

On most of the presses we have sold, we have recommended Teflon tooling. The tools are made up of a tool holder, metal insert, and teflon insert. As the Teflon insert wears, it can be readily replaced. The tool holder and metal insert wear very little and consequently, replacement is very seldom. We are able to use Teflon in this application, due to the light pressure requirements, 700–800 psi.

We have tried other materials in our laboratory, including silicon spray, different types of resins, etc., but it still appears that Teflon is the best material. In conjunction with the Teflon insert, we have been using a brushing and silicone spray system. After the tablet is ejected, the upper and lower punches are held in position and pass over and under a nylon brush. A silicone emulsion has been sprayed on the brush so that the Teflon face is wiped clean by the brush and at the same time, given a light coating of silicone. With this approach, we have been able to eliminate the sticking problems.

Due to the gummy nature of the detergent materials they tend to build up in various areas on the tablet press. Some materials we have seen will build up on any metallic surface. In this case, it is necessary to eliminate any material to metal contact. To do so,

it is necessary to use Teflon in all contact areas. In addition, it is necessary to coat the feed frames and other members with Teflon that comes in contact with the material.

Most detergent materials tend to build up in the die bore. To eliminate this, it is necessary to use an undercut on the lower punches. This undercut serves as a scraper that cleans the die bore as the lower punch is dropped to its fill position. The material is scrapped off and drops through the punch clearances where it is picked up by the vacuum system of the press. You will notice that we use as small an area as possible on the diameter to keep the contact area to a minimum.

Another area of build up is the lower punch socket. When running the tools it is necessary to have clearance between the punch and the die. This clearance enables the fine material to sift down to the punch sockets. To prevent this it is recommended to have an excellent vacuum system at the position underneath the die bore and just above the punch socket.

This has been a brief coverage of detergent tableting. To conclude, we again point out the most important factors in a successful tableting operation are the preparation of feed material and the design of the press.

Phosphate Builders for Detergents

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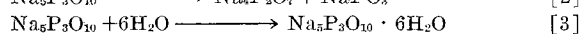
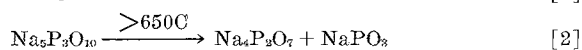
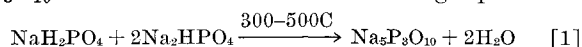
CRYSTALLINE sodium tripolyphosphate was first made by Schwartz (1) in 1895, by melting together the proper proportions of $\text{Na}_4\text{P}_2\text{O}_7$ and NaPO_3 , and slowly cooling the melt.

At the present time, two crystalline anhydrous polymorphic forms of sodium tripolyphosphate are known: the low temperature form—Type II, and the high temperature form—Type I. There is also an hydrated form— $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$ which results when either anhydrous form is crystallized from water.

Sodium tripolyphosphate is made commercially by the thermal intermolecular dehydration of a mixture of NaH_2PO_4 and Na_2HPO_4 , such that Na/P is 1.67. When such a mixture is heated above 200C, $\text{Na}_5\text{P}_3\text{O}_{10}$ starts to form at an appreciable rate—the Na_2HPO_4 portion of the mix passing through $\text{Na}_4\text{P}_2\text{O}_7$. At 300C, the rate of conversion is sufficiently fast to form $\text{Na}_5\text{P}_3\text{O}_{10}$ in a few minutes. When the orthophosphate mixture is heated to a final temperature of 350–400C, the product resulting is mostly $\text{Na}_5\text{P}_3\text{O}_{10}$ -II.

In the temperature range of 450–500C, there is a rapid transformation of $\text{Na}_5\text{P}_3\text{O}_{10}$ -II to $\text{Na}_5\text{P}_3\text{O}_{10}$ -I. This conversion is usually not complete and the commercial mixed type product contains 25–35% $\text{Na}_5\text{P}_3\text{O}_{10}$ -I. With continued rise in temperature, $\text{Na}_5\text{P}_3\text{O}_{10}$ melts incongruently at 622C, forming NaPO_3 and $\text{Na}_4\text{P}_2\text{O}_7$.

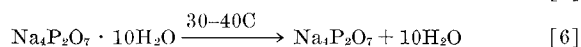
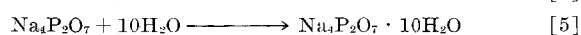
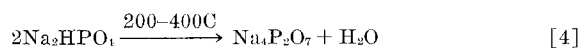
The formation, decomposition and hydration of $\text{Na}_5\text{P}_3\text{O}_{10}$ are summarized in the following equations:



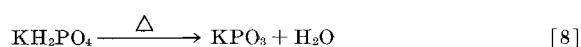
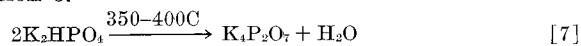
The thermal intermolecular dehydration of Na_2HPO_4 to $\text{Na}_4\text{P}_2\text{O}_7$ is much more simple and

straightforward than the case of $\text{Na}_5\text{P}_3\text{O}_{10}$. Heating Na_2HPO_4 in the range 175–250C causes a disappearance of the orthophosphate and a simultaneous buildup of pyrophosphate. No further change appears to take place until the melting point of $\text{Na}_4\text{P}_2\text{O}_7$ is reached at 985C.

Commercially, a solution of sodium orthophosphate, having a Na/P of 2, is evaporated and thermally converted to $\text{Na}_4\text{P}_2\text{O}_7$. The formation, hydration and dehydration of $\text{Na}_4\text{P}_2\text{O}_7$ are shown here:



The preparation of $\text{K}_4\text{P}_2\text{O}_7$ is quite analogous to that of $\text{Na}_4\text{P}_2\text{O}_7$. K_2HPO_4 begins to dehydrate at temperatures near 250C, the dehydration becoming rapid at 350C. A complication in this process is the ease with which KH_2PO_4 is converted to insoluble KPO_3 . It is therefore necessary to adjust the K/P ratio carefully to 2 and convert rapidly, so that the disproportionation of K_2HPO_4 into KH_2PO_4 is minimized, in order to produce a completely soluble product. The preparation of $\text{K}_4\text{P}_2\text{O}_7$ is shown in equation 7. The formation of KPO_3 is given in equation 8.



The preparation of the glassy sodium polyphosphates is a relatively simple matter. Sodium orthophosphate of the desired Na/P ratio is heated above its melting point of 628C, e.g., 700C, and then quenched by cooling rapidly to room temperature. Graham's salt, which has the theoretical Na/P of 1, contains 69.6% P_2O_5 . The glassy polyphosphate of

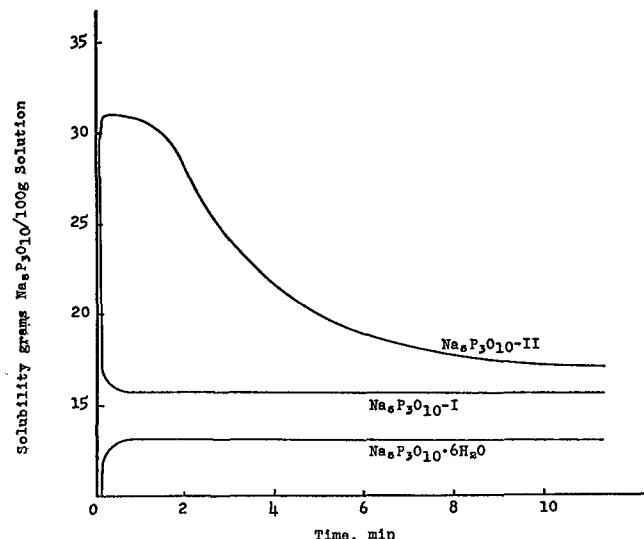
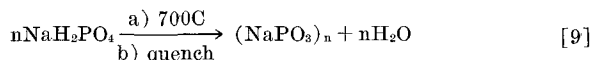


FIG. 1. Solubility of $\text{Na}_5\text{P}_3\text{O}_{10}$.

commerce has a lower P_2O_5 content, i.e., 67.0 to 67.5%, corresponding to Na/P ratio of 1.1.

Glassy sodium polyphosphates do not contain one chemical species, but rather a series of polyphosphates of varying P-O-P chain lengths. They are usually described in terms of the average chain length, which is a weighted average of all chain lengths present. The average chain length, \bar{n} , is a function of two things: P_2O_5 content and water content. For example, a 69.0% P_2O_5 containing glass will have an \bar{n} of 70 for no H_2O and an \bar{n} of 30 for 0.5% H_2O . The 67% P_2O_5 glass of commerce has an \bar{n} of 12.

The preparation of glassy sodium polyphosphate is shown in this equation.



The solubility of $\text{Na}_5\text{P}_3\text{O}_{10}$ in water is limited by the solubility of $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$, which is the saturating crystal phase over the entire temperature range of 0–100°C. This solubility is in the order of 15%. The rate of hydration of Type I $\text{Na}_5\text{P}_3\text{O}_{10}$ is greater than the rate of hydration of Type II $\text{Na}_5\text{P}_3\text{O}_{10}$. As seen in Figure 1, when Type II $\text{Na}_5\text{P}_3\text{O}_{10}$ is dissolved in water, there is an initial apparent solubility of more than 30%. With time, as hydration takes place, the solubility drops toward the equilibrium figure for $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$. In the case of $\text{Na}_5\text{P}_3\text{O}_{10}$ -I, there is a rapid drop in the concentration of dissolved solids toward the equilibrium value. When the two forms of $\text{Na}_5\text{P}_3\text{O}_{10}$ are dissolved in water, there is the release of considerable heat of hydration. The integral heat of solution for $\text{Na}_5\text{P}_3\text{O}_{10}$ -I is 16.1 kcal/mol and that for $\text{Na}_5\text{P}_3\text{O}_{10}$ -II is 14.0 kcal/mol.

The lumping observed when $\text{Na}_5\text{P}_3\text{O}_{10}$ -I is dissolved in water is caused by the high solubility of $\text{Na}_5\text{P}_3\text{O}_{10}$ -I in water, together with the rapid hydration to $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$, which results in a matting together of the entire mass by a mesh of $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$ crystals. $\text{Na}_5\text{P}_3\text{O}_{10}$ -II does not give this lumping effect.

The fact that $\text{Na}_5\text{P}_3\text{O}_{10}$ -I hydrates more rapidly than $\text{Na}_5\text{P}_3\text{O}_{10}$ -II is the basis for the empirically derived Temperature Rise Test (TRT) by which it is possible to determine the ratio of Type I and Type II $\text{Na}_5\text{P}_3\text{O}_{10}$ in mixtures of both. The percentage of Type I is given by equation 10.

$$\% \text{Na}_5\text{P}_3\text{O}_{10} \text{-I} = 4[\text{temp rise in } ^\circ\text{C}] - 6 \quad [10]$$

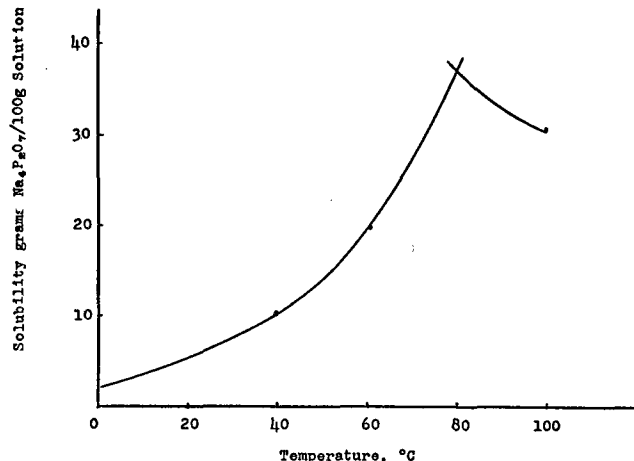


FIG. 2. Solubility of $\text{Na}_4\text{P}_2\text{O}_7$.

Thus, pure $\text{Na}_5\text{P}_3\text{O}_{10}$ -II has a TRT of 6C, pure $\text{Na}_5\text{P}_3\text{O}_{10}$ -I has a TRT of 30C, commercial mixed type $\text{Na}_5\text{P}_3\text{O}_{10}$ has a TRT of 12–14C.

The solubility of $\text{Na}_4\text{P}_2\text{O}_7$ is shown in Figure 2. Below 79.5°C, the saturating crystal phase is $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$; above 79.5°C, it is anhydrous $\text{Na}_4\text{P}_2\text{O}_7$. The heat of solution of $\text{Na}_4\text{P}_2\text{O}_7$ in 800 moles of water is 11.65 kcal/mol.

The solubility of $\text{K}_4\text{P}_2\text{O}_7$ in water is shown in Figure 3. The high solubility of $\text{K}_4\text{P}_2\text{O}_7$ makes it suitable for use in heavy duty liquid detergents. The integral heat of solution of $\text{K}_4\text{P}_2\text{O}_7$ in 75 moles of water is 13 kcal/mol. The solid in equilibrium with the saturated solution, in this case, is $\text{K}_4\text{P}_2\text{O}_7 \cdot 3\text{H}_2\text{O}$.

All of the sodium phosphate glasses having Na/P ratios between 1 and 1.67 are water soluble. Glasses with $\text{Na}/\text{P} < 1.5$ appear miscible in all proportions with water. There is an increase in viscosity as the concentration of polyphosphate increases. For example, the viscosity of a 70% solution is about one million times that of pure water. After long storage, concentrated solutions of glassy polyphosphates precipitate less soluble short chain products of hydrolysis.

In neutral solutions at room temperature, the hydrolytic degradation of condensed phosphates is

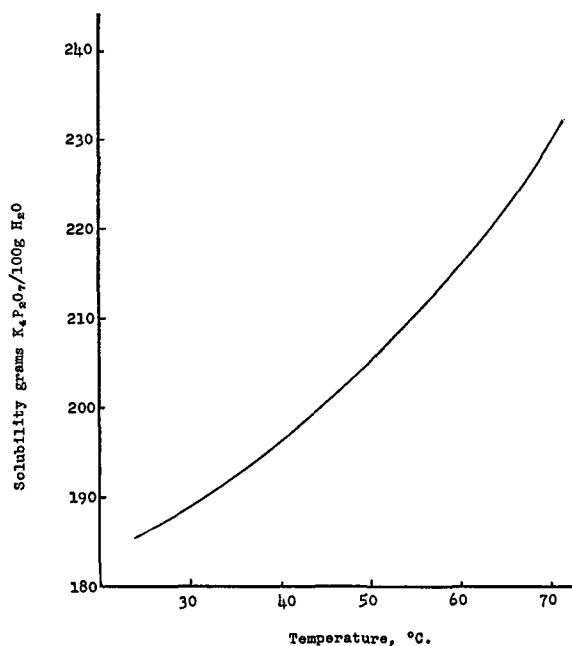


FIG. 3. Solubility of $\text{K}_4\text{P}_2\text{O}_7$.

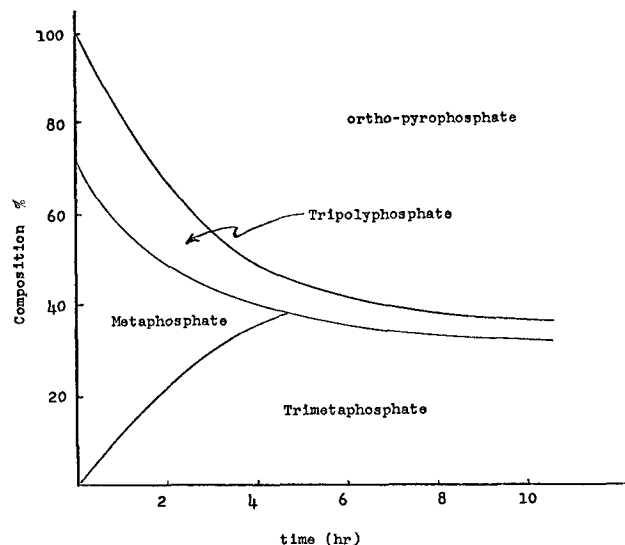
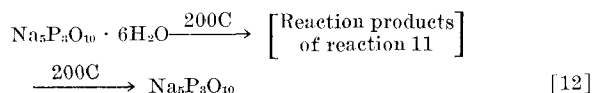
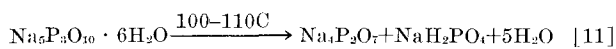


FIG. 4. Hydrolysis of commercial sodium metaphosphate glass (1% solution in water at 100C).

very slow. For example, $\text{Na}_5\text{P}_3\text{O}_{10}$ at 25C/pH 7 has a half-life of more than one year. The rate of hydrolysis is dependent upon concentration, pH and temperature.

Despite the slow hydrolysis of dilute alkaline solutions of tripolyphosphates, the rate is very much faster when concentrated slurries are heated; as for example, when detergent mixtures are spray-dried. The extreme example is the case of the dehydration of $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$. As is shown in equations 11,12, when $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$ is heated at 100–110C, the product is not anhydrous $\text{Na}_5\text{P}_3\text{O}_{10}$; but rather, a mixture of crystalline $\text{Na}_4\text{P}_2\text{O}_7$ and amorphous acidic sodium ortho- and pyrophosphates. Only at temperatures above 120C, and more rapidly at 200C, does recombination to $\text{Na}_5\text{P}_3\text{O}_{10}$ occur.



The hydrolysis of glassy polyphosphates differs in one respect from the hydrolysis of short chain phosphates, in that small rings—mainly trimetaphos-

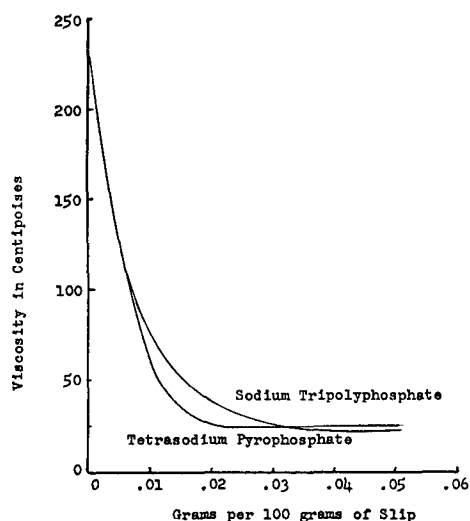


FIG. 5. Deflocculation of china clay slip containing 55% solids at 25C.

TABLE I
1962¹ U. S. Production

	Short tons
$\text{Na}_5\text{P}_3\text{O}_{10}$	770252
$\text{Na}_4\text{P}_2\text{O}_7$	97175
$\text{K}_4\text{P}_2\text{O}_7$	30007
$(\text{NaPO}_3)_n$	65755

¹ Preliminary Report—Dept. of Commerce.

phate—are formed during the degradation of the long chains. These rings result when the ends of long chains are split off in neutral and alkaline solutions. In acid solutions there is also some random splitting along the chain. The rate of hydrolysis is in the same order of magnitude as that for pyro- and tripolyphosphate. The hydrolysis of glassy polyphosphate solutions is shown in Figure 4.

The polyphosphates are used as peptizing, deflocculating and dispersing agents, as would be predicted from their high charge. This high charge also contributes to their action as detergent builders, by aiding in salting out and stabilizing organic surfactant micelles.

Figure 5 shows the deflocculating action of sodium polyphosphate on a china clay slip containing 55% solids at 25C.

Chain phosphates are strongly adsorbed on surfaces. The inhibition of formation of calcium carbonate crystals by adsorption of chain phosphate on the calcite crystal nuclei—the threshold effect—is an application of this property. The addition of only a few ppm of polyphosphate to hard water results in an extremely low rate of nucleation of calcite crystals. Ring phosphates are ineffective in this application.

Large quantities of sodium and potassium polyphosphates are produced annually in the United States, much of it going into detergent uses. Table I lists production figures for 1962.

In recent years, granular $\text{Na}_5\text{P}_3\text{O}_{10}$ has been produced in an ever increasing volume. The main reason for this large increase has been the many different types of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ which have appeared. The density of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ has been varied from a low 24–28 lb/cu ft to a high of over 60 lb/cu ft. The heavier density $\text{Na}_5\text{P}_3\text{O}_{10}$ is produced largely in rotary kilns, while the extremely light density $\text{Na}_5\text{P}_3\text{O}_{10}$ is produced by spray-drying and the intermediate densities are produced by various agglomeration techniques.

The physical properties of these various density $\text{Na}_5\text{P}_3\text{O}_{10}$'s vary considerably and allow the user to select a particular grade.

Table II gives typical sieving analyses for various grades of granular $\text{Na}_5\text{P}_3\text{O}_{10}$. The granulation of the lower density material can be varied over a fairly wide range. The variation in the denser ranges for $\text{Na}_5\text{P}_3\text{O}_{10}$ is considerably less.

One of the outstanding properties of granular $\text{Na}_5\text{P}_3\text{O}_{10}$, particularly the low density $\text{Na}_5\text{P}_3\text{O}_{10}$, is their ability to absorb nonaqueous liquid, usually

TABLE II
Granulation of Various Grades of Granular Sodium Tripolyphosphate

Density range (Lb/cu ft)	24–30	32–38	40–50	50–60
Sieving range:				
% On 20 Mesh	0–50	0–50	0–10	0–10
% On 30 Mesh	0–50	0–50	5–30	5–30
% On 40 Mesh	20–60	20–50	20–50	20–50
% On 60 Mesh	20–60	20–50	20–50	20–50
% On 80 Mesh	10–30	10–30	10–30	10–30
% On 100 Mesh	5–10	5–10	5–10	5–10

TABLE III
Absorption Capacity of Various Grades of Granular Sodium Tripolyphosphate

Density of STPP (Lb/cu ft)	% Absorption
24-28	10-14
30-34	10-22
36-45	9-12
45-50	6-10
60-	4-6

liquid wetting agents, and still remain in a free-flowing condition.

Table III shows the absorption of various densities of granular $\text{Na}_5\text{P}_3\text{O}_{10}$. The absorption capacity was measured by slowly adding a liquid wetting agent to granular $\text{Na}_5\text{P}_3\text{O}_{10}$, with good stirring, until the material was too clammy to flow through a funnel which had an orifice of approximately one half inch. As can be seen from the data, the lower density $\text{Na}_5\text{P}_3\text{O}_{10}$ has a higher absorption capacity than the heavier density $\text{Na}_5\text{P}_3\text{O}_{10}$. However, it is possible to have two samples of $\text{Na}_5\text{P}_3\text{O}_{10}$ with the same density, which show a great difference in absorption capacity. This can be explained by the difference in granulation of the two samples.

In Table IV, is shown the absorption capacity of various granulations of $\text{Na}_5\text{P}_3\text{O}_{10}$, which were screened from a sample having a density of 32 lb/cu ft. In the second column is given the density of the various granulations. Thus we see that the granulation plays an important part in the absorption capacity of various types of low density $\text{Na}_5\text{P}_3\text{O}_{10}$.

In the past three Tables it has been shown that many different densities of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ have been made and the absorption capacity of the lower densities can be varied considerably by changing the granulation of the $\text{Na}_5\text{P}_3\text{O}_{10}$. However, it must be pointed out that samples of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ have been found which have identical densities and very similar sievings and yet the absorption capacity of one may be 50% greater than the other. This can probably be explained by variations in method of manufacture, which cause changes in the shape and porosity of the $\text{Na}_5\text{P}_3\text{O}_{10}$ granule. $\text{Na}_5\text{P}_3\text{O}_{10}$ granules which have small voids present, as seen under the microscope, tend to have higher absorption capacity than $\text{Na}_5\text{P}_3\text{O}_{10}$ granules which are spherical in shape and have such minute voids that they do not readily absorb heavy liquids.

Another important property of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ is the hardness of the $\text{Na}_5\text{P}_3\text{O}_{10}$ granule. If the granule is very light, as is true in low density $\text{Na}_5\text{P}_3\text{O}_{10}$ (24-28 lb/cu ft), it may not have sufficient strength to resist breakdown during handling and shipping. The user may find that the density has increased considerably. With this increase in density, an increase in the amount of powdered $\text{Na}_5\text{P}_3\text{O}_{10}$ is found, which causes the user trouble due to dustiness and segregation.

Table V shows the hardness of various densities of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ as measured by a) per cent density increase, and b) increase in the amount of powder present. The test for measuring these differences consist of putting granular $\text{Na}_5\text{P}_3\text{O}_{10}$ in gallon jars containing several rubber balls (1¼ in. diam) and rolling the jars on ball mill equipment for a definite period of time. The rolling action of the rubber balls on the $\text{Na}_5\text{P}_3\text{O}_{10}$ granules tests their strength and resistance to breakdown. The density of the $\text{Na}_5\text{P}_3\text{O}_{10}$, measured before and after the test, as well as complete sieving analysis, gives an indica-

TABLE IV
Density and Absorption of Various Granulations of Sodium Tripolyphosphate

Mesh	% Absorption	Density of material (Lb/cu ft)
20	22	28
30	20	29
40	18	30
60	14	34
80	9	36

tion of the strength of various density ranges of $\text{Na}_5\text{P}_3\text{O}_{10}$. From the data, it can be seen that, in general, the lower the density, the weaker the $\text{Na}_5\text{P}_3\text{O}_{10}$ granule, as indicated by the percentage density increase and increase in the amount of power present. It must be pointed out, however, that some samples of $\text{Na}_5\text{P}_3\text{O}_{10}$ having the same original density and which have the same percentage density increase, show a considerable difference in the amount of powder produced. It has been found that this is not due to different granulations, but is apparently due to different methods of producing the $\text{Na}_5\text{P}_3\text{O}_{10}$.

One can see from the various densities, granulations, absorption capacities and particle strengths of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ now being produced, that the user has a versatile product which he can use in many different ways. One way in which an ever-increasing number of people are using these various grades of $\text{Na}_5\text{P}_3\text{O}_{10}$ is in dry blending of detergents and cleaning products. In dry blending, the appropriate grade of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ is used in absorbing any liquids present, and proper selection of other materials used reduce segregation to a minimum. The dry blending of detergents and cleaning products requires a minimum of equipment which allows small operators to produce a satisfactory product without the costly step of spray-drying. The densities of dry blended products are generally higher than those which have been spray-dried, but they have found acceptance by the consumer and this type of formulating is expected to continue growing. The various densities of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ available, as well as other low density-high absorbent material such as Flozan—a light density Na_2CO_3 —have enable the formulators to vary the density of their finished product over a fairly wide range.

One of the largest outlets for granular $\text{Na}_5\text{P}_3\text{O}_{10}$ is for use in making detergent tables. This is a relatively new market and the predicted future promises a steady growth for this product. There are two approaches in using $\text{Na}_5\text{P}_3\text{O}_{10}$ in making tablets. One is spray-drying of part or all of the material and then tableting, while the other approach is blending of the material in various types of mixers and then tableting. It is the latter form with which granular $\text{Na}_5\text{P}_3\text{O}_{10}$ plays a most important part.

TABLE V
Hardness of Sodium Tripolyphosphate Granules

Sample	Density (Lb/cu ft)		% Powder (T-100 Mesh)	
	Original	% Increase after ball milling	Original	After ball milling
1	24	14	4.3	11.4
2	28	14	5.8	12.7
3	30	16	3.6	12.5
4	32	13	0.8	4.5
5	32	14	2.8	8.2
6	34	12	1.2	4.7
7	36	10	2.0	4.0
8	40	5	1.5	1.9
9	45	4	1.3	1.7
10	50	4	1.2	1.6
11	60	2	1.2	1.6
12	65	2	0.3	0.5

TABLE VI
Disintegration Rate of Tablets as a Function of TRT of Granular Sodium Tripolyphosphate

Tablet	TRT of STPP	Disintegration rate
1	6	Over 5 min
2	8	Over 5 min
3	10	5 min
4	11	3 min
5	12	2 min
6	14	2 min

The problem of making a detergent tablet with the proper physical properties is quite frustrating. One must mix the material before tableting in such a way as to give a uniform formulation, which will flow readily and not cause sticking problems in the tableting machine. At the same time, the mixture must have enough tackiness to produce a tablet which has sufficient strength to be handled without breaking. Finally, the housewife buys the product, expects to find it still in one piece, and hard enough to resist breaking if she accidentally drops it; and yet, when she places it in the washing machine, she expects it to disintegrate immediately.

There are several different types of formulations which can be tabulated successfully and since each formulation requires a different type of treatment, a generalization of the properties of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ which are important in making tablets will be given:

Density: The density of the granular $\text{Na}_5\text{P}_3\text{O}_{10}$ can vary from 30–45 lb/cu ft depending upon the amount used and the density of the other materials in the formulation. When the density of the mix, before tableting, is above 50 lb/cu ft, it becomes quite difficult to produce a satisfactory tablet.

Absorption: The absorption of the granular $\text{Na}_5\text{P}_3\text{O}_{10}$ used depends upon the amount of liquids used in the formulation.

Granulation: In general, the coarser the granula-

tion the better it is for tableting, provided there is sufficient tackiness to give good strength to the tablets. The coarser granules provide small voids in the tablets; which, when the tablet is placed in water allow rapid water penetration and increase the rate of disintegration.

TRT: It has been found that when the TRT of the granular $\text{Na}_5\text{P}_3\text{O}_{10}$ is 11 or above, the tablet disintegrates much faster than for lower TRT's. The high heat of hydration and the rapid rate of formation of tripoly hexahydrate, as produced by granular $\text{Na}_5\text{P}_3\text{O}_{10}$ with TRT of 11 or above, causes the tablets to swell when placed in water and disintegration is quite rapid.

Table VI shows the disintegration rate of detergent tablets made from the same formulation, the same tablet weight, and pressure with $\text{Na}_5\text{P}_3\text{O}_{10}$'s having different TRT's. The rate of disintegration was determined by placing the tablet in one liter of water at 120F and visually observing how long the tablet took to completely break apart. No agitation is used during the test.

Thus, it can be seen that the TRT of granular $\text{Na}_5\text{P}_3\text{O}_{10}$ is an important factor in controlling the disintegration rate of a tableted product.

Summary

In summary, the chemistry of the preparation of sodium and potassium polyphosphates, and the properties of importance to their use as detergent builders, such as hydrolysis, hydration rates and solubilities have been discussed. The properties of granular $\text{Na}_5\text{P}_3\text{O}_{10}$, such as density, particle size, frangibility and absorptivity, were discussed in connection with their use in the dry blending and tableting of detergent products.

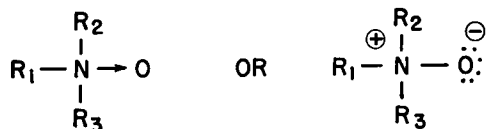
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Recent Advances in Fatty Amine Oxides. Part I. Chemistry and Preparation

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AMINE OXIDES are reaction products of tertiary amines and hydrogen peroxide or peroxyacids. Aliphatic tertiary amines are readily oxidized by hydrogen peroxide, whereas aromatic and heterocyclic amines require the use of peroxyacids. The structure of amine oxides may be represented in either of the following ways:



R_1 , R_2 , and R_3 may be aliphatic, aromatic, heterocyclic or alicyclic. For example, the oxides of triethylamine, pyridine, dimethylaniline and dimethylcyclohexylamine are known as are those of hundreds of other amines in these classes. The amine oxides of interest in detergents are those derived from fatty amines such as dimethyldodecylamine.

Amine oxides were first studied in the last decade of the nineteenth century but little was done at that time toward defining their properties or finding prac-

tical applications. In the 1930's and '40's, investigators found evidence of amine oxide structures in alkaloids and other naturally occurring materials, and the widespread occurrence of amine oxides in nature was soon recognized. Chemotherapeutic investigations showed that alkaloid amine oxides retained the physiological and therapeutic effectiveness of the parent bases but were much less toxic. This, plus the mild antibacterial activity shown by certain amine oxides, prompted extensive investigations resulting in numerous publications and patents relating primarily to heterocyclic and heteroaromatic amine oxides. Examples of successful ventures resulting from this work are diazepam oxide tranquilizers and pyridine oxide antibacterials.

The utility of aliphatic amine oxides as surfactants was first noted by Du Pont, who obtained a patent in 1939 relating to dialkylaminoacid oxides for use as detergents and foam stabilizers (1). A U.S. patent granted to I. G. Farbenindustrie A.-G. later the same year disclosed dimethyldodecylamine oxide as a wetting, cleansing, and dispersing agent (2). This oxide has found a limited market in the textile industry,