Detergent Analysis

Sir: Analysis of some current formulations of detergent products has shown that standard analytical methods for phosphate are not applicable. Borax, perborate or high levels of silicate cause positive interference when detergents are analyzed for phosphate by AOCS Method Da 20b-57 or ASTM D 820-58. In view of current widespread interest in phosphate content, we believe it is important that the need for modification of these methods be recognized. This communication is intended to bring the problem to the attention of others concerned with the analysis of synthetic detergents and to suggest means for modifying the procedures.

Both AOCS method Da 20b-57 and ASTM D 820-58 (Reapproved 1970) are based on a titration procedure suggested by Andrews (JAOCS 31:192, 1954). After removal of organic matter by dissolving in alcohol or ashing, the phosphates are converted to the *ortho* form and determined by titration between pH 4.3 and 8.8. Weak acids lead to high results and following the standard method does not remove the amount of borax (or perborate) now incorporated in some products appearing on the market. Silicate if present at SiO_2 to P_2O_5 ratios greatly exceeding 0.2 is not completely dehydrated by the current procedure.

A simple modification of the procedure avoids these interferences. Boron compounds are removed by volatilization as methyl borate. Silicate interference is avoided by additional dehydration.

AOCS Method Da 20b-57 should be modified in the following way unless the analyst is certain that the interferences are absent.

Delete paragraph C3 and substitute the following:

C3 – Built synthetic detergent samples are treated by ashing. Weigh a sample, of size chosen by the formula above (but do not exceed 10 g) to the nearest 0.001 g. Place sample in a porcelain or silica evaporating dish, or large crucible, and ignite gently over a low gas burner until most of the volatile combustible matter is burned off. Transfer to a muffle, operated at not over 550 C, for 10 to 15 min. The ignited residue need not be free from carbon and usually is of a grayish color.

Cool and add cautiously 10 ml of HCl. Evaporate to dryness on open steam. If the ratio % SiO_2/% P_2O_5 approaches or exceeds 0.2 or is unknown, dehydrate the silicates completely by cooling the sample and repeating the HCl addition and evaporation two additional times. After the third evaporation, continue to heat the residue for an additional 15 to 20 min after dryness is attained to insure complete dehydration of SiO_2.

Cool the sample and transfer into a 400 ml beaker using distilled water and proceed as in (a) or (b) below.

(a) If the sample contains perborate or borate, evaporate to dryness on a steam bath, add about 200 ml methanol, 10 ml HCl and 2 or 3 boiling stones. Partially cover the beaker with a watch glass and boil down to a volume of about 20 ml (boiling time must be at least 30 min). Evaporate down to less than 10 ml on a steam bath under a stream of nitrogen or clean, dry air. Proceed as described under Section D.

(b) If the sample is known to be free from perborate and borate, add distilled water to make a total of about 90 ml. Add 10 ml concentrated HCl and proceed as in Section D.

The analytical results in Table I illustrate the effect of various levels of perborate. Analysis of known mixtures showed the results by the modified method to be correct.

A detergent product having a ratio of 0.28 SiO_2 to P_2O_5 showed 16.2% P_2O_5 by AOCS Da 20b-57 and 15.9%, the correct value, by the modified procedure.

Neither the standard nor the modified methods will give accurate results on detergent products containing a few per cent or less phosphate. For example, a sample containing carbonate, perborate and silicate but no phosphate showed more than 0.4% apparent P_2O_5 by both methods.

TABLE I

	Per cent P_2O_5			
Sample	Da 20b-57	Modified method	Difference	% perborate
1	43.1	37.9	5.2	26.4
2	24.9	22.6	2.3	11.6
3	26.3	25.1	1.2	4.5
4	27.2	28.3	-0.1	0

J.C. ABBOTT
E.M. SALLEE
Procter & Gamble Company
Ivorydale Technical Center
Cincinnati, Ohio 45217

[Received September 28, 1970]