jury to the glossy surface. The gloss appears to be imparted to the surface by the steam treatment causing a partial solution and partial melting of the surface layer of the soap, whereby minor surface irregularities are evened out. The invention further consists in apparatus suitable for carrying out the process in a preferred manner, consisting of a steam chamber, a hot-air tunnel and an endless conveyor belt, passing over pulleys at each end and driven by a geared motor, the arrangement being such that the hot air may reach the tablet as quickly as possible after the latter has passed through the steaming zone. *(Perfumery and Essential Oil Record.)*

COLD-FORMED SOAP. Hermann Plauson. *German 696,733.* Addn. to *Ger. 693,241.* The fat acids, rosin, and talloil are dispersed in the required amt. of water in a colloidal mill or similar apparatus without the use of dispersants. It is then saponified in the usual kneading machines with concd. caustic.

STABILIZING AGENT FOR OXYGEN-CONTG. PER-COMPOUNDS. Otto Lind and Herbert Colonius (Procter and Gamble Co.). *U. S. 2,254,434.* As an agent for stabilizing and controlling the flow of oxygen from oxygen-contg. per-compounds (in a bleaching detergent), the combination comprising water-soluble salts of amino acetic acid, having at least two carboxyl groups in the alpha positions for each basic nitrogen atom present and magnesium silicate, said water-soluble salts being present in excess of the quantity of said magnesium silicate.

GERMICIDAL SOAP. Walter Hartung (Sharp and Dohme, Inc.). *U. S. 2,251,934.* A germicidal and detergent soap composition exhibiting effective germicidal activity and possessing effective detergent and washing properties, which composition comprises an alkali metal soap of at least one saturated fatty acid and from about 1-10%, based on the soap, of an alkyl phenol capable of acting as a germicide, which soap is free from soaps of unsaturated fatty acids and substantially free from unsaponifiable and unsaponified material other than phenol. *U. S. 2,251,935* relates to saturated fatty acids reacted with caustic and mixed with phenol to form a germicidal soap with good detergent properties.

Report of the Soap Analysis Committee—1941

A year ago our Committee reported on some studies in the determination of tetra sodium pyrophosphate in soap. In that work a sample of soap powder containing about 12 per cent of pyrophosphate in addition to appreciable percentages of sodium silicate and sodium carbonate was analyzed using two methods: (1) a gravimetric procedure in which an aqueous solution of the alcohol insoluble was treated with a solution of zinc acetate, after adjusting to a pH of 3.5. The precipitate of zinc pyrophosphate was then washed, ignited and weighed; (2) by conversion of the pyrophosphate to the ortho salt by acid treatment and determination of the P_2O_5 by precipitation and weighing according to the official A.O.C.S. procedure. As a result of this work, the Committee decided at that time not to recommend adoption of either method but outlined some further studies to be carried out on the same sample of soap using a modified procedure which, in brief, was as follows:

1. Obtain alcohol insoluble in usual manner.
2. Dissolve alcohol insoluble in water and adjust to pH of 3.8 using glass electrode.
3. Add a measured excess of zinc sulphate solution.
4. Titrate liberated sulphuric acid with standard alkali, again using the glass electrode in determining the endpoint.
5. Calibrate the standardized alkali against recrystallized and dried tetra sodium pyrophosphate using the glass electrode in the same manner as in the actual determination.

During the present year eleven laboratories collaborated on this method, the results ranging between 11.69 per cent and 12.20 per cent or an average of 11.93 per cent tetra sodium pyrophosphate. The results are in close agreement showing a variation of only 0.51 per cent between laboratories reporting. The endpoint using the glass electrode appears to be much superior to that obtained with the procedure used last year in which a pH color indicator was used. Compilation of results is given in Table I.

Conclusions

The Committee at their meeting held in Chicago on October 7, 1941, agreed to recommend tentative adoption of the volumetric procedure using a glass electrode. Incidentally, the method outlined and recommended for adoption follows substantially that adopted by the A.S.T.M. for evaluation of commercial tetra sodium pyrophosphate.

1941 A. O. C. S. Soap Committee Cooperative Results

TABLE I

Sample for Determination of $Na_4P_2O_7$

Collaborator	Moisture	% $Na_4P_2O_7$ as received	% $Na_4P_2O_7$ calc. to 5% moisture
Armour & Co. 31st St. Aux.	3.83% 3.81%	12.30% 12.39% 12.35%	12.15% 12.24% 12.20%
	Ave. 3.82%	12.35%	12.20%
U. S. Dept. of Commerce Nat'l Bureau of Standards	3.71%		12.00%
Fels & Co.	3.46%	12.05%	11.88%
Foster D. Snell, Inc.			11.94%
Hercules Experimental Station	3.4% 3.4	11.9% 11.9	11.69% 11.69
	Ave. 3.4%	11.9%	11.69%
Hooker Electro-chemical Co.	3.63%	11.99%	11.83%
Lever Bros. Co.	3.59%	12.20%	12.02%
Los Angeles Soap Co.	3.31% 3.41	12.19% 12.11	11.98% 11.90
	Ave. 3.36%	12.15%	11.94%
Procter & Gamble	2.96%	*12.02%	11.77%
Swift & Co.	3.15%	....	11.90%
Allen B. Wrisley Distributing Co.	3.71%	12.22%	12.05%
			Ave. 11.93%
			High 12.20
			Low 11.69

* Caustic solution was standardized against constant boiling HCl instead of C. P. $Na_4P_2O_7$.

The method of analysis depends upon an indirect determination by the titration of the H_2SO_4 liberated by the action of zinc sulphate on an acid pyrophosphate. The analysis for pyrophosphate is inaccurate in the presence of polyphosphates. The procedure followed in the Committee's studies is shown in detail below:

Determination of Tetra Sodium Pyrophosphate ($Na_4P_2O_7$) in Soap Products

Reagents Required:

1. Neutral Ethyl Alcohol (94 per cent or higher). Denatured Alcohol formula No. 30 or 3-A may be used.
2. N/2 HCl.
3. N/10 H_2SO_4.
4. Zinc Sulphate, 7 per cent solution. Dissolve 125 gm. of C. P. $ZnSO_4 \cdot 7H_2O$ in water and make up to a liter, filter and adjust the pH to exactly 3.8 with the glass electrode using N/10 H_2SO_4.
5. N/2 NaOH. (See procedure for standardization.)

Procedure: Accurately weigh a sample containing an equivalent of approximately 1 gm. of $Na_4P_2O_7$ and digest with 200 c.c. of hot, freshly boiled, neutral alcohol. Filter through a filter paper or Gooch crucible with suction washing with hot alcohol until free from soap. Change receivers and wash the alcohol insoluble portion remaining on the filter paper with hot water until washings are neutral to phenolphathalein. Transfer to a suitable vessel, cool, and add sufficient distilled water so that the resulting solution will just cover the electrodes of a glass electrode titration assembly. Adjust the pH of the solution to exactly 3.8 with N/2 HCl. Add 50 c.c. of the 7 per cent zinc sulphate solution and allow 5 minutes for this reaction to become complete as shown by the pH becoming constant. Titrate the liberated acid with N/2 NaOH until a pH of 3.8 is again reached. This titration is a measure of the pyrophosphate content. Standardize the N/2 NaOH against C. P. $Na_4P_2O_7$ prepared by recrystallizing the technical product three times from water and igniting at 400° C. to constant weight.

Calculations

$$\frac{\text{c.c. NaOH} \times F \times 100}{\text{Wt. of Sample}} = \text{per cent } Na_4P_2O_7$$

where F = grams of $Na_4P_2O_7$ equivalent to 1 c.c. of the NaOH solution used for titration as calculated by standardization against the C. P. $Na_4P_2O_7$.

Free Caustic Alkali and Carbonate in Potash Paste Soaps

For some time the Committee has recognized that the present official method for free KOH and K_2CO_3 in potash paste soaps gives erroneous results and has initiated several collaborative studies in an effort to work out an improved procedure. None of these studies resulted in enough improvement to justify making any change. Another attempt has been made during the past year to work out a satisfactory method, this time at the suggestion of one of the member laboratories who had recommended the use of isopropyl alcohol instead of ethyl alcohol as a solvent, the method otherwise being substantially identical to the present official procedure.

Ten laboratories collaborated in the tests in this procedure using three samples of soap, one of which contained relatively low percentages of free caustic alkali as KOH and carbonate as K_2CO_3, a second which contained added free carbonate and a third, an added amount of free caustic. The results are given in Tables II, III, and IV.

As a matter of record, the procedures followed in testing the three cooperative potash soap samples are shown below:

I—Free Caustic (as KOH) and carbonates (as K_2CO_3) were determined by the following methods, using freshly boiled, neutral, ethyl alcohol Formula No. 30 or 3-A may be used).

a) Dissolve the sample in 200 c.c. of freshly boiled, neutral, ethyl alcohol and as soon as solution is complete and before filtering, titrate the free caustic with standard acid to the phenolphthalein endpoint and calculate as free KOH. After titrating the free alkali, filter, wash the filter paper with hot, neutral alcohol until free from soap, then dissolve the alcohol insoluble in hot water, titrate with standard acid to the methyl orange endpoint and calculate to K_2CO_3.

TABLE II

Sample A—Regular Soft Soap

Collaborator	Method Ia — Ethyl alcohol —Caustic before filtration	Method Ib — Ethyl alcohol —Caustic after filtration	Method IIa — Isopropyl alcohol —Caustic before filtration	Method IIb — Isopropyl alcohol —Caustic after filtration	Carb. as K_2CO_3 by evolution volumetric method
Armour & Co. 31st St. Aux.	Caus. 0.07% Carb. 0.10	0.03% 0.10	0.07% 0.14	0.02% 0.23	.... 0.13%
U. S. Dept. of Commerce, Nat'l Bureau of St'rds	Caus. 0.05% Carb. 0.10	Acid 0.12%	0.04% 0.13	Acid 0.19%	....
Fels & Co.	Caus. 0.035% Carb. 0.045	0.014% 0.064	0.024% 0.107	0.000% 0.128	....
Hercules Exp'tal Station	Caus. 0.033% Carb. 0.10	0.00% 0.13	0.045% 0.20	0.00% 0.17	....
Hooker Electro-Chemical Co.	Caus. 0.07% Carb. 0.04	0.03% 0.05	0.03% 0.08	Trace 0.14%	....
Lever Bros. Co.	Caus. 0.08% Carb. 0.04	0.04% 0.09	0.06% 0.12	0.01% 0.16	....
Los Angeles Soap Co.	Caus. 0.060% Carb. 0.065	0.020% 0.080	0.045% 0.115	0.000% 0.170	....
New York Produce Exchange	Caus. 0.040% Carb. 0.032	0.026% 0.029	*0.046% 0.069	*0.011% 0.115	....
Procter & Gamble	Caus. 0.031% Carb. 0.142	0.000% 0.177	0.010% 0.212	0.000% 0.212	....
**(Special Method)	Caus. 0.010% Carb. 0.115	0.000% 0.124	....	....	....
Swift & Co.	Caus. 0.067% Carb. 0.11	0.022% 0.13	0.062% 0.166	0.000% 0.221	....

* 99% Isopropyl Alcohol was used.

** Special Method based on the addition of 10 gms. salt to 20 gms. soap, mixing, dissolving in 200 c.c. of 95% alcohol and determining caustic in the alcohol soluble portion. K_2CO_3 determined by evolution.

b) Dissolve the sample in 200 c.c. of freshly boiled, neutral, ethyl alcohol, and, as soon as solution is complete, filter, washing the filter paper with neutral alcohol until free from soap. Combine the filtrate and washings, heat to incipient boiling and titrate the free alkali with standard acid to the phenolphthalein endpoint and calculate as free KOH. Dissolve the alcohol insoluble portion remaining on the filter paper in hot water and titrate with standard acid to the methyl orange endpoint and calculate as K_2CO_3.

II—Free Caustic (as KOH) and carbonates (as K_2CO_3) were determined by the following methods using freshly boiled, neutral, isopropyl alcohol, 91 per cent.

a) Same as described under Ia, except using isopropyl alcohol.

b) Same as described under Ib, except using isopropyl alcohol.

Conclusions

Although the results lack the desired uniformity, they show a definite trend under the various methods employed. Assuming that the K_2CO_3 determined by the Evolution Volumetric method represents the true CO_2 content present, the following deductions can be made:

TABLE II—*Sample A containing relatively low caustic and carbonate contents.* Using ethyl alcohol and not filtering, the results for carbonate are on the low side indicating some solubility of K_2CO_3 in the alcohol. The use of isopropyl alcohol without filtering gives carbonate results approaching rather closely the apparent true figure. On the other hand, filtration of the sample when isopropyl alcohol is used gives high carbonate values indicating partial insolubility or carbonation of free caustic.

TABLE III

Sample B—Soft Soap With Added Carbonate

Collaborator	Method Ia — Ethyl alcohol —Caustic before filtration	Method Ib — Ethyl alcohol —Caustic after filtration	Method IIa — Isopropyl alcohol —Caustic before filtration	Method IIb — Isopropyl alcohol —Caustic after filtration	Carb. as K_2CO_3 by evolution volumetric method
Armour & Co. 31st St. Aux.	Caus. 0.31% Carb. 0.21	0.21% 0.28	0.17% 0.64	0.03% 0.83	.... 0.99%
U. S. Dept. of Commerce, Nat'l Bureau of St'rds	Caus. 0.26% Carb. 0.19	0.06% 0.41	0.08% 0.75	Acid 0.89%	....
Fels & Co.	Caus. 0.000% Carb. 0.069	0.000% 0.069	0.000% 0.155	0.000% 0.153	....
Hercules Exp'tal Station	Caus. 0.27% Carb. 0.27	0.15% 0.38	0.10% 0.75	0.04% 0.73	....
Hooker Electro-Chemical Co.	Caus. 0.35% Carb. 0.05	0.18% 0.20	0.03% 0.73	Trace 0.80%	....
Lever Bros. Co.	Caus. 0.38% Carb. 0.10	0.29% 0.15	0.06% 0.90	0.01% 1.02	....
Los Angeles Soap Co.	Caus. 0.330% Carb. 0.165	0.110% 0.305	0.145% 0.650	0.010% 0.850	....
New York Produce Exchange	Caus. 0.065% Carb. 0.33	0.046% 0.26	*0.050% 0.69	*0.028% 0.29	....
Procter & Gamble	Caus. 0.246% Carb. 0.212	0.072% 0.424	0.082% 0.761	0.000% 0.955	....
** (Special Method)	Caus. 0.036% Carb. 0.759	0.000% 0.865	....	....	....
Swift & Co.	Caus. 0.302% Carb. 0.186	0.213% 0.255	0.010% 0.752	0.000% 0.828	....

* 99% Isopropyl Alcohol was used.

** Special Method based on the addition of 10 gms. salt to 20 gms. soap, mixing, dissolving in 200 c.c. of 95% alcohol and determining caustic in the alcohol soluble portion. K_2CO_3 determined by evolution.

TABLE III—*Sample B containing an added amount of free potassium carbonate.* Results definitely show that potassium carbonate is appreciably soluble in ethyl alcohol resulting in high free caustic values. The procedure using isopropyl alcohol and including filtering appears to yield results approaching more closely the apparent true values.

TABLE IV—*Sample C containing an added amount of free caustic.* The results as a whole show rather high carbonate content indicating either insolubility or carbonation of free caustic.

TABLE IV

Sample C—Soft Soap With Added Caustic

Collaborator	Method Ia — Ethyl alcohol —Caustic before filtration	Method Ib — Ethyl alcohol —Caustic after filtration	Method IIa — Isopropyl alcohol —Caustic before filtration	Method IIb — Isopropyl alcohol —Caustic after filtration	Carb. as K_2CO_3 by evolution volumetric method
Armour & Co. 31st St. Aux.	Caus. 0.31% Carb. 0.25	0.19% 0.16	0.28% 0.24	0.04% 0.52	.... 0.13%
U. S. Dept. of Commerce, Nat'l Bureau of St'rds	Caus. 0.34% Carb. 0.12	0.15% 0.29	0.22% 0.30	0.02% 0.52	....
Fels & Co.	Caus. 0.358% Carb. 0.055	0.210% 0.166	0.286% 0.187	0.129% 0.346	....
Hercules Exp'tal Station	Caus. 0.23% Carb. 0.14	0.10% 0.16	0.08% 0.54	0.03% 0.44	....
Hooker Electro-Chemical Co.	Caus. 0.34% Carb. 0.04	0.24% 0.09	0.22% 0.35	0.17% 0.29	....
Lever Bros. Co.	Caus. 0.37% Carb. 0.05	0.32% 0.09	0.40% 0.08	0.24% 0.32	....
Los Angeles Soap Co.	Caus. 0.380% Carb. 0.070	0.155% 0.130	0.325% 0.105	0.035% 0.465	....
New York Produce Exchange	Caus. 0.367% Carb. 0.045	0.31% 0.034	*0.356% 0.065	*0.19% 0.31	....
Procter & Gamble	Caus. 0.236% Carb. 0.142	0.102% 0.247	0.143% 0.319	0.000% 0.584	....
** (Special Method)	Caus. 0.077% Carb. 0.362	0.000% 0.459	....	....	....
Swift & Co.	Caus. 0.403% Carb. 0.09	0.19% 0.207	0.358% 0.221	0.034% 0.559	....

* 99% Isopropyl Alcohol was used.

** Special Method based on the addition of 10 gms. salt to 20 gms. soap, mixing, dissolving in 200 c.c. of 95% alcohol and determining caustic in the alcohol soluble portion. K_2CO_3 determined by evolution.

To summarize the above, the Committee concluded that while the proposed method offers some possibilities, there was too much divergence in results between laboratories to warrant adoption of the proposed method. Therefore, it was agreed to withhold any recommendation for making any change in the present method at this time. A sub-committee of five members agreed to undertake some further studies of the proposed method in order to determine, if possible, the reason for the variable results. Briefly, this further work is to consist of the addition of known amounts of KOH and K_2CO_3 to soaps prepared in the alcoholic media using fatty acids instead of glycerides.

Summary

Recommendations of the Committee are as follows:

1) Pyrophosphate in soap — Recommend tentative adoption of the volumetric method using the glass electrode.
2) Free alkali in potash paste soaps—No changes recommended in the method at the present time. Further studies will be undertaken.

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