. The finer screens, including the number 200, are placed in order of size under the coarsest, the number 200 being at the bottom. The tier of screens is then shaken until no more than 0.05 of a gram passes any screen after one minute of shaking each time. This point is determined by weighing the residue on the coarsest screen, reshaking for a minute until finished, then going to the next size and repeating the process.

#### B. CHEMICAL TESTS

#### GENERAL

Feldspar is analyzed for SiO<sub>2</sub>, "R<sub>2</sub>O<sub>3</sub>," Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, K<sub>2</sub>O, Na<sub>2</sub>O, and ignition loss. SiO<sub>2</sub>, "R<sub>2</sub>O<sub>3</sub>," CaO, MgO, K<sub>2</sub>O, and Na<sub>2</sub>O are determined in 0.5 g samples. Ignition loss is determined in a 5 g sample. Fe<sub>2</sub>O<sub>3</sub> is determined potentiometrically in a 10 g sample, and the result is subtracted from the percentage of "R<sub>2</sub>O<sub>3</sub>" to give Al<sub>2</sub>O<sub>3</sub>. The result for the latter will of course include constituents such as TiO<sub>2</sub>, ZrO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub> and the like.

Accurate results in the analysis of feldspar are not easy to obtain, especially in the cases of SiO<sub>2</sub> and " $R_2O_3$ ." Attention must be paid to all details. Variations in check analyses should be within the following limits: SiO<sub>2</sub>, 0.2 per cent; " $R_2O_3$ ," 0.2 per cent; CaO, 0.05 per cent; MgO, 0.05 per cent;  $K_2O$  plus Na<sub>2</sub>O, 0.2 per cent.

Analyses should be repeated if the summation does not fall between 99.75 and 100.5 per cent.

Analyses of potash feldspars should be checked against the Bureau of Standards standard sample No. 70, and analyses of soda feldspars should be checked against the Bureau of Standards standard sample No. 99.

All results should be corrected by blank runs on reagents and factors should be calculated from the current International Table of Atomic Weights.

#### PROCEDURE

Preparation of sample.—The sample should be ground to pass a No. 100 screen and then well mixed. All samples for analysis must be taken from material that has been dried at 105° to 110° C.

Ignition loss.—Weigh a 5 g sample in a 30 ml platinum crucible. Cover the crucible with a well-fitting cover, and heat for 15 minutes at 1,000° C. The heating can be done over a burner or in a muffle, and should be sufficiently gentle at the start so that there is no danger of mechanical loss. Cool over a good desiccant and weigh. The loss in weight multiplied by 20 represents the per cent of ignition loss.  $SiO_2$ , " $R_2O_3$ ," CaO, and MgO.—Weigh a 0.5 g sample in a 30 ml

 $S_{102}$ ,  $R_{203}$ , Ca0, and Mg0.—Weigh a 0.5 g sample in a 30 mi platinum crucible and add 4.0 g of anhydrous sodium carbonate. Mix thoroughly with a small spatula and clean off the spatula. Cover the crucible, gradually heat to about 1,000° C., and continue the heating at this temperature, usually about 15 minutes, until the melt is quiescent.

Remove the cover, grasp the crucible near the top with a pair of platinum-tipped crucible tongs, and gently rotate the crucible so that the melt solidifies in a thin layer on the inside of the crucible. Replace the cover and allow the crucible to cool. When cool, remove the cover, invert the crucible over a 300 ml evaporating dish, and loosen the melt by tapping on the bottom of the crucible.

It is preferable that the evaporating dish be of platinum or of platinum-lined gold. If a porcelain dish is used it must be free from crusted rims on the inside, and especial care must subsequently be taken to remove all silica. If the melt does not loosen readily, reheat and again cool.

Wash the crucible and cover with hot water to remove adhering particles, allowing the water to drain into the dish containing the melt. Add enough hot water to total 100 ml and break up lumps by pressing with a blunt stirring rod. When disintegration is complete, cover the dish with a cover glass and through the lip add 20 ml of concentrated hydrochloric acid. This should be measured in the fusion crucible and introduced carefully so as to avoid loss by too rapid effervescence. Make sure that the crucible and cover are now clean.

Warm the evaporating dish, remove and wash the cover and inside of the dish as soon as effervescence ceases, and evaporate the solution to dryness on the steam bath. Remove the dish from steam bath, cover and drench the residue with 10 ml of concentrated hydrochloric acid. Let stand one to two minutes, add 100 ml of hot water, remove and rinse the cover, and heat on the steam bath for five minutes. Filter through a 11 cm No. 42 Whatman, or equivalent paper, catching the filtrate in a 600 ml Pyrex beaker. Transfer as much of the silica as can be removed by rinsing the dish with hot dilute hydrochloric acid (5:95). There is no need to scrub the dish at this time. Wash the paper and silica with six 10 ml portions of the hot dilute acid and then with ten 10 ml portions of hot water at each washing; the solution should be first directed around the edge of the filter paper and then spirally down in order to churn up the silica. Reserve the paper and silica.

When the washing is finished, transfer the filtrate to the evaporating dish, rinse the beaker, and again evaporate the solution to dryness. When dry, cover, place the dish in a constant temperature oven, and bake for 15 to 30 minutes at 105° to 110° C. Remove the dish from the oven, drench the residue with 5 ml of concentrated hydrochloric acid, and warm on the steam bath for one to two minutes. Add 50 ml of hot water, rinse off the cover glass, and immediately filter on a 9 cm paper. Scrub the dish thoroughly with small portions of cool dilute hydrochloric acid (1:99), each time using the portion to rinse the paper and residue. When all of the silica has been transferred wash the paper and residue with five 10 ml portions of warm water. Reserve the filtrate for the determination of " $R_2O_3$ ," etc.

Determination of SiO<sub>2</sub>.—Remove the filter papers containing the silica from the funnels and place in a weighed clean 20 ml platinum crucible. Heat gently until the papers and contents are dry, increase the heat so that the papers char without flaming, and then burn the carbon at as low a temperature as possible. Cover the crucible with a well-fitting cover and heat at 1,200° C. for 15 minutes. If a blast lamp is used, the crucible should be sunk for two-thirds of its depth in an asbestos shield to prevent loss of the light, fluffy silica by drafts from the blast. Cool over a good desiccant and weigh. Re-peat the heating until constant weight is obtained. Moisten the silica with a few drops of water, add two drops of dilute sulphuric acid (1:1) and then 10 ml of hydrofluoric acid. Carefully evaporate until fumes of sulphuric acid are given off and then still more carefully until the sulphuric acid has been expelled and the sulphates of iron, aluminum, etc., decomposed. Finally heat at about 1,000° C. for about five minutes, cool in a desiccator, and weight. The difference in weights between the crucible with the silica and the crucible after the treatment with the hydrofluoric acid represents the weight of the silica obtained in the two evaporations. A small amount of silica, usually not much more than 1 mg, still remains in solution and is subsequently caught in the " $R_2O_3$ ." In analyses of the highest accuracy this is recovered by fusing the " $R_2O_3$ " with pyrosulphate, dissolving the melt in sulphuric acid, evaporating to fumes, diluting with water and immediately filtering and washing. To obtain the percentage of silica, multiply the weight of silica found by 200.

If the residue left in the crucible after the volatilization of the silica weighs no more than 3 mg and is amorphous rather than glassy (as is the case in proper work) it can be considered as " $R_2O_3$ " and weighed together with the precipitate obtained in the determination of " $R_2O_3$ ." If the residue is heavy, or appears to be fused (alkali salts) the determination of silica is open to question. In order to avoid error in the determination of " $R_2O_3$ " the residue must be fused with 1 g of sodium carbonate, the melt dissolved in dilute acid and added to the filtrate from the silica.

Determination of " $R_2O_3$ ."—Cover the beaker containing the filtrate from the silica determination and heat to boiling. Remove from the hot plate, add two drops of methyl red indicator solution and then add 1:1 ammonium hydroxide dropwise and with stirring until the solution turns distinctly yellow. Return the beaker to the hot plate and boil for one or two minutes. The solution should remain yellow. Filter immediately on a No. 589 S. & S. black ribbon, or similar filter paper, catching the filtrate in another 600 ml Pyrex beaker. Wash the beaker, precipitate and filter paper with two or three small portions of a hot 2 per cent solution of ammonium chloride. Reserve the filtrate. Remove the precipitate from the filter paper with a jet of hot water, washing it into the beaker in which the precipitation was just made. Dissolve the precipitate in 25 ml of hot, dilute hydrochloric acid (1:1). Dilute to 200 ml with hot water, add the filter paper, rinse the funnel with a little acid and water, and stir the solution until the paper is pulped. Repeat the precipitation as before using methyl red and dilute ammonium hydroxide. Filter and wash thoroughly with 200 ml of the hot 2 per cent solution of ammonium chloride. Keep the filter paper nearly full of the wash solution. Make sure that the transfer of the precipitate is complete and combine the filtrate with the reserved filtrate and reserve for the determination of calcium oxide.

Place the paper and precipitate in a weighed platinum crucible (see determination of  $SiO_2$ ) and heat gently over a burner or in an open muffle, until the filter paper has burned without flaming and the residue is perfectly white. Finally heat at 1,200° C. for half an hour. Cool in a desiccator containing concentrated sulphuric acid, and weigh. Ignition of " $R_2O_3$ " at 1,200° C. is very necessary to affect complete dehydration. The cover of the crucible must be tight fitting and the weighing must be made rapidly. Ignited alumina is a more powerful desiccant than calcium chloride. The weight of the ignited residue multiplied by 200 represents the percentage of " $R_2O_3$ " in the sample.

Determination of calcium oxide.—Acidify with hydrochloric acid the combined filtrates reserved in the determination of " $R_2O_3$ " and evaporate to a volume of about 200 ml. Heat to boiling, make slightly ammoniacal and add dropwise while still boiling, 10 ml of a saturated solution of ammonium oxalate. Remove the beaker from the hot plate and digest on the water bath for one-half hour. Remove and allow to stand for one hour, or until cool. When the precipitate has settled, filter and transfer all of the precipitate by scrubbing and the use of a 0.1 per cent solution of ammonium oxalate. Finally wash the paper from the rim downward with ten 10 ml portions of the wash solution. Reserve the filtrate for the determination of magnesia. Place the paper and precipitate in a weighed platinum crucible and ignite carefully until carbon has been destroyed. Cover with a well-fitting cover, ignite at about 1,000° C., cool over a good desiccant and weigh. The weight multiplied by 200 represents the percentage of CaO in the sample.

Determination of magnesia.—The volume of the reserved solution should not exceed 300 ml. Render barely acid with hydrochloric acid and add 0.5 g of diammonium hydrogen phosphate. Make slightly alkaline with ammonium hydroxide and then add one-ninth the total volume in excess. The beaker may now be placed in an ice bath or stream of cold water which tends to hasten the precipitation. A minimum of four hours is required for the complete precipitation. If time permits, the solution should be allowed to stand over night.

Filter, transfer the precipitate to the paper, scrub the beaker until free from precipitate, and then wash the precipitate ten times with 10 ml portions of dilute ammonium hydroxide (5:95). Place paper and precipitate in a weighed platinum crucible and dry in a drying oven or over a small flame. Char the paper without flaming, burn the carbon at as low a temperature as possible, and then ignite at 1,100° C. until constant weight is obtained. The weight of the ignited residue multiplied by 72.42 represents the percentage of magnesia in the sample.

Determination of alkalies ( $K_2O$  and  $Na_2O$ ).—Transfer 0.5 g of the sample to an agate mortar, add 0.5 g of ammonium chloride and grind the mixture until thoroughly mixed, taking care to avoid mechanical loss by dusting or otherwise. Add 3 g of precipitated calcium carbonate (for alkali determinations) and mix thoroughly. Transfer to a glazed paper and from this to a 25 ml crucible (a J. L. Smith crucible is better). Clean the mortar, pestle, and paper with 1 g of the calcium carbonate and add to the contents of the crucible. Cover the crucible with a snugly fitting cover and place it in a hole in a one-eighth inch thick asbestos board so that a little less than half of the crucible sticks through. Heat the bottom of the crucible so gently that the odor of ammonia is just distinguishable. Gradually raise the heat until ammonia can not be smelled, then sinter for one hour at about 1,050° C. During this period it is desirable to put a small beaker of water on the crucible cover. Cool, transfer the sintered cake to a 250 ml casserole, slake, and add 100 ml of hot water, nearly fill the crucible with hot water, warm, and add the solution to the solution in the casserole. Let settle somewhat, and filter by decantation upon a 9 cm filter and catch the filtrate in a 1,000 ml casserole. Keep as much as possible of the residue in the dish. Add 50 ml more of hot water, break up lumps by gentle pressure with a pestle, let settle, and again decant. Repeat the extraction six times.

To the filtrate, of approximately 500 ml volume, add 20 ml of a saturated solution of ammonium carbonate, cover, and heat on a steam bath. Add small portions of the ammonium carbonate solution until it is judged that no more calcium can be precipitated. Heat until effervescence ceases and simmer carefully to expel carbon dioxide. Remove and wash the cover and filter into a 1,000 ml casserole and wash casserole, paper, and residue with twenty 10 ml portions of hot water. Reserve the filtrate. Dissolve the precipitate in as little dilute hydrochloric acid as possible, reprecipitate with ammonium carbonate as before, filter, and add the filtrate to the reserved filtrate. Evaporate the combined filtrates to dryness and heat gently to remove ammonium salts. Dissolve the residue in 25 ml of water, add a few drops of ammonium hydroxide, heat to boiling, add ammonium oxalate until in excess (about five drops in excess) and let stand over night. Filter through a 5.5 cm filter into a 75 ml platinum dish, wash moderately with a 0.1 per cent solution of ammonium oxalate. Evaporate to dryness, cover the dish, and heat at 110° C. Remove the cover, and cautiously ignite until ammonium salts have been volatilized. Cool, dissolve the residue in a little water, filter on a small paper, and catch the solution in a small vessel. Wash the paper with small portions of hot water and add a few drops of hydrochloric acid to the filtrate. Transfer the solution to a weighed 75 ml platinum dish, evaporate the solution to dryness, cover, and heat at 110° C. Ignite at faint dull red heat-not over 500° C., cool in a desiccator, and weigh as NaCl plus KCl. Add 20 ml of hot water, a drop of hydrochloric acid and 1 to 2 ml of perchloric acid, specific gravity 1.54. Evaporate to dryness at a tem-perature not over 350° C., cool, add 1 ml of perchloric acid and repeat the treatment. Cool, add 20 ml of absolute ethyl alcohol con-taining 0.2 per cent of perchloric acid, specific gravity 1.54. Break up the residue, let stand 15 minutes, and filter the clear solution through a tared Gooch crucible. Wash the container and residue once or twice with absolute ethyl alcohol containing 0.2 per cent of perchloric acid. Suck dry as possible and dissolve the residue in water. Add 1 ml of perchloric acid, dry, and extract as before. Filter, transfer the residue to the Gooch crucible (which has been washed once or twice with absolute alcohol to remove the water) by

means of not over 100 ml of absolute ethyl alcohol containing 0.2 per cent of perchloric acid and saturated shortly before use by shaking with crystals of potassium perchlorate for 5 to 10 minutes at room temperature, letting settle and pouring off the clear solution. Suck dry, heat at 120° to 130° C., cool, and weigh as  $KClO_4$ .

Calculate the weight of  $K_2O$  and multiply by 200 to obtain the percentage of  $K_2O$ .

<sup>1</sup> Calculate the weight of KCl, subtract from the weight of KCl plus NaCl, calculate the weight of Na<sub>2</sub>O and multiply by 200 to obtain the percentage of Na<sub>2</sub>O.

Blank determinations must be carried through all steps of the method and proper corrections made for any alkalies found.

Determination of iron.-Weight a 10 g sample of the feldspar to be analyzed using counterbalanced watch glasses. Transfer the sample to a platinum evaporating dish of 250 ml capacity. Moisten the contents of the dish with distilled water and then add 5 ml of concentrated sulphuric acid. Stir until all of the sample is wet by the This may be done by rotating the dish. Cautiously add liquid. hydrofluoric acid until the dish is two-thirds filled. Spattering of the solution when the HF is added can be avoided by first igniting the sample for a short time. Place the evaporating dish on a sand bath or on a hot plate and evaporate to apparent dryness or until fuming of the sulphuric acid takes place. The last part of the evap-oration should be done carefully to avoid spattering. Remove the dish from the hot plate and drive off most of the remaining sulphuric acid over a low flame or in a muffle furnace which is not so hot as to cause loss by spattering. The residue should now be in a dry white cake. When cool, add 25 ml of dilute hydrochloric acid 1:1 and digest on the hot plate until the residue has been entirely disintegrated. Remove and dilute with hot distilled water. Allow to settle for a few minutes and filter through a 11 cm paper, catching the filtrate in a 500 ml beaker. Rinse the dish and filter paper with small portions of hot water. Return the filter paper containing the residue to the platinum dish, dry, burn the filter paper, and ignite until the residue is white. Break up the residue with a lump of potassium pyrosulphate and add enough additional pyrosulphate to fuse the residue. This is five to ten times the weight of residue and may be from 15 to 30 g depending on the weight of the material to be treated. The fusion should be started at a low temperature as some effervesence will nearly always take place. The melt should be kept just liquid by adjustment of the burner flame. When the melt is clear or no particles of the residue remain (30 minutes to 1 hour) remove from the flame and cool. Dissolve the melt by digesting with dilute hydrochloric acid (1:4) on the hot plate. Filter, and catch the filtrate in the same beaker that was used for the first filtration. Ignite the filter paper in a small platinum crucible and if any residue remains fuse it with 1 g of anhydrous sodium carbonate until the melt is clear. Dissolve the melt in dilute hydrochloric acid (1:1) and add to the main portion of the solution in the beaker. Neutralize the solution with a dilute solution of sodium hydroxide (20 per cent) and then make slightly acid with dilute sulphuric acid (1:1). Thoroughly saturate the solution with hydrogen sulphide and let stand over night. Filter, and wash the filter thoroughly

with warm water. Boil to remove the excess of hydrogen sulphide and to reduce the volume to 100 ml. Add dilute permanganate until the solution turns pink and transfer the solution to a 500 ml Erlenmeyer flask. Add concentrated hydrochloric acid to the flask until the solution is approximately 1:1 with respect to the acid. Heat the solution to boiling, remove and reduce the hot solution with a minimum number of drops of stannous chloride solution. Adjust the titration head and proceed with the potentiometric titration of the iron, using a standard solution of potassium dichromate.

The titration must be corrected for iron contained in the reagents, carrying them through all the manipulations of the procedure. If the analyst is careful to perform all the operations in a standard manner as to time and temperature the blank will be constant.

The final volume of the solution should be kept as small as possible in order to obtain sharp end points. In the case of the blank determination, in which the titre should be in the region of 2 or 3 ml of standard solution, the volume of the solution should not greatly exceed 50 ml. With feldspar the volume should be about 200 ml.

The concentration of the solution with respect to hydrochloric acid is important as it has an effect on the sensitivity of the end point. The titration must be carried out with speed. This aids in sharp end points and lessens the chance of oxidation of iron by air in the flask.

For details concerning the potentiometric titration of iron see the Bulletin of the American Ceramic Society, vol. 6, No. 4, page 100 (April, 1927), and the Journal of the American Ceramic Society, vol. 11, No. 6, page 370 (June, 1928).

# GENERAL CONFERENCE

Pursuant to a request from the Feldspar Grinders' Institute, a general conference of feldspar producers, distributors, and consumers was held at the National Bureau of Standards, Washington, D. C., on January 14, 1930, to consider the approval of commercial standard classifications for feldspar as developed by a committee of the above institute.

At this conference some differences of opinion were indicated regarding the commercial standard as presented, but those in attendance reiterated their desire for a standard classification for the product as a common basis of understanding for the entire industry. A committee representative of all interests was, therefore, appointed to complete the formulation of a commercial standard on the basis of that submitted by the Feldspar Grinders' Institute which was later to be directly submitted to the industry for formal approval.

The committee met in Toronto, Canada, on February 18, 1930, and decided upon a recommended commercial standard classification, which, at their request, was submitted to the chemistry division of the National Bureau of Standards for correlation and adjustment of the methods covering chemical analysis.

The recommended standard was then circulated to the entire industry and enthusiastically accepted by all interests as indicated by the 198 acceptances already recorded. Those in attendance at the general conference January 14 are listed herewith.

ADAMS, WILLIAM L., JR., manager, Oxford Mining & Milling Co. AMBERG, CHARLES R., professor ceramic engineering, Alfred University. BARRINGER, L. E., engineer of insulations, General Electric Co. BOWLES, OLIVER, supervising engineer, Bureau of Mines, and American Institute of Mining and Metallurgical Engineers. BROCKMAN, E. D., The Roessler & Hasslacher Chemical Co. BROWNAN, B. D., The incession of massacher chemical co. BROWN, GEORGE H., director, department of ceramics, Rutgers University. BURGESS, B. C., manager, Tennessee Mineral Products Co. EARNILL, T. C., director, research and development, Owens-Illinois Glass Co. FLINT, F. C., chief chemist, Hazel Atlas Glass Co. FRINK, R. L., consulting engineer, Lancaster, Ohio. GOLDING, CHARLES E., president, Standard Flint & Spar Corporation. FULLER, D. H., ceramic engineer. HADLEY, J. M., secretary, standardization division, American Mining Congress. HAMILTON, JAMES, general superintendent, Trenton Potteries Co. HAMILTON, JAMES, general superintendent, Trenton Fottenes Co. HANNA, HENRY N., sales executive, Seaboard Feldspar Co. HERR, HORACE H., "Ceramic Industry." HILL, G. C., superintendent, Locke Insulator Corporation. KELSEY, V. V., sales manager, Consolidated Feldspar Corporation. KNIGHT, FRANK P., JR., chief chemist, United Feldspar Corporation. KOENIG, E. W., chief chemist, Consolidated Feldspar Corporation. KOENIG, E. W., chief chemist, Consolidated Feldspar Corporation. LAWSON, ROBERT W., vice president, Consolidated Feldspar Corporation. MALSCH, W., manager ceramic dept., The Roessler & Hasslacher Chemical Co. MANOR, J. M., vice president and general manager, The Golding Sons Co. MARGERUM, H. P., president, Erwin Feldspar Co. MCLEAR, H. G., vice president, Green Hill Mining Co. (Inc.). PARKER, W. J., commissioner, Feldspar Grinders' Institute. PARKELE, C. W., head, department ceramic engineering, University of Illinois. PEDDRICK, C. H., Jr., vice president, United Feldspar Corporation. PORST, E. G., chemist, Porcelain Enamel & Manufacturing Co. RICHARD, HARRY A., salesman, The Roessler & Hasslacher Chemical Co. RODGERS, JOSEPH P., secretary-treasurer, Seaboard Feldspar Co. RODGERS, JOSEPH P., secretary-treasurer, Seaboard Feldspar Co. RODGERS, JOSEPH P., secretary-treasurer, Seaboard Feldspar Co.
SCHRAMM, EDWARD, chemist, Onondaga Pottery Co.
SCHURECHT, H. G., research associate, National Terra Cotta Society.
SHEARER, WALTER L., chemist, Rouse & Shearer (Inc.).
THROPP, HERRELD D., vice president, Eureka Flint & Spar Co.
TURNER, JAMES, treasurer, Trenton Flint & Spar Co.
WAINFORD, R. H., president, Trenton Flint & Spar Co.
WALKER, MYRON R., U. S. Tariff Commission.
WATTS, ARTHUR S., professor ceramic engineering, Ohio State University.
WEIS, J. H., manager, United States Feldspar Corporation.
WILKES, JOHN C., general manager, Eureka Flint & Spar Co. (Inc.).
DEPARTMENT OF COMMERCE:
BATES, P. H., chief, division of clay and silicate products, National Bureau of Standards.

of Standards.

HARRISON, W. N., chief, enamel section, National Bureau of Standards. JUDSON, L. V., chief, length section, National Bureau of Standards. SLIGH, W. H., assistant physicist, National Bureau of Standards. STEDLE, HARRY H., division of trade standards, National Bureau of Standards.

WRAY, G. W., junior scientist, National Bureau of Standards.

# EFFECTIVE DATE

The effective date for new production under the commercial standard classification was fixed at three months after announcement of the success of the project, which establishes the effective date as September 1, 1930.

#### STANDING COMMITTEE

Because the standardization committee as appointed at the January 14 conference was thought to be truly representative of the entire industry this committee was continued as the standing committee to consider annually any comment or suggestion relative to the need of revising the standard to keep it abreast of the current practices.

The personnel of this committee is as follows:

B. C. BURGESS, chairman, Tennessee Mineral Products Co.; feldspar grinders. V. V. KELSEY, Consolidated Feldspar Corporation; feldspar grinders.

JOSEPH P. RODGERS, Seaboard Feldspar Co.; feldspar grinders.

JOSEPH F. RODGERS, Beaboard Feduspar Co., Icluspar grinders. ARTHUR S. WATTS, Ohio State University; ceramic schools. P. H. BATES, elay and silicate products division; Bureau of Standards. JAMES HAMILTON, Trenton Potteries Co.; whiteware manufacturers. EDW. SCHRAMM, Onondaga Pottery Co.; whiteware manufacturers. L. E. BAREINGER, General Electric Co.; whiteware manufacturers.

F. C. FLINT, Hazel-Atlas Glass Co.; glass manufacturers.

T. C. EARNILL, Owens-Illinois Glass Co.; glass manufacturers. H. C. WOLFRAM, Porcelain Enamel & Manufacturing Co.; enamel manufacturers. HARRY H. STEIDLE, ex-officio secretary, division of trade standards; Bureau of

Standards.

#### COMMERCIAL STANDARDS SERVICE

Industry has long sensed the need for a wider application and use of specifications developed and approved by nationally recognized organizations. To assist these bodies and the producers and consumers in securing this result and as a natural outgrowth of the movement toward elimination of waste through simplified practice, the National Bureau of Standards has set up a procedure under which specifications, properly indorsed, may be printed as official publications of the Department of Commerce and promulgated as "Commercial Standards." This service parallels that of simplified practice in many respects, and is available only upon request.

Broadly speaking, the aim is to continue the same character of cooperative service in this field that is being rendered in simplification. The division of trade standards is not designed to act as a standardizing body, nor will it engage in the preparation of specifications. Its service is mainly promotional in character, since its chief mission is to get behind a standard or a specification which any branch of industry may want to promulgate on a nation-wide basis; to determine its eligibility for promulgation; to publish and broadcast it in the event the prerequisites of procedure have been met, including a satisfactory majority acceptance; to facilitate the application of the certification plan for the assurance and convenience of the purchaser; to provide means for periodic audits of adherence; and to cooperate with the Bureau of Foreign and Domestic Commerce in determining the desire of industry relative to translation and promulgation of such specifications as a basis for foreign commerce.

In general, it may be said that a simplification covers types, sizes, and varieties of a commodity which are retained by industry on the basis of demand, whereas a commercial standard establishes definite requirements as to grade, quality, or dimensional tolerances in addition to any limitation of variety desired and accepted by the industry.

# ORGANIZATION AND DUTIES OF STANDING COMMITTEE

In order to carry on the aims and desires of the industry in the standardization of their product, a standing committee is appointed at the general conference. This committee consists of members from each division of the industry, namely, producers, distributors, and consumers, and thus reflects the well-balanced viewpoint of all concerned.

The members of the committee receive all suggestions regarding the commercial standard and consider its revision in the event that such action is desirable and mutually beneficial.

If the commercial standard does not warrant revision, it is reaffirmed in its existing form, but if any important changes are found desirable, their adoption is recommended by the committee, whereupon the industry is again solicited for written acceptance of the standard in its revised form.

The committee is in effect a centralizing agency for criticisms and comments regarding the commercial standard and is charged with the responsibility of recommending revisions to keep the standard abreast with current industrial practice.

The proper functioning of the committee requires that, when necessary, its members be willing to attend meetings held at some central place, although in many cases it will be possible to conduct the work by correspondence.

When any deceptions in reference to the commercial standard are reported to the standing committee, it applies moral suasion or such other corrective measures as seem desirable. The Department of Commerce has no "police power" to compel adherence; therefore, it is incumbent upon the standing committee to do all in its power to encourage all divisions of the industry to follow the provisions of the commercial standard and contribute in every way possible to its general adoption and usefulness.

## YOUR COOPERATION

As a producer, distributor, or consumer of some of the commodities for which commercial standards have already been established, you are in a position to avail yourself of the benefits arising from the use of quality standards and incidentally to add impetus to this method of eliminating waste.

The first step is a declaration in favor of the standard by recording your intention to adhere, as closely as circumstances will allow, to the standards for those products which you may buy or sell.

The receipt of your signed acceptance will permit the listing of your company in new editions of the commercial standards that you accept.

You will, of course, want to examine any commercial standards before signing a formal acceptance. The National Bureau of Standards will, therefore, furnish a copy of any standard under consideration for acceptance. To facilitate this procedure, a list appears on page 18 that may be checked and mailed to the Division of Trade Standards, National Bureau of Standards, Washington, D. C. The publications may also be secured singly or in quantities at a nominal price from the Government Printing Office. Prices will be furnished upon request. The acceptance of a commercial standard is an entirely voluntary action and applies to the production, sale, and use of stock items. It is not meant to interfere with the manufacture or sale of special sizes and types sometimes required.

Trade associations and individual companies often distribute large numbers of the printed standard for the information and guidance of their members or customers. In such cases it is possible to extend the scope and degree of adherence by urging each recipient to send in an acceptance, bearing in mind that the practical value of any standardization is measured by the observance it receives.

An acceptance form for the commercial standard herein covered is included on page 15.

# ACCEPTANCE OF COMMERCIAL STANDARD

Please sign and return this sheet to Division of Trade Standards, Bureau of Standards, Washington, D. C.

Date \_\_\_\_\_

DIVISION OF TRADE STANDARDS, NATIONAL BUREAU OF STANDARDS,

Washington, D. C.

GENTLEMEN: We, the undersigned, do hereby accept the original draft of the commercial standard, as our standard

practice in the  $\begin{cases} \text{production}^{1} \\ \text{distribution}^{1} \\ \text{use}^{1} \end{cases}$  of feldspar, beginning \_\_\_\_\_

(Date) its general adoption.

To permit intelligent review of the effectiveness of the commercial standard every year by an accredited committee of all interests, working in cooperation with the Department of Commerce, we plan to supply all data, upon request, which may be necessary for the development of constructive revisions. It is understood that any suggested modifications will be submitted as soon as formulated, and shall not be promulgated until accepted in form similar to this recommendation.

Signature
(Kindly typewrite or print the following lines)
Title
Company
Street address
City and State We are members of the following associations or other organizations interested in the production, sale, or use of feldspar:

<sup>1</sup> Please designate which group you represent by drawing lines through the other two. In the case of related interests, trade papers, colleges, etc., desiring to record their general approval the words "in principle" should be added after the signature.

(Cut on this line)

#### TO THE ACCEPTOR

In signing the acceptance blank, please bear the following points clearly in mind:

1. Adherence.—The Department of Commerce has no regulatory powers to enforce adherence to the commercial standards. Instead, this wate-elimination program is based on voluntary cooperation and self-government in industry. To make this specific standardization operate as a satisfactory example of self-government, it is highly desirable that it be kept distinct from any plan or method of governmental regulation or control. It will be successful according to the degree to which manufacturers, distributors, and purchasers adhere to its terms and conditions.

2. The industry's responsibility.—The department cooperates only on the request of the industry and assumes no responsibility for industrial acceptance or adherence. This program was developed by the industry on its own initiative. Its success depends wholly on the active cooperation of those concerned.

3. The acceptor's responsibility.—You are entering into an entirely voluntary arrangement, whereby the members of the industry—the distributors and consumers of the product, and others concerned hope to secure the benefits inherent in commercial standardization. Those responsible for this standard realize that instances may occur in which it will be necessary to supply or purchase items not included therein. The purpose is, however, to secure wider support for nationally recognized standards covering grade, quality, and other characteristics of products. Consumers can make the program a success if, in their purchasing, they will make a definite and conscientious effort to specify in terms of this commercial standard.

4. The department's responsibility.—The function performed by the Department of Commerce is fourfold: First, to act as a referee to insure adequate consideration of the needs of all interests; second, to supply such assistance and advice in the development of this program as past experience with similar programs may suggest; third, to solicit and record the extent of adoption and adherence to the standard; and fourth, to add all possible prestige to this standardization movement by publication and promulgation if and when it is adopted and accepted by all elements directly concerned.

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# REQUEST FOR COMMERCIAL STANDARDS

Date\_\_\_\_\_

Division of Trade Standards, National Bureau of Standards,

NATIONAL BUREAU OF STANDARDS, Washington, D. C.

GENTLEMEN: The undersigned wishes to examine the commercial standards checked on the reverse side of this page, with a view toward accepting them as our standard of practice in the production, distribution, or consumption of the standardized lines.

(Kindly typewrite or print the following lines)	· <b></b>
Title	· <b></b>
Company	<b></b>
Street address	·
City and State	

# COMMERCIAL STANDARDS

 $\bigcirc$ 

#### CS. No.

# 0-30. The commercial standards service and its value to business. 1-28. Clinical thermometers. 2-30. Mopsticks (in preparation). 3-28. Stoddard solvent. 4-29. Staple porcelain (all-clay) plumbing fixtures. 5-29. Steel pipe nipples. 6-29. Wrought-iron pipe nipples. 7-29. Standard weight malleable iron or steel screwed unions. 8-30. Plain and thread plug and ring gage blanks. 9-29. Builders' template hardware. 10-29. Regain of mercerized cotton yarns. 12-29. Domestic and industrial fuel oils. 13-30. Dress patterns. 0-30. The commercial standards service and its

Item

- - 18

CS. No.

- CS. No. Item
  14-30. Boys' blouses, button-on waists, shirts, and junior shirts (in preparation).
  15-29. Men's pajamas.
  16-29. Wallpaper.
  17-30. Diamond core drill fittings.
  18-29. Hickory golf shafts.
  19-30. Foundry patterns of wood.
  20-30. Staple vitreous china plumbing fixtures.
  21-30. Interchangeable ground glass joints.
  22-30. Builders' hardware (nontemplate).
  23-30. Feldspar.
  24-30. Standard screw threads.
  25-30. Special screw threads.

Item

- 25-30. Special screw threads. 26-30. Aromatic red-cedar closet lining (in prep-
- aration).
- 27-30. Plate-glass mirrors (in preparation).