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Evaluation of Detergent Formulations

Final Report

for DEPARTMENT OF THE ENVIRONMENT

INLAND WATERS DIRECTORATE

August 17, 1972 M.J. Williams

EVALUATION OF DETERGENT FORMULATIONS

1.0 INTRODUCTION AND SUMMARY

The purpose of this study was to compare the efficiency of different detergent formulations from the standpoint of soil removing ability. A standard detergent formulation based on Canadian Government Specification Board (CGSB) specifications was used throughout the study and the effect of varying the concentration of phosphate, NTA and citrate at different levels of water hardness was investigated.

Standard soiled fabrics were used and soil removal was assessed after washing under standardized domestic laundering conditions. The amount of dirt present on any sample was assessed by measuring its white light reflectance. A block of magnesium oxide was taken as a standard representing 100%. The initial reflectance values for any given type of fabric was the same and hence the values after washing could be taken as a measure of laundering efficiency. It should be noted, however, that the initial values did differ considerably from one fabric type to another. When comparing any efficiencies it should be borne in mind that a difference of 3% between reflectance values is fairly easy for the human eye to detect.

Another point to consider when reading this report is that different builders only reach optimum efficiency with certain surfactant formulations. For obvious reasons, however, in this program it was necessary to standardize the basic formulation and vary only the builder type and concentration. It is conceivable, therefore, that certain of the experimental products, particularly those containing NTA and/or citrates could have been improved by adjustment of surfactant composition.

2.0 MATERIALS

2.1 Soiled Fabrics

Artificially soiled fabrics were used in the evaluations. To include samples of the type of fabric, particularly with respect to fibre

composition, that a consumer would encounter the following were selected:

- (a) All cotton fabric EMPA 112 soiled with cocoa sweetened milk mixture representing a type of pigment fat soil which often occurs on table linen (COTTON CMS).
- (b) Polyester/cotton 65/35 fabric with durable press finish (Soil Cloth #26 Specification SIS-47 U.S. Bureau of Ships) soiled with mixture containing ethyl cellulose, lamp black, hydrogenated vegetable oil, mineral oil, corn starch, oleic acid, etc. (DACRON/COTTON STC).
- (c) All cotton fabric soiled with the same mixture as fabric (b) (COTTON STC).
- (d) Spun polyester fabric soiled with the same mixture as fabric (b) (DACRON STC).
- (e) Spun nylon fabric soiled with the same mixture as fabric (b) (NYLON STC).
- (f) Spun Acrylic fabric soiled with the same mixture as fabric (b) (ORLON STC).

2.2 Detergents

The basic detergent formulation used throughout the study was one conforming to CGSB provisional standard for detergent, laundry, power-built 2GP-115P July 1970, with the exception that the phosphate was omitted and replaced by sodium sulphate. This formulation is shown below:

Sodium alkyl benzene sulphonate (CEDAPON	S-85 flakes)							
Chemical Developments of Canada Ltd.	25%							
Sodium carboxymethyl cellulose CMC D435								
Chemical Developments of Canada Ltd.	1%							
Sodium silicate BRITESIL C-20								
National Silicates Ltd.	10%							
Sodium sulphate anhydrous B.D.H.	64%							

Different levels of phosphate, NTA and citrate were achieved by replacing the appropriate proportion of sodium sulphate with sodium tripolyphosphate, nitrilotriacetic acid and sodium citrate respectively. The different formulations investigated are shown in Table I.

2.3 Water Supply

All tests were carried out using four different hardness levels. Nominally these were chosen as 80, 135, 350 and 550 ppm (expressed as $CaCO_3$). To obtain the required levels adjustments were made either by suitable dilution with distilled water or by the addition of sufficient calcium chloride ($CaCl_22H_20$) and magnesium sulphate ($MgSO_4.7H_20$) in the ratio of 3:1 when expressed in equivalents of $CaCO_3$. The actual water hardness values were determined by removing aliquots from the wash liquors and analyzing by the Ethylene Diamine Titrimetric Method - Method 122B Standard Methods for the Evaluation of Water and Waste Water. The four levels of water hardness were calculated to be 80, 133, 330 and 550 ppm. These values are typical of the range of hardnesses that exist across Canzda, although 65% of the population use water of 133 ppm hardness or less, and 85% use water of 330 ppm or less.

3.0 SPECIMENS AND REPLICATIONS

Each test specimen composed a 30" x 30" piece of undyed cotton broadcloth carrier fabric to which 4" x 4" squares of the six artificially soiled fabrics were attached by stapling along the edges. Duplicate samples of each soiled material were included in each wash and the whole procedure was carried out by two technicians using separate washing machines. The experiment therefore provided results for four replications.

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	. <u>A</u>	B	C	D	E	F	G	H	I	J	K	L	M	<u>N</u>	0	<u> </u>	Q	R	S	<u>T</u>	<u>U</u>	<u>v</u>	W	X	Y	Z	_α	β	8
LAS	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	. 25	25	25	25	25	25	25	25	25	25
CMC	1	1	1.	1 .	1	1	1	1	. 1	1	1	1	1	1	1	· 1 ·	1	1	1	. 1	1	1	1	1	1	1	1	1	1
S. SIL	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
S. SUL	64	55.2	46.5	37.7	29	59	54	49	44	59	54	49	44	40.2	36.5	32.7	40.2	36.5	32.7	7 44	44	44	38.3	26.5	26.5	30.2	30.2	23.7	22.7
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STPP		5	10	15	20	_	-		_	-	-	_	-	5	10	15	5	10	15	-	. –		7	10	10	5	5	15	15
CITRATE	-	-		-	· <u>-</u>	· · -	-	_	-	, 5	10	15	20	15	10	5	-	- .	. - ·	15	10	5	7	15	5	10	15	5	10
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NTA - nitrilotriacetic acid

STPP - sodium tripoly-phosphate (expressed as P_2O_5)

CITRATE - sodium citrate

4.0 EXPERIMENTAL

4.1 Washing Procedure

All launderings were carried out in Kenmore Model 600 automatic domestic washing machines having capacities of approximately 14 imperial gallons. The regular wash cycle was used with an initial wash liquor temperature of 147°F. When dilution with distilled water was needed the required volume was preheated to this temperature.

Sufficient undyed cotton was added to make up a six-pound load and 100 g of each detergent was used. The test specimens were subsequently ironed to dryness.

4.2 Assessment of Soil Removal

Reflectance readings of the original soiled and laundered swatches were made at four different locations on each swatch using an incandescent light source with a narrow band filter allowing light to be transmitted at 570 nm wavelength. The measurements were made on a Zeiss Elrepho photometer, calibration being made against a standard block of magnesium oxide representing 100% reflectance. The mean white light reflectance values after laundering the soiled fabrics with the detergents listed in Table I are recorded in the six Interim Reports which are appended.

5.0 RESULTS

The effectiveness of phosphate, NTA and citrate as detergent builders can be compared with reference to Figs. 1-6.

The effectiveness of formulations containing NTA as well as those containing 20% citrate can be compared with reference to Figs. 1d to 6d.

The results for bi and tri component blends of builders are not recorded here, although they can be found in the appendix. The reason is that no indications of any synergism was found and the behaviour of the blends is predictable from the results for the individual components.

6.0 STATISTICAL ANALYSES

Each data point in graphs 1-6 represents the mean of a sample of test specimens. The sample size varied between 12 to 24 test specimens, depending on the specific type of fabrics as well as the type of detergent under study. The confidence range for each calculated mean was estimated via the use of t - statistic as described below:

> By definition $t = \frac{|\bar{x} - \mu|}{S(\bar{x})}$ (1) where t = t value $\bar{x} = mean \text{ of the sample with size N}$ $\mu = true mean$ $S(\bar{x}) = standard error of the mean$

Transposing Equation (1)

 $\mu = \bar{\mathbf{x}} \pm t S_{(\bar{\mathbf{x}})} \qquad \dots \qquad (2)$ since $S_{(\bar{\mathbf{x}})} = \frac{S_{(\mathbf{x})}}{\sqrt{N}} \qquad \dots \qquad (3)$

where S(x) = standard deviation of the sample with size N

Substituting (3) into (2)

$$\mu = \bar{\mathbf{x}} \pm t \frac{S(\mathbf{x})}{\sqrt{N}} \qquad \dots \dots (4)$$

95% confidence range of the mean

An example of the calculation is given below for illustration purpose: Reflectance data are taken from the 16 test specimens on Cotton STC washed in 10% phosphate and 133 ppm of water hardness.

$$x = 36.3, 35.0, 34.4, 34.2, 37.8, 32.9, 36.8, 33.3, 34.7, 35.5, 35.9, 35.1, 35.9, 36.3, 37.9, 36.0 \bar{x} = 35.5 S(x) = \sqrt{\frac{(x - \bar{x})^2}{N - 1}} = 1.41751 t.05, 15 = 2.131$$

Substituting the values into equation (5), 95% confidence range of the mean is: 36.3 to 34.7.

7.0 CONCLUSIONS

At the two lower levels of water hardness which were investigated (80 and 133 ppm - representing 65% of Canadian population):

(a) The presence of phosphate in excess of 5% (expressed as P_2O_5) in detergent formulations lowers their cleaning efficiency towards Dacron and Dacron/ cotton blends.

(b) At higher (>15%) concentration levels citrates and NTA improve the laundering efficiency of detergents towards Dacron and Dacron/cotton blends.

At the higher levels of water hardness (330 and 550 ppm): (c) Phosphates are significantly more effective than NTA or citrates, although at least 15% as P_2O_5 is needed to obtain satisfactory results.

(d) The efficiencies of some of the experimental formulations could probably be improved if emulsion stabilizers were included. This is particularly true of those containing NTA and citrate since phosphates themselves probably possess some emulsion stabilizing properties.

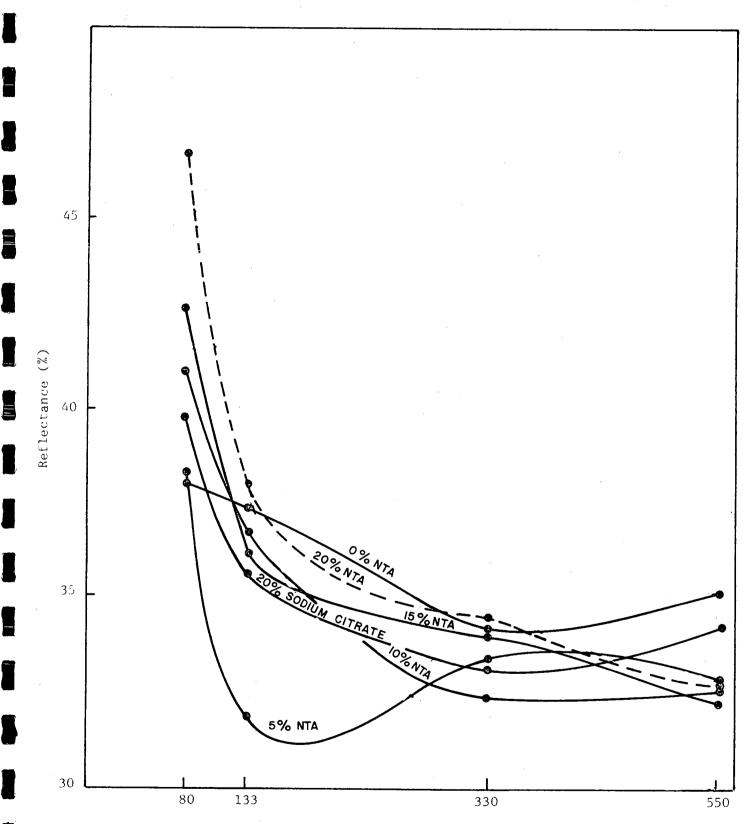
(e) NTA and Phosphate are markedly more effective than citrate, formulated with the same surfactant, in cleaning all cotton fabrics provided at least 15% is present. Little is gained by increasing the concentration beyond this figure except at the higher levels of water hardness.

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M.J. Williams Department of Textiles

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Water Hardness (ppm)

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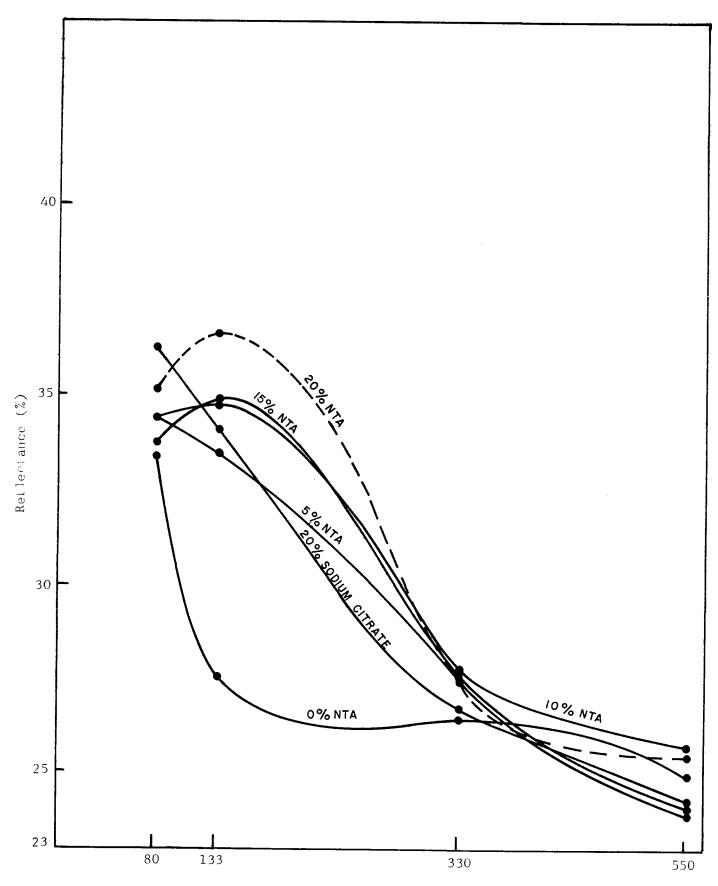
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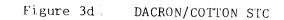
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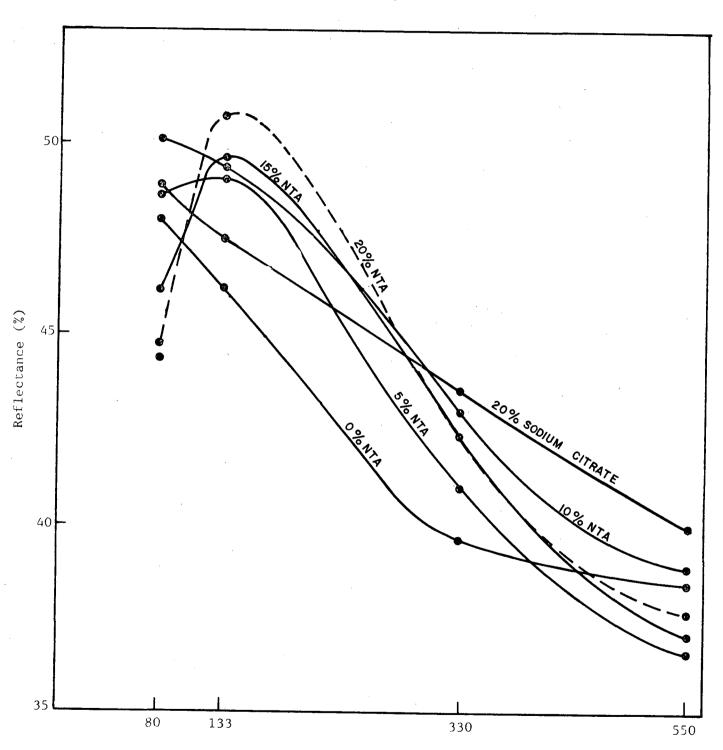
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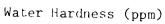




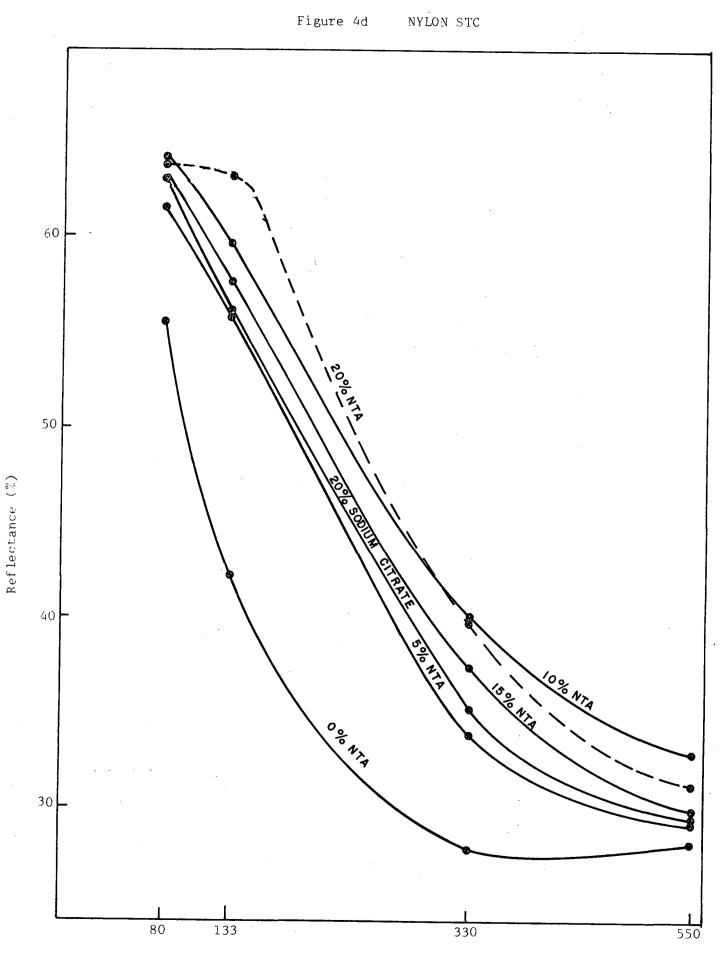
Water Hardness (ppm)

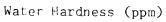




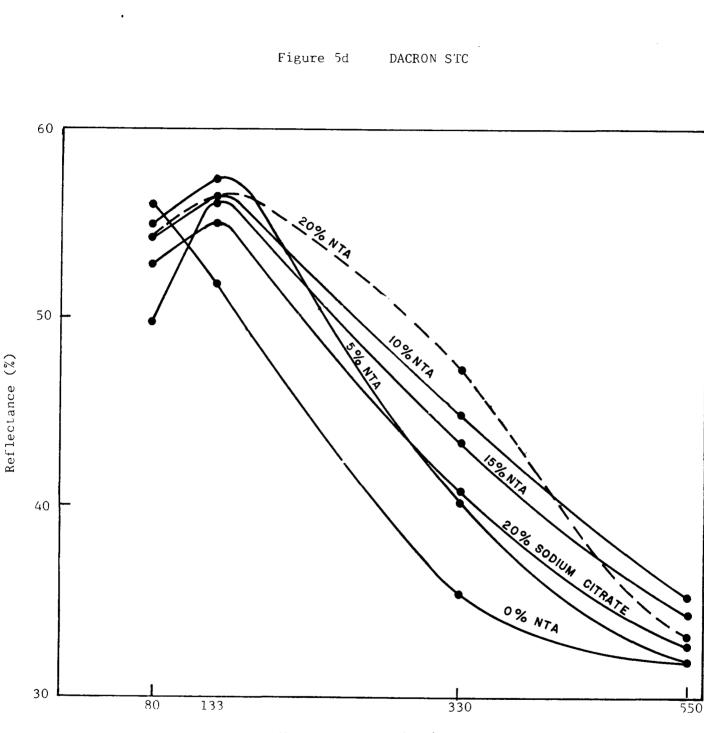


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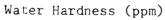


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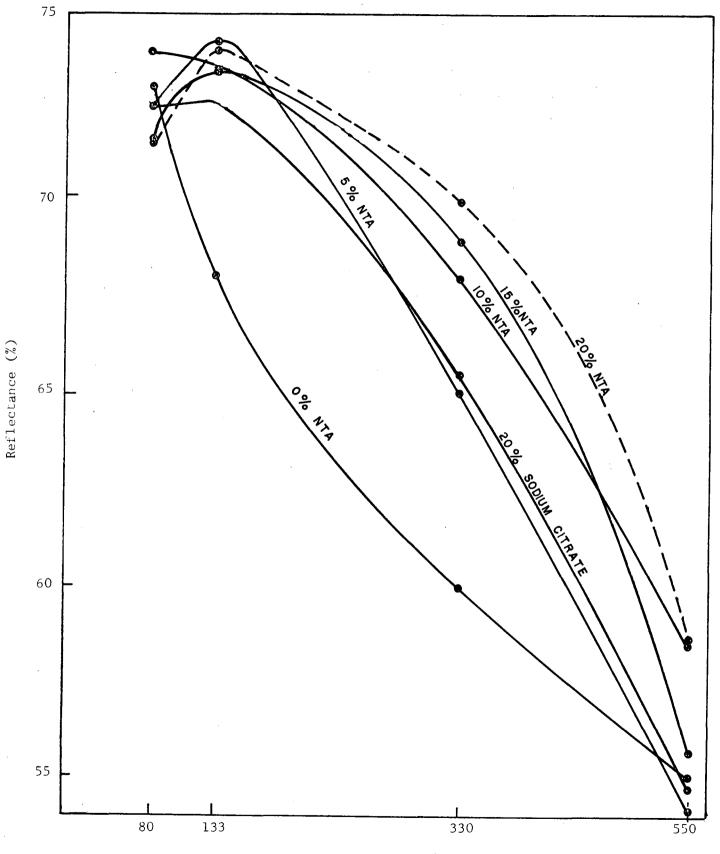
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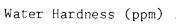
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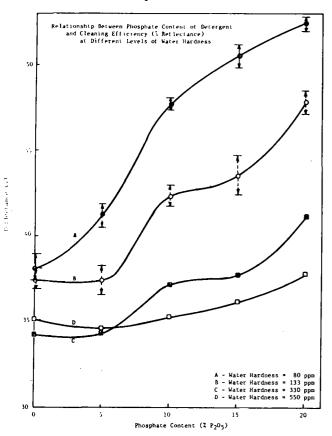
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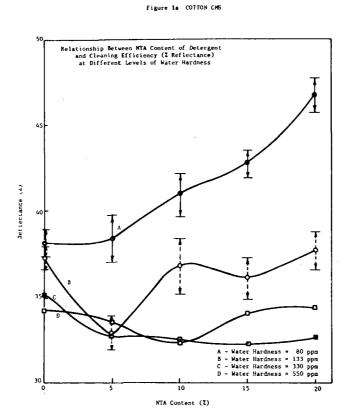


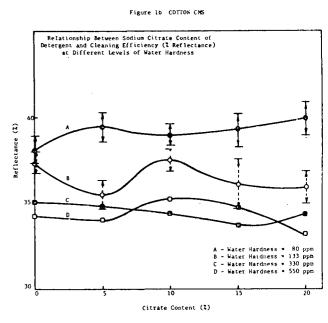


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Figure 1 COTION CMS

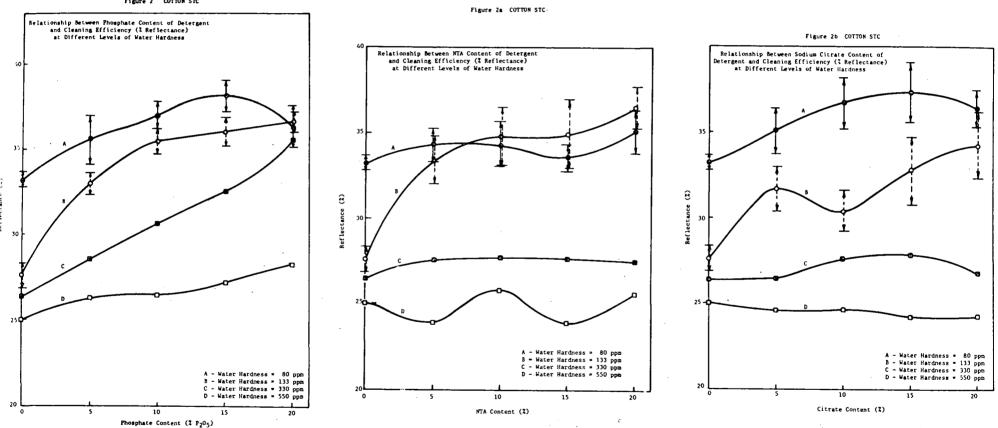






In above Figures ranges represent 95% confidence limits

FIG.1



In above Figures ranges represent 95% confidence limits

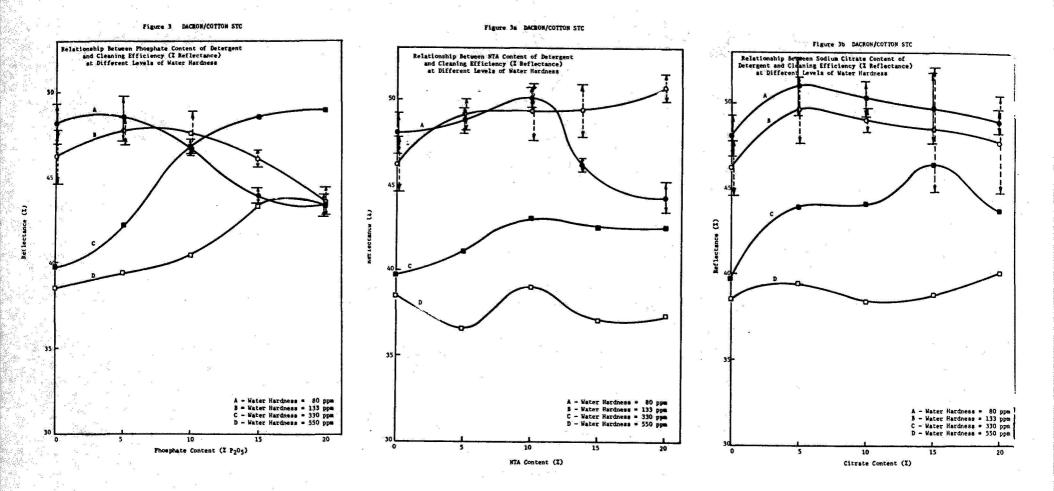
FIG. 2

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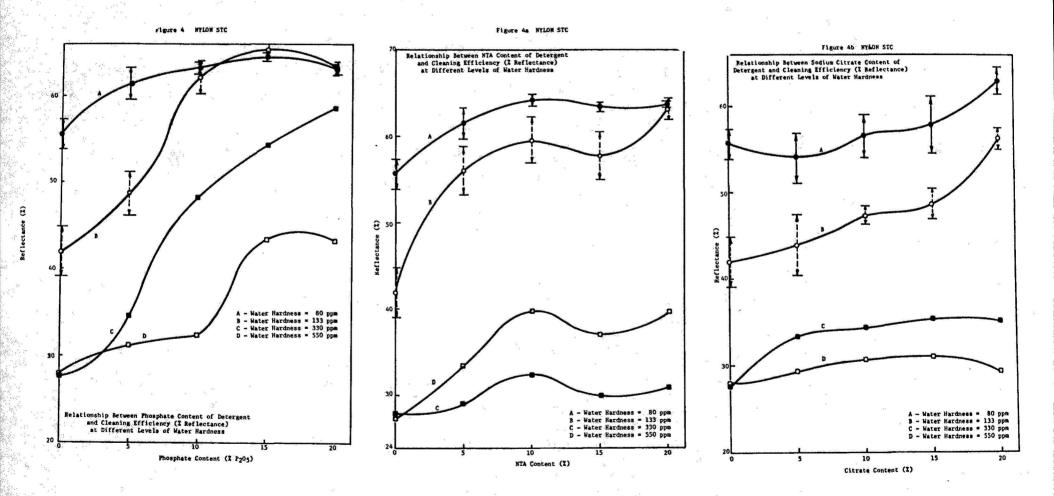
Figure 2 COTTON STC

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FIG.3







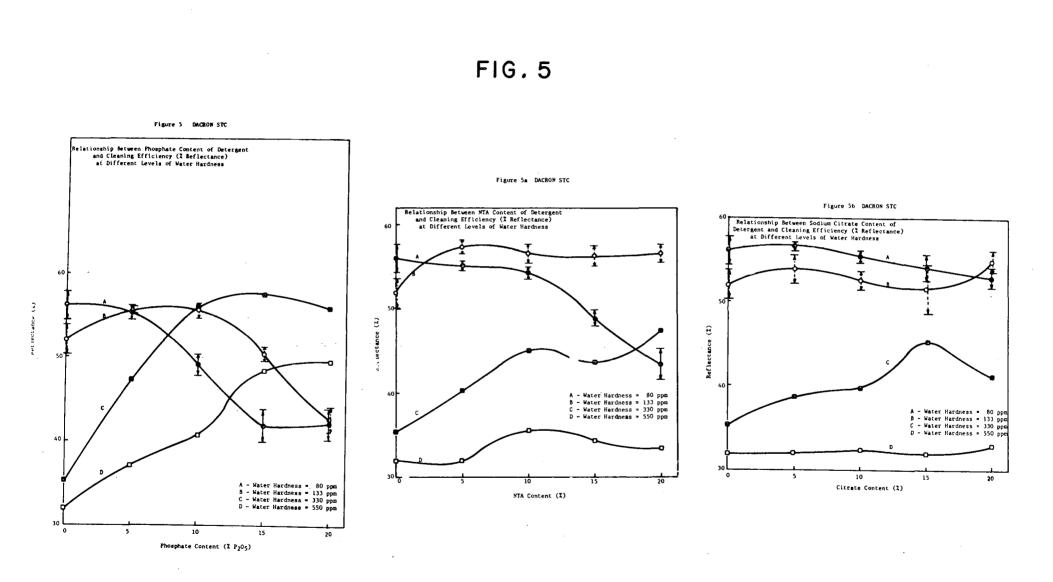
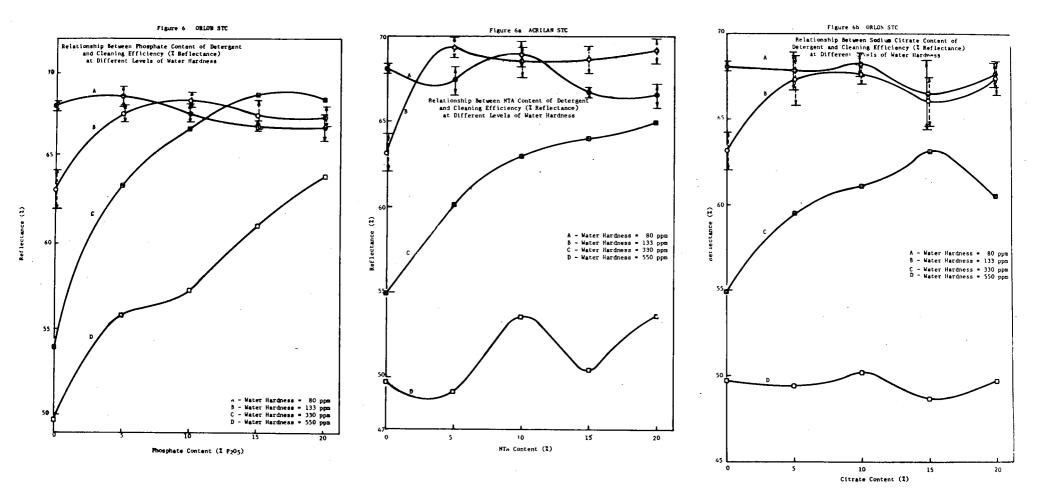


FIG.6



EVALUATION OF DETERGENT FORMULATIONS

Interim Report for Period Dec. 18, 1971 to Feb. 17, 1972

1.0 SUMMARY

The overall objective of this research program is to evaluate the relative efficiencies of different detergent formulations at different levels of water hardness. This interim report is concerned with the first phase of this study, and is specifically involved with the effect of variations in phosphate content. Several detergents, based on C.G.S.B. specifications, were prepared containing between 0 and 20% phosphate (expressed a P_2O_5). Their cleaning ability was compared by laundering a variety of soiled fabrics under standard washing conditions and instrumentally measuring the improvement in white light reflectivity.

2.0 MATERIALS

2.1 Soiled Fabrics

Artificially soiled fabrics were used in the tests. To include samples of the types of fabric, particularly with respect to fibre composition, that the consumer would normally encounter, the following were selected. The abbreviations which are used in subsequent parts of this report are given in brackets after the description of the soiled fabric*.

- All cotton fabric EMPA 112, soiled with cocoa-sweetened milk mixture representing a type of pigment-fat soil that often occurs on table linen. (COTTON CMS).
- (2) Cotton soil test cloth prepared to specification 51 S 47 (INT) Bureau of Ships, soiled with mixture containing ethyl cellulose, lamp black, hydrogenated vegetable oil, corn starch and oleic acid. (COTTON STC).
- (3) Polyester/cotton (65/35) fabric with durable press finish, soiled with same mixture as fabric (2). (DACRON/COTTON STC).

*All soiled fabrics were obtained from Testfabrics Inc., N.Y.

(4) Spun nylon fabric soiled with same mixture as fabric (2). (NYLON STC).

(5) Spun polyester fabric soiled with same mixture as fabric (2). (DACRON STC).

(6) Spun acrylic fabric soiled with the same mixture as fabric (2). (ORLON STC).

2.2 Detergents

The basic detergent formulation used throughout the study was one conforming to C.G.S.B. Provisional Standard for Detergent, Laundry: Power, Built 2-GP-115 P July 1970, with the exception that the phosphate was omitted and replaced by sodium sulphate. This formulation is shown below:

Sodium Alkyl Benzene Sulphonate (CEDEPON S-85 FLAKES)		
Chemical Developments of Canada Ltd.	-	25%
Sodium Carboxymethyl Cellulose (CMC D435) .		
Chemical Developments of Canada Ltd.	-	1%
Sodium Silicate (BRITESIL C-20)		
National Silicates Ltd.	-	10%
Sodium Sulphate (Anhydrous) B.D.H.	-	64%

Different levels of phosphate content were achieved by replacing the appropriate proportion of sodium sulphate with sodium tripolyphosphate (STPP). The five formulations investigated in this phase of the program are described below:

	Det	tergent	ication		
	A	B	<u> </u>	D	E
CEDEPON S-85	25%	25%	25%	25%	25%
CMC D435	1	1	1	1	1
BRITESIL C-20	10	10	10	10	10
SODIUM SULPHATE	64.0	55.2	46.5	37.7	29.0
STPP (as P ₂ 0 ₅)	0	5	10	15	20

2.3 Water Supply

In accordance with our Proposal P-982 all tests were carried out using 4 different hardness levels. Nominally these were chosen as 80, 135, 350 and 550 ppm expressed as $CaCO_3$. To obtain the required levels, adjustments were made either by suitable dilution with distilled water or by the addition of sufficient calcium chloride $(CaCl_22H_2^0)$ and magnesium sulphate $(MgSO_4, 7H_2^0)$ in the ratio of 3:1 when expressed in equivalents of calcium carbonate. The actual water hardness values were determined by removing aliquots from the wash liquors and analyzing by the Ethylanediamine Titrimetric Method (Method 122B Standard Methods for the Evaluation of Water and Wastewater).

The four levels of water hardness were calculated to be 80, 133, 330 and 550 ppm expressed as $CaCO_3$. These values are typical of the range of hardnesses that exist across Canada.

3.0 SPECIMENS AND REPLICATIONS

Each test specimen comprised a 30" \times 30" piece of undyed cotton broadcloth carrier fabric to which 4" \times 4" squares of the six artificially soiled fabrics were attached by stapling along the edges. Duplicate samples of each soiled material were included in each wash and the whole procedure was carried out by two technicians using separate washing machines. The experiment therefore provided results for four replications.

4.0 EXPERIMENTAL

4.1 Washing Procedure

All tests were carried out in Kenmore Model 600 automatic domestic washing machines having capacities of approximately 14 imperial gallons. The "regular" wash cycle was used with an initial wash liquor temperature of 147°F. When dilution with distilled water was needed the required volume was preheated to this temperature. Sufficient undyed cotton was added to make up a 6 lb load. The test specimens were subsequently ironed to dryness.

4.2 Assessment of Soil Removal

Reflectance readings of the original soiled and the laundered swatches were made at four different locations on each swatch using an incandescent light source with a narrow band filter allowing light to be transmitted at 570 nm wavelength. The measurements were made on a Zeiss Elrepho Photometer, calibration being made against a standard block of magnesium oxide representing 100% reflectance. 5.0 RESULTS

The mean white light reflectance values after laundering the soiled fabrics with detergents of different phosphate content at different levels of water hardness are shown in Tables I to VI.

		Wat	ter Hardı	ness (ppr	n)
<u>%</u> P ₂ 05	Detergent	80	133	330	550
0	А	38.1%	37.3%	34.2%	35.1%
5	В	41.2	37.3	34.6	34.5
10	С	47.7	42.3	37.1	35.3
15	D D	50.5	43.5	37.7 .	36.1
20	E	524	47.8	41.1	378

Table I

Reflectance Values for COTTON CMS (Original Reflectance = 34.8%)

Table II

<u>Ref lectance</u>	Values for COTTON	_STC (<u>Original</u>	Reflecta	nce = 18	<u>.1%)</u>					
		Water Hardness (ppm)									
<u>% P205</u>	Detergent	80	_133	330	550						
0	А	33.2%	27.6%	26.4%	25.0%						
5	В	35.7	33.0	28.6	26.3						
10	C	37.0	35.5	30.7	26.5						
15	D	38.2	38.2	32.6	27.2						
20	E	36.4	36.7	35.6	28.3						

Table III

Reflectance	Values for	 DACRON/COTTON 	STC	(Original	Reflectance	= 28.2%

		Water Hardness (ppm)							
<u>% P205</u>	Detergent	80	133	330	550				
0	A	48.1	46.2	39.7	38.5				
5	В	48.5	47.7	42.1	39.4				
10	С	46.7	47.6	46.8	40.4				
15	D	43.9	46.1	48.5	43.3				
20	E	43.4	43.6	48.9	43.4				

Reflectance	Values for NYLON	STC ((Original	Reflecta	nce = 24.2%					
		Water Hardness (ppm)								
<u>% P205</u>	Detergent	80	133	330	550					
0	Α	55.6%	41.9 %	27.6%	27.9%					
5	В	61.4	46.2	34.5	31.1					
10	С	63.2	62.1	48.2	32.3					
15	D	64.5	65.4	54.4	43.4					
20	Е	63.2	63.2	58.6	43.2					

Table IV

Table V

Reflectance Values for DACRON STC (Original Reflectance = 27.9%)

		Water Hardness (ppm)								
<u>% P205</u>	Detergent	80	133	330	550					
0	А	56.1%	51.9%	35.3%	31.9%					
5	В	55.3	55.6	47.3	37.1					
10	. C	49.1	55.7	56.0	40.7					
15	D	41.9	50.4	57.4	48.4					
20 .	E	42.2	42.7	55.9	49.4					

Reflectance	Values for ORLO	N STC (0	riginal 1	Reflecta	nce = 41.9%)
		Wa	ter Hard	ness (pp	m)
<u>% P205</u>	Detergent	80	133	330	550
0	А	68.1%	63.1%	54.9%	49.7%
· 5	B ·	68.6	67.6	63.4	55.8
10	С	67.6	68.4	66.7	56.3
15	D	66.9	67.5	68.7	61.0
20	E	66.8	67.4	68.4	63.9

The results in the above tables are recorded graphically in Figures 1 - 6.

Table VI

Figure 1 COTTON CMS

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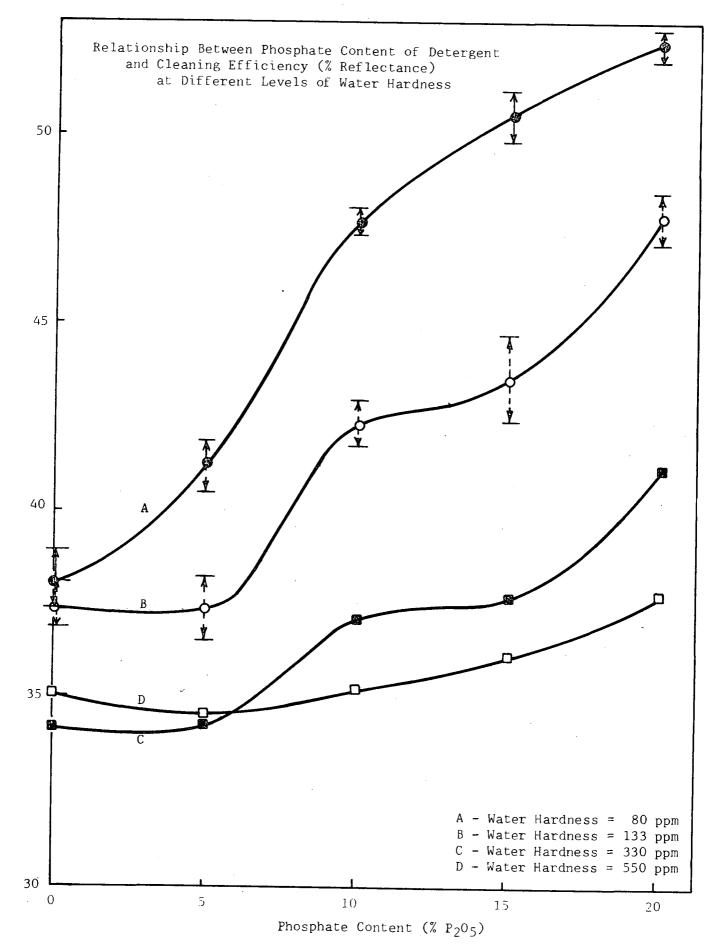
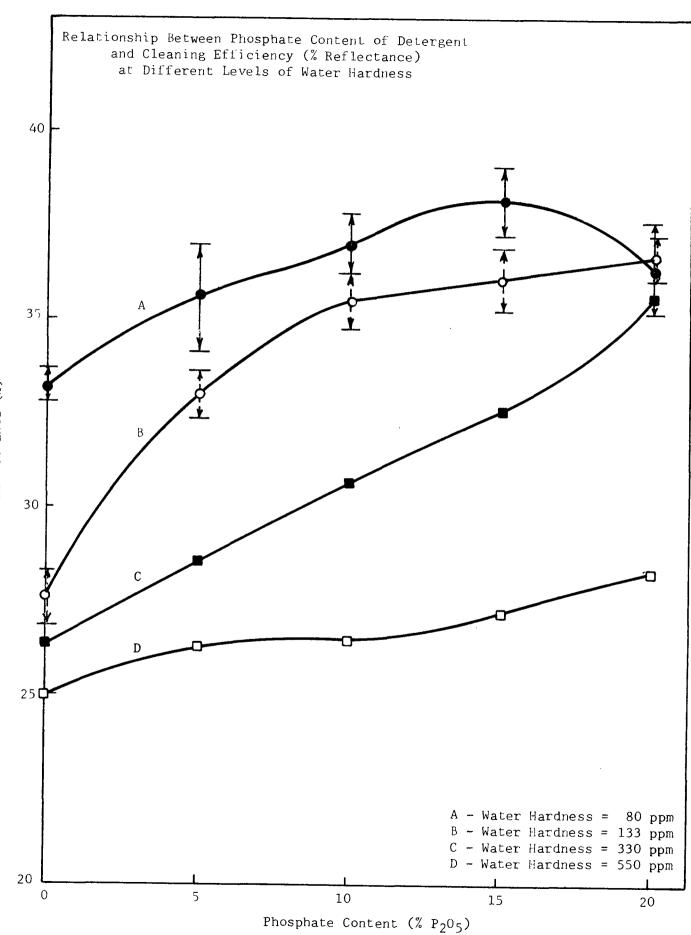
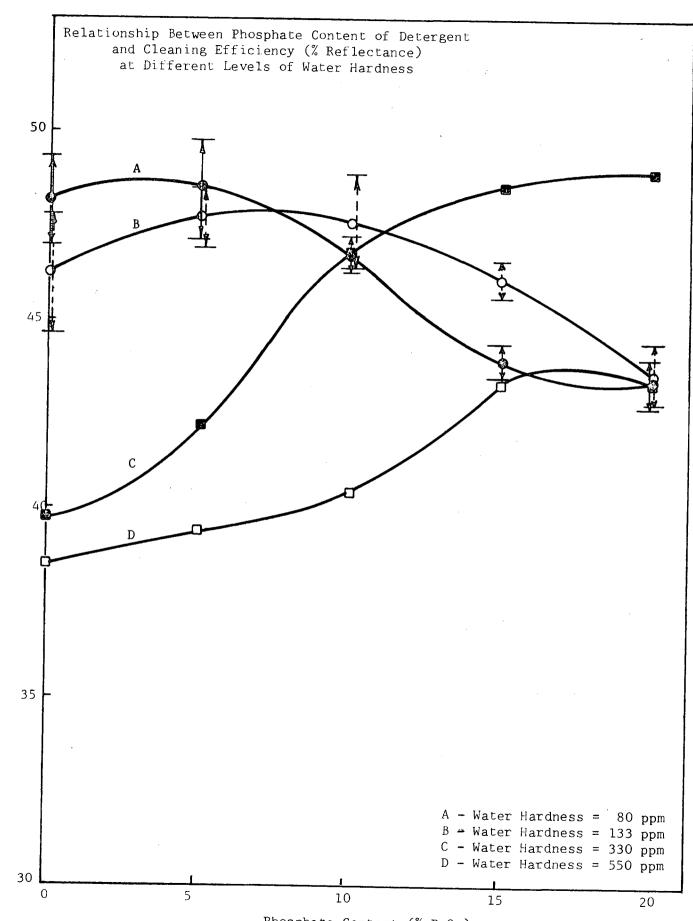


Figure 2 COTTON STC

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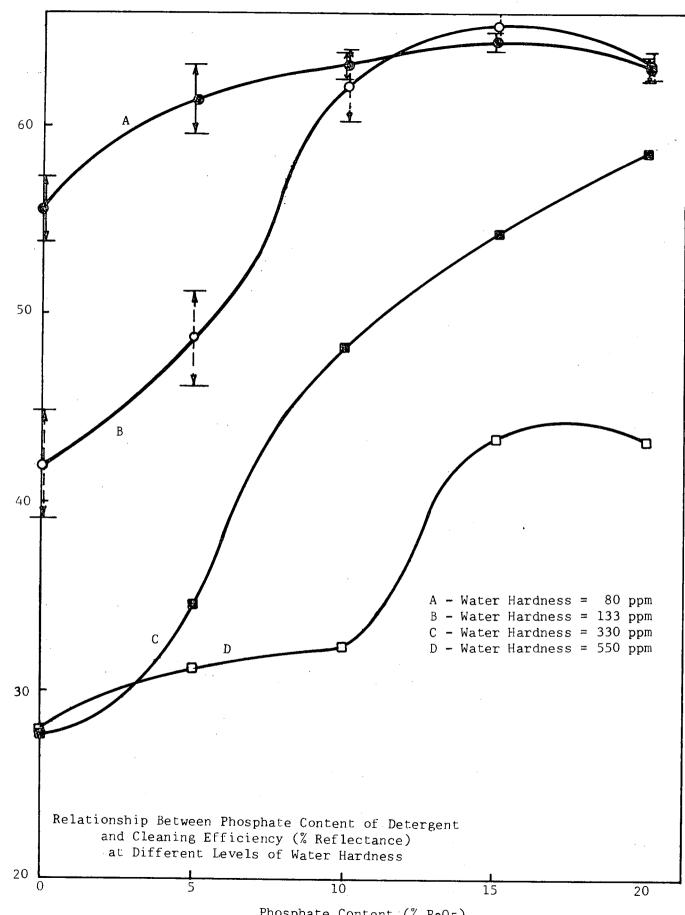
- 21 -Figure 3 DACRON/COTTON STC



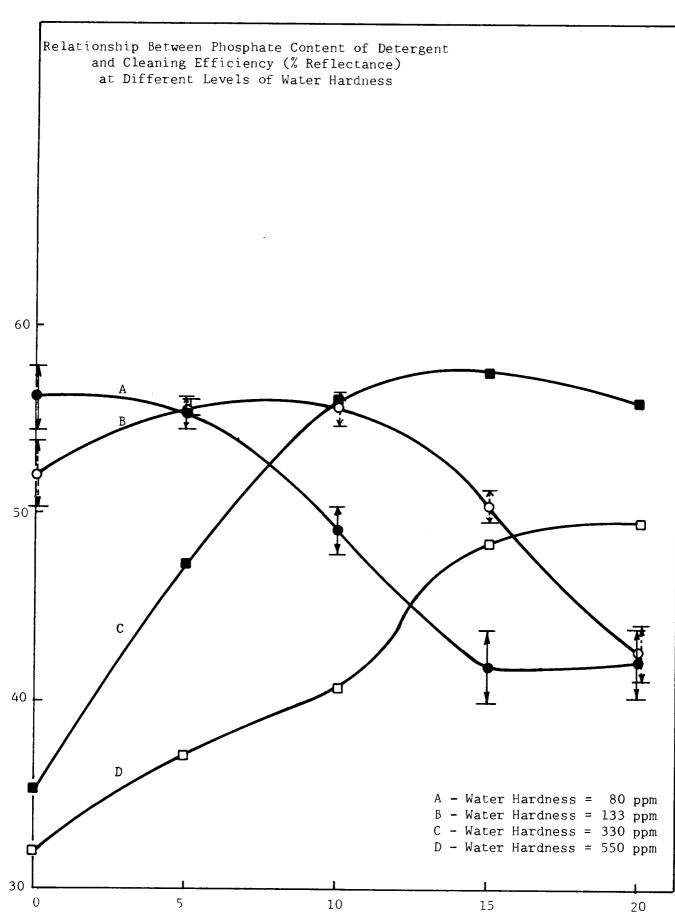
Phosphate Content (% P_2O_5)

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- 22 -Figure 4 NYLON STC



Phosphate Content (% P205)

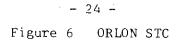


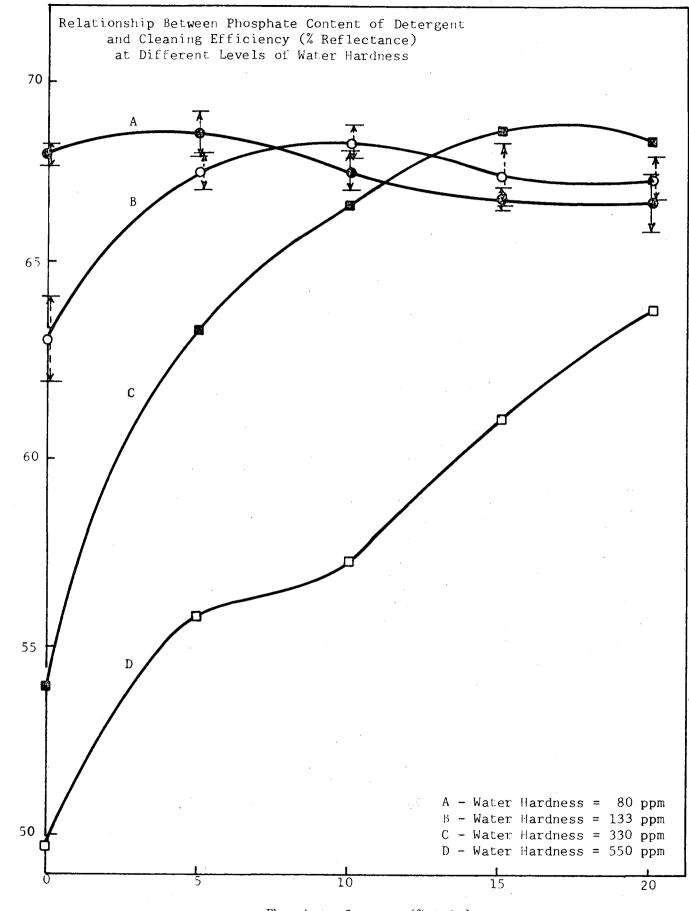
Phosphate Content (% P205)

Retlectanco (%)

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Figure 5 DACRON STC





Rellectance (%)

Phosphate Content (% P_2O_5)

EVALUATION OF DETERGENT FORMULATIONS

Interim Report for Period February 18 - March 9, 1972

1.0 SUMMARY

This part of the overall research program is concerned with the effect of variation in NTA content on detergent efficiency. Several detergents, based on CGSB specifications, were prepared containing up to and including 20% NTA. Their cleaning ability was compared by laundering swatches of the soiled fabrics described in our interim report of February 17, 1972. The water supplies used, the specimens and replications, and the assessment of soil removal were also as described in that report.

The NTA was obtained from the Clough Chemical Company Limited, Quebec, and the five formulations investigated in this phase of the program are described below:

Detergent Identification

	A	F	G	<u> </u>	
CEDEPON S-85	25%	25%	25%	25%	25%
CMC D435	1	1	1	1	1
BRITESIL C-20	10	10	10	10	10
SODIUM SULPHATE	64	59	54	49	44
NTA	0	5	10	15	20

2.0 RESULTS

The mean white light reflectance values after laundering the soiled fabrics with detergents of different NTA content at different levels of water hardness are shown in Tables Ia - VIa.

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Reflecta	ance Value	es for COTTON	CMS (Ori	iginal 1	Reflecta	ance = 34	.8%)
			Wate	er Hardi	ness (pp	om)	
	<u>% NTA</u>	Detergent	80	133	330	550	
	0	А	38.1	37.3	34.2	35.1	
	5	F	38.4	32.9	33.5	32.7	
	10	G	41.0	36.8	32.3	32.5	
	15	Н	42.8	36.1	34.0	32.2	
	20	I	46.7	37.7	34.3	32.6	
						,	

Table Ia

Table IIa

1

Reflecta	nce Value	s for COTTON S	TC (Ori	ginal R	eflecta	nce = 18.1%)
	a .		Wate	r Hardn	ess (pp	m)
	<u>% NTA</u>	Detergent	80	<u>133</u>	330	550
	0	А	33.2	27.6	26.4	25.0
	5	F	34.3	33.3	27.5	23.9
	10	G	34.3	34.8	27.7	25.8
	15	Н	33.6	34.9	27.6	24.0
	20	I	35.1	36.5	27.5	25.6

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Table IIIa

Reflectance Values for DACRON/COTTON STC (Original Reflectance = 28.2%)

		Wate	Water Hardness (ppm)						
<u>% NTA</u>	Detergent	80	133	330	550				
0	А	48.1	46.2	39.7	38.5				
5	F	48.8	49.1	41.1	36.6				
10	G	50.1	49.3	43.1	39.0				
15	H	46.2	49.4	42.5	37.1				
20	I	44.3	50.7	42,5	37.4				

Table IVa

Reflectance Values for NYLON STC (Original Reflectance = 24.2%)

		Wate	Water Hardness (ppm)						
<u>% NTA</u>	Detergent	80	133	330	550				
0	А	55.6	41.9	27.6	27.9				
5	F	61.7	55.9	33.3	29.1				
10	G	64.1	59.6	39.9	32.4				
15	H .	63.4	57.7	37.1	30.1				
20	I	63.7	63.4	39.8	31.0				
-	TIDE	63.4	62.3	62.0	50.1				

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Reflectance Va	lues for DACRON	STC (01	riginal	Ref lect	tance =	27.9%)
·		Wate	er Hardı	ness (pj	pm)	
<u>% NTA</u>	Detergent	80	133	_330	550	
0	А	56.1	51.9	35.3	31.9	
5	F	55.1	57.4	40.3	31.9	
10	G	54.3	56.6	45.0	35.6	
15	Н	48.8	56.3	43.7	34.3	
20	I	43.3	56.6	47.4	33.4	
					;	
					3	

Table Va

Table VIa

Reflectance	Values for ORLON	STC	(Original	Reflec	tance =	41.9%)
		Wa	ater Hardr	ness (p	opm)	
<u>% NTA</u>	Detergent	80	133	330	550	
0	Α	68.	1 63.1	54.9	49.7	
5	F	67.4	4 69.3	60.1	49.1	
10	G	68.9	9 68.5	62.9	53.5	
15	Н	66.7	7 68.6	64.0	50.5	
20	I	66.5	5 69.1	64.9	53.6	

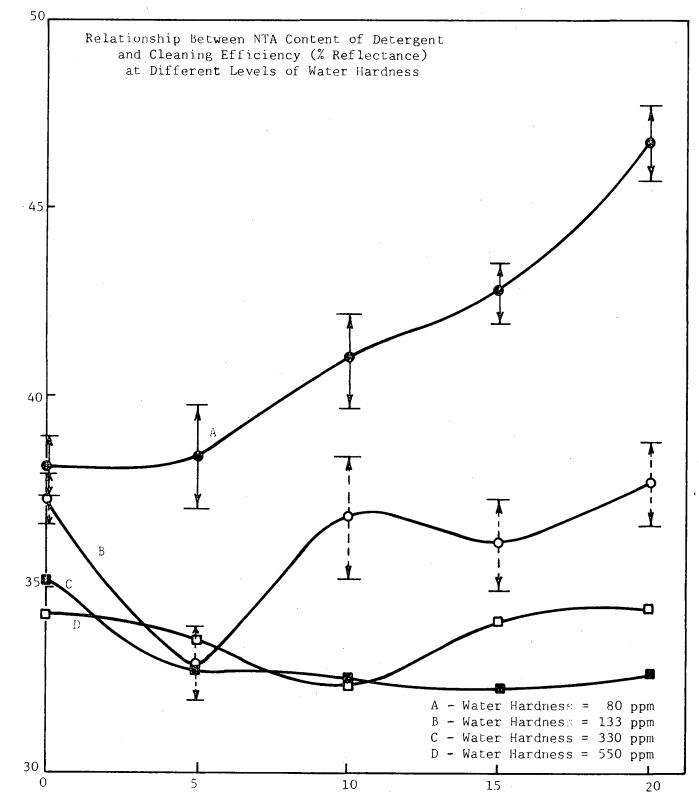
The results in the above tables are shown graphically in Figures

la to 6a.

1n

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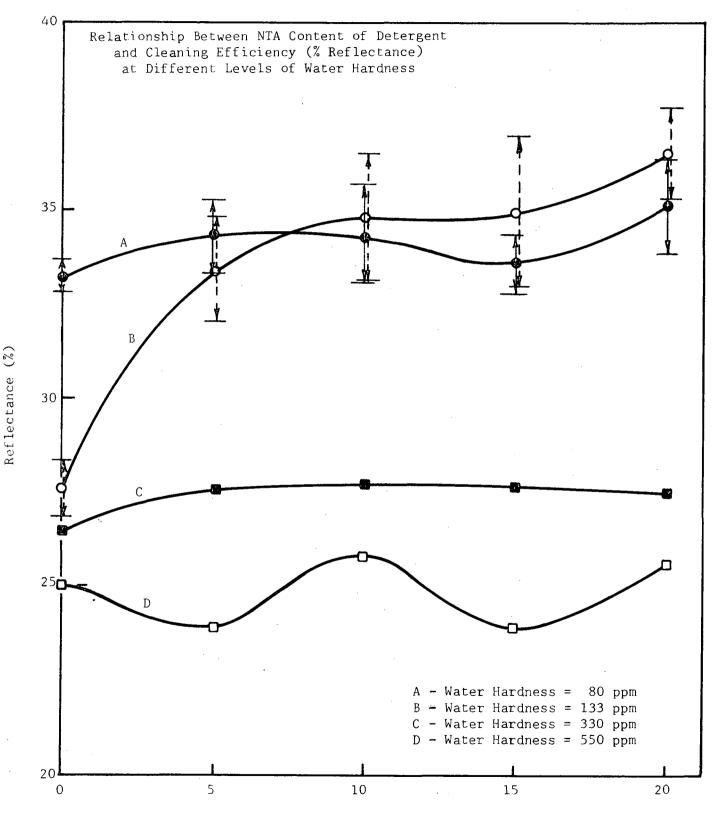
Reflectance (%)

NTA Content (%)

- 29 -

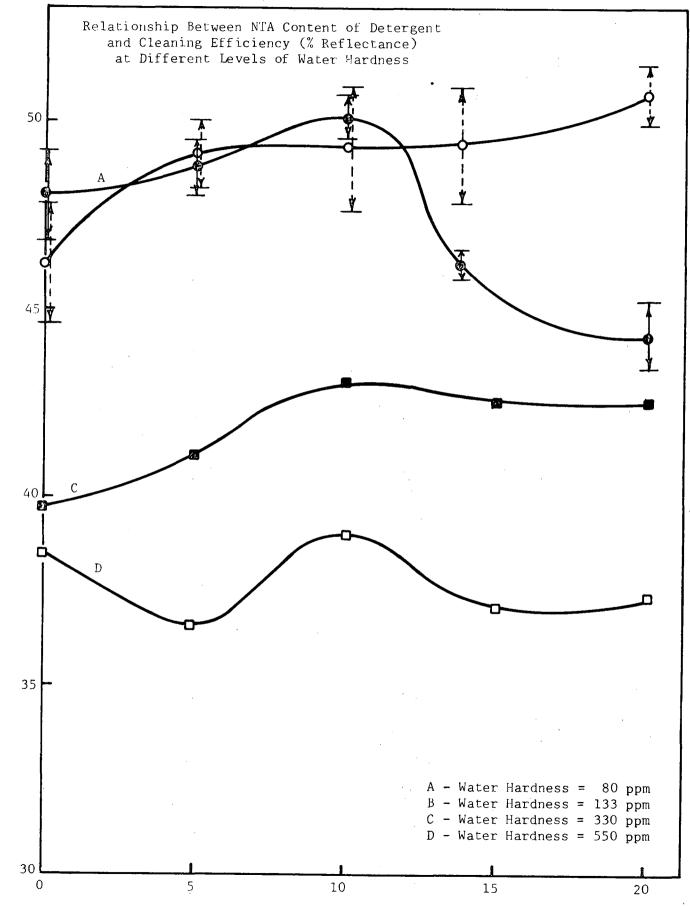
Figure 2a COTTON STC

- 30 -



NTA Content (%)

Figure 3a DACRON/COTTON STC

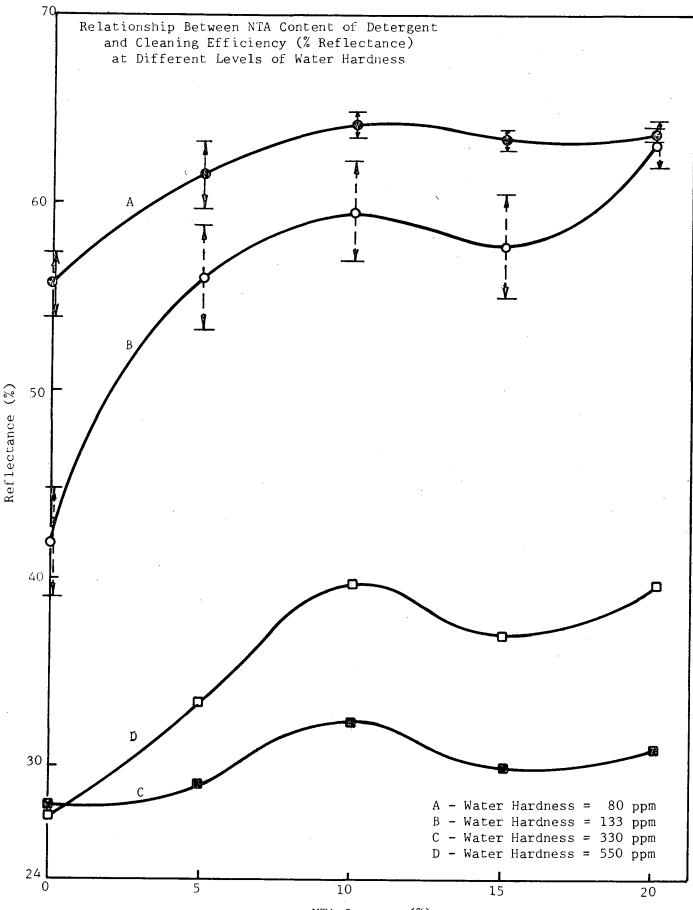


NTA Content (%)

Reflectance (%)

- 31 -

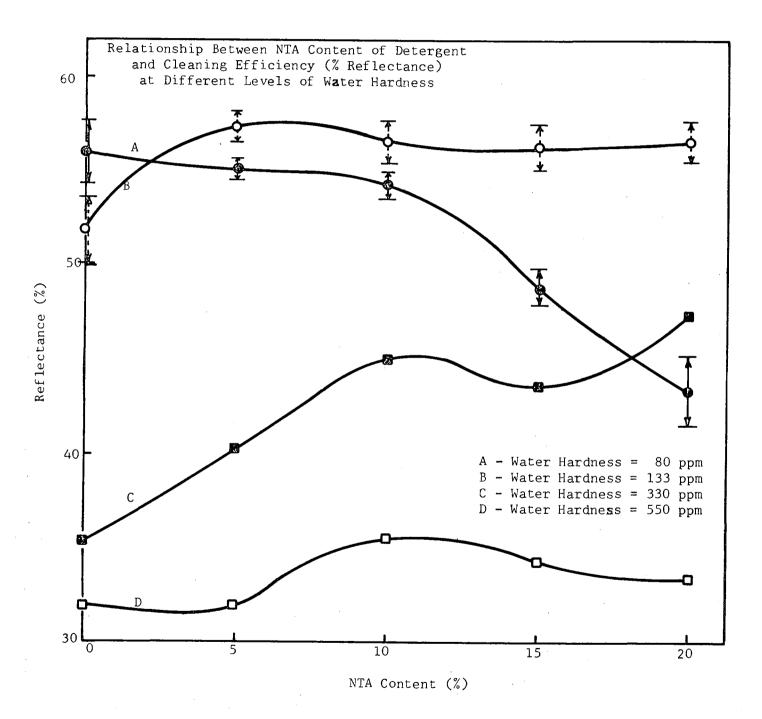
Figure 4a NYLON STC



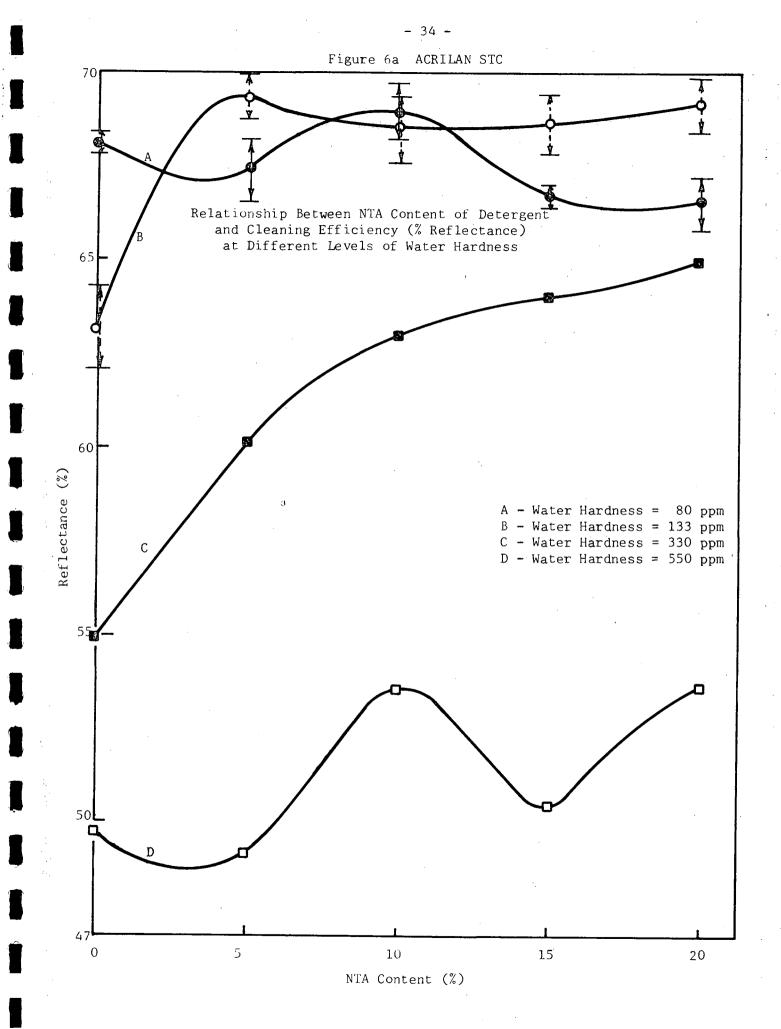
NTA Content (%)

- 32 -

Figure 5a DACRON STC



- 33 -



EVALUATION OF DETERGENT FORMULATIONS

Interim Report for Period March 10 - March 30, 1972

1.0 Summary

This part of the overall research program is concerned with the effect of variation in sodium citrate content on detergent efficiency. Several detergents, whose formulations were based on C.G.S.B. specifications, were prepared containing up to and including 20% sodium citrate. The cleaning ability of these detergents was compared by laundering swatches of the soiled fabrics described in our Interim Report of February 17, 1972. The degrees of water hardness, and the procedure for carrying out the tests and assessing soil removal were also the same as previously described.

The sodium citrate used was obtained from the J.T. Baker Chemical Company and the compositions of the five formulations investigated in this phase of the program are given below:

		Detergen	t Identi:	fication	
	_A		K		M
CEDEPON S-85	25%	25%	25%	25%	25%
CMC D435	1	1	1	1	1
BRITESIL C-20	10	10	10	10	10
SODIUM SULPHATE	64	59	54	49	. 44
SODIUM CITRATE	0	5	10	15	20

2.0 RESULTS

The mean white light reflectance values of the soiled fabrics after laundering with detergents of different sodium citrate content at different levels of water hardness are shown in Tables Ib to VIb.

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Reflectance Value	s for COTTON	CMS (Ori	ginal R	eflecta	nce = 34.8%)
% Sodium Citrate	Detergent	Wat	er Hard	<u>ness (p</u> 330	opm) 550
0	А	38.1%	37.3%	34.2%	35.1%
5	J	39.4	35.5	34.0	34.8
10	K	38.9	37.5	35.2	34.4
15	L	39.3	36.1	34.7	33.7
20	М	39.9	35.9	33.2	34.3

Table Ib

Table IIb

Reflectance Values for COTTON STC (Original Reflectance = 18.1%)

% Sodium		Water Hardness (ppm)						
Citrate	Detergent	80	133	_330	550			
0	A	33.2%	27.6%	26.4%	25.0%			
5	J	35.1	31.7	26.4	24.6			
10	K	36.7	30.4	27.6	24.6			
15	· L	37.3	32.8	27.8	24.2			
20	Μ	36.4	34.3	26.8	24.3			

Table	Ι	Ι	Ι	t
-------	---	---	---	---

Reflectance Valu	es for DACRON/COTT	CON STC	(Origin	al Refl	ectance	= 28.2%)
% Sodium		Wat	er Hard	lness (p	opm)	
Citrate	Detergent	80	133	330	550	
0	Α	48.1%	46.2%	39.7%	38.5%	
5	J	51.0	49.6	43.9	39.4	
10	К	50.3	49.0	44.1	38.4	
15	L	49.7	48.5	46.4	38.8	
20	М	49.0	47.7	43.7	40.1	

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Reflectance Val	lues for NYLON	STC (Ori	ginal R	eflecta	nce = 2	4.2%
% Sodium		Wat	er Hard	ness (p	pm)	
Citrate	Detergent	80	133	330	550	
0	А	55.6%	41.9%	27.6%	27.9%	
5	J	54.0	43.8	33.3	29.2	
10	К	56.5	47.3	34.4	30.7	
15	L	57.8	48.6	35.3	30.9	
20	М	62.9	56.3	35.1	29.3	

Table IVb

- 37 -

Table Vb

Reflectance Values for DACRON STC (Original Reflectance = 27.9%)

% Sodium		Water Hardness (ppm)						
Citrate	Detergent	80	133	330	550			
0	А	56.1%	51.9%	35.3%	31.9%			
5	J	56.6	53.8	38.6	32.0			
10	K	55.4	52.5	39.6	32.4			
15	L	54.1	51.6	45.1	31.9			
20	М	53.0	54.9	41.2	3 2. 9			

Table VIb

Reflectance	Values for	ORLON STC	(Original	Reflectance	= 41.9%

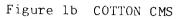
% Sodium		Water Hardness (ppm)						
Citrate	Detergent	80	133	330	550			
0	Α	68.1%	63.1%	54.9%	49.7%			
5	J	67.8	67.3	59.5	49.4			
10	К	68.2	67.6	61.1	50.2			
15	L	66.1	66.5	63.1	48.7			
20	Μ	67.4	67.6	60.5	49.7			

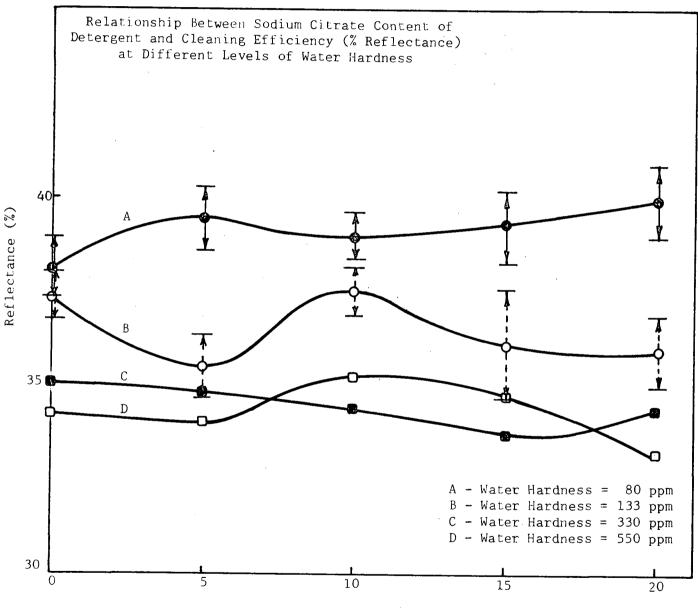
The results of the above tables are shown graphically in Figures 1b to 6b.

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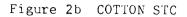
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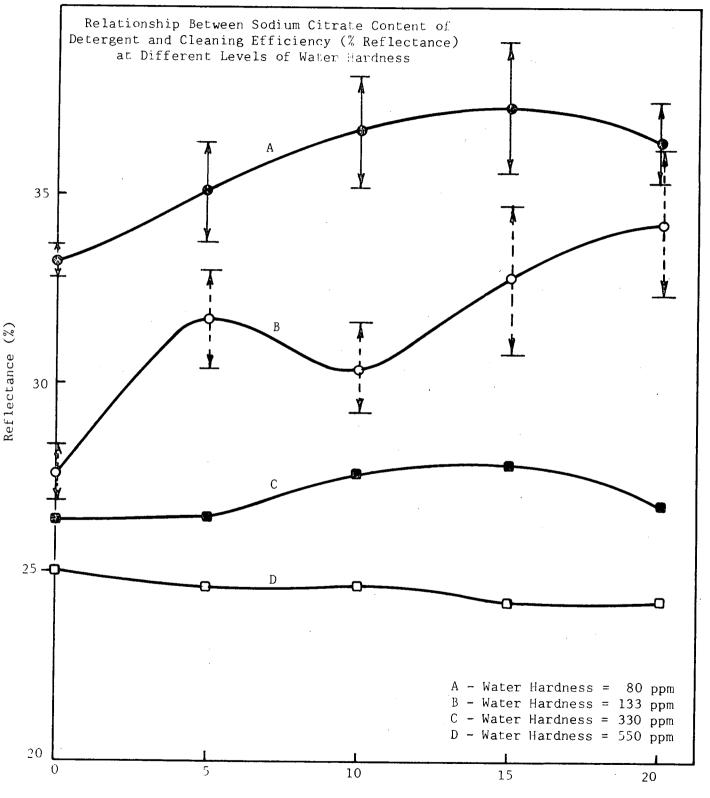
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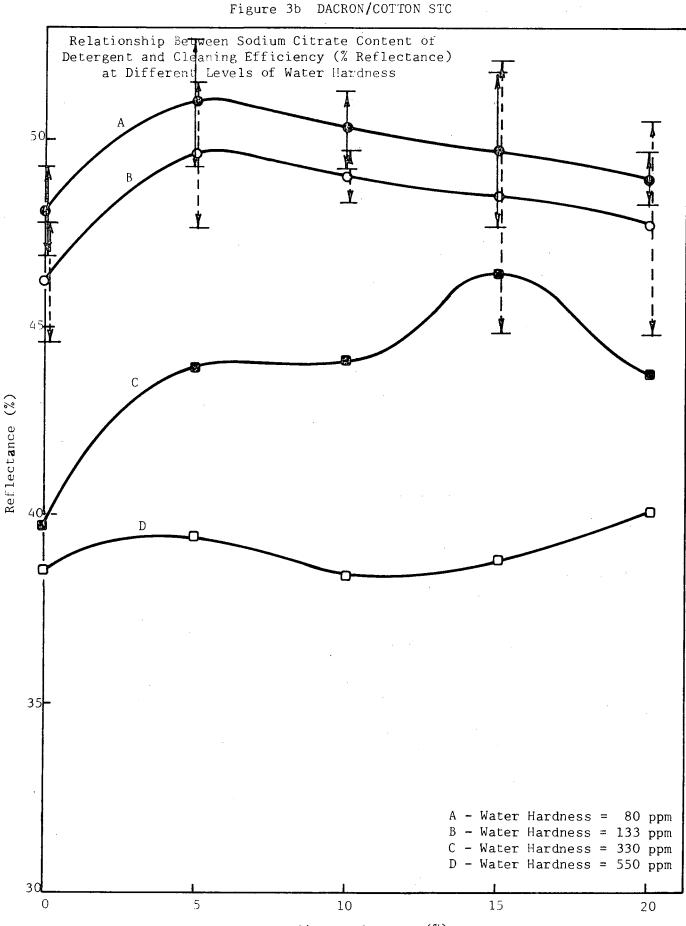
Citrate Content (%)





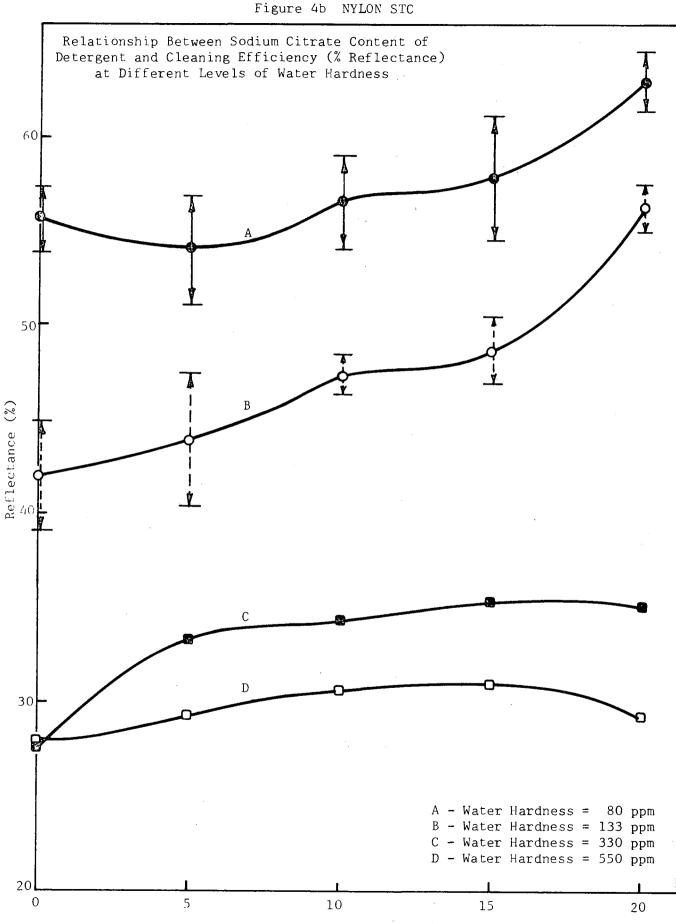


- 39 -



- 40 -

Citrate Content (%)



Citrate Content (%)

- 41 -

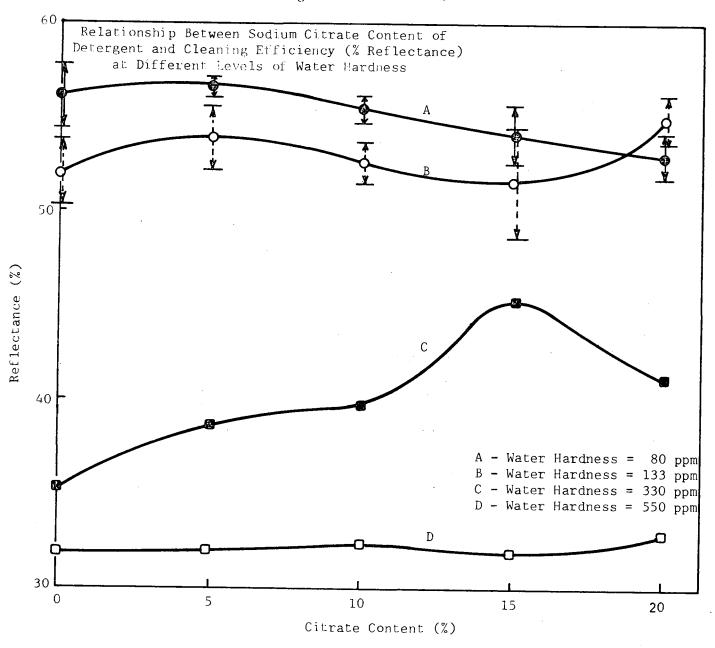
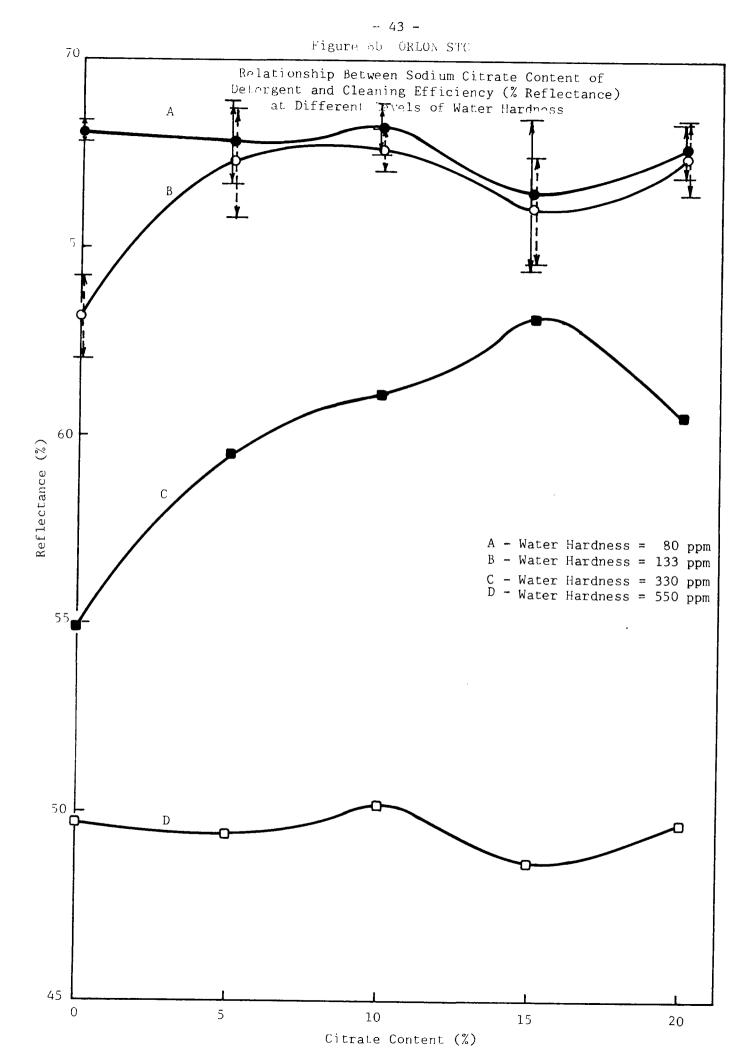


Figure 5b DACRON STC

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Evaluation of Detergent Formulations

Interim Report for Period April 1 - April 28, 1972

1.0 Summary

This part of the overall research program is concerned with the effect of variation in component blends of NTA, phosphate and citrate on detergency efficiency. Several detergents, based on CGSB specifications, were prepared containing between 5 and 15% of each component in the blend. Their cleaning ability was compared by laundering swatches of the soiled fabrics described in our interim report of February 17, 1972. The water supplies used, the specimens and replications, and the assessment of soil removal were also as described in that report.

The twelve formulations investigated in this phase of the program are described below.

	E	<u> </u>	<u>M</u>	<u>N</u>	_0	P	Q	R	<u> </u>	Т	U	v
Cedepon S-85	25%	25%	25%	25%	25%	25%	25%	25%	25%	25%	25%	25%
CMC D435	1	1	1	1	1	1	1	1	1	1	1	1
Britesil C-20	10	10	10	10	10	10	10	10	10	10	10	10
Sodium Sulphate	29.0	44.0	44.0	40.2	36.5	32.7	40.2	36.5	32.7	44	44	44
NTA	0	20	0	0	0	0	15	10	5	5	10	15
STPP (as P ₂ O ₅)	20	0	0	5	10	15	5	10	15	0	0	0
Sodium Citrate	0	0	20	15	10	5	0	0	0	15	10	5

Detergent Idenitifcation

2.0 Results

The mean white light reflectance values after laundering the soiled fabrics with detergents of different NTA, phosphate and citrate content at different levels of water hardness are shown in Tables Ic to VIc.

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Ta	h	16	Т	с
±α	v	TC		ັ

Reflectance Values for Cotton CMS (Original Reflectance = 34.8%)								
	% Phosphate		Wate	er Hardı	ness (pj	om)		
<u>% NTA</u>	(as P205)	Detergent	80	133	330	550		
20	0	I	46.7	37.7	34.3	32.6		
15	5	Q	41.8	37.2	33.3	32.9		
10	10	R	42.5	38.9	34.0	33.7		
5	15	S	43.3	41.7	36.0	33.8		
0	20	E	52.4	47.8	41.1	37.8		

<u>% NTA</u>	% Citrate	Detergent	80	133	330	550
20	0	I	46.7	37.7	34.3	32.6
15	5	V	41.3	36.4	33.0	33.4
10	10	U	41.5	35.0	32.4	31.9
5	15	Т	39.1	34.8	33.4	32.4
0	20	М	39.9	35.9	33.2	34.3

/0						
Phosphate	% Citrate	Detergent	80	133	330	550
0	20	М	39.9	35.9	33.2	34.3
5	15	W	41.3	39.3	34.9	33.7
10	10	0	42.8	40.5	36.0	34.4
15	5	Р	44.2	41.4	36.9	36.0
20	0	E	52.4	47.8	41.1	37.8

%

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	% Phosphate		Wat	er Hard	lness (p	pm)
<u>% NTA</u>	(as P ₂ 0 ₅)	Detergent	80	133	330	550
20	0	I	35.1	36.5	27.5	25.6
15	5	Q	38.5	38.1	28.8	25.5
10	10	R	36.1	37.8	30.1	25.7
5	15	S	37.9	36.1	31.2	27.4
0	20	Ε	36.4	36.7	35.6	28.4
<u>% N'FA</u>	% Citrate	Detergent	80	133	330	550
29	0	 I	35.1	36.5	27.5	25.6
15	5	v	37.7	35.6	28.3	25.0
10	10	U	39.7	34.5	27.9	25.6
5	15	Т	39.1	35.1	28.3	25.5
0	20	М	36.4	34.3	26.8	24.3

Phosphate	% Citrate	Detergent	80	133	330	550
0	20	М	36.4	34.3	26.8	24.3
5	15	Ν	37.6	37.8	29.0	24.7
10	10	0	39.0	40.0	31.2	25.2
15	5	Р	37.6	38.8	32.5	26.8
20	0	Е	36.4	36.7	35.6	28.4

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Τa	ble	IIc

Та	b	le	11	Ιc

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ardness (ppm)
3 330 550
.7 32.7 27.2
.8 33.3 29.1
.9 35.5 29.3
.7 36.5 33.5
.2 36.1 34.2
3 330 550
.7 32.7 27.2
7 32.9 27.3
3 32.9 27.2
1 33.2 27.5
.8 31.5 27.8
51.55 27.00
330 550
8 31.5 27.8
3 32.3 28.4
5 35.1 29.5
5 36.1 32.0
2 36.1 34.2

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	% Phosphate		Wate	er Hardı	ness (pj	om)
<u>% NTA</u>	$(as P_205)$	Detergent	80	133	330	550
20	0	I	63.7	63.4	39.8	31.0
15	5	Q	65.8	64.5	47.3	33.7
10	10	R	64.9	65.2	45.6	38.2
5	15	S	64.3	65.9	50.7	36.4
0	20	E	64.0	63.2	58.7	43.3
% NTA	% Citrate	Detergent	80	133	330_	550
20	0	I	63.7	63.4	39.8	31.0
15	5	V	66.2	60.0	43.2	35.5
10	10	U	65.6	54.8	34.9	31.3
5	15	Т	66.0	52.9	36.9	31.1
0	20	М	62.9	56.3	35.1	29.3

%						
Phosphate	<u>% Citrate</u>	Detergent	80	133	330	550
0	20	М	62.9	56.3	35.1	29.3
5	15	Ν	64.7	58.0	41.2	33.9
10	10	0	64.4	58.2	44.5	32.5
15	5	Р	64.6	55.2	48.0	35.6
20	0	E	64.0	63.2	58.7	43.3

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Reflectance Values for Nylon STC (Original Reflectance = 24.2%)

Τa	bl	е	Vc

Reflecta	ance V a lues for	Dacron STC (Or	iginal I	Reflect	ance = 2	25.0%)
%_ NTA	% Phosphate	_			iness (p	opm)
	(as P205)	Detergent	80	133	330	550
20	0	I	42.1	53.1	44.8	29.7
15	5	Q	44.6	50.5	51.0	38.1
10	10	R	40.7	45.7	51.9	40.7
5	15	S	35.6	41.9	51.2	48.3
0	20	E	33.4	35.6	50.4	51.4
	,					
% NTA	<u>% Citrate</u>	Detergent	80	133	330	550
20	0	I	42.1	53.1	44.8	29.7
15	5	v	47.5	50.5	42.9	30.1
10	10	U	48.2	49.5	42.6	28.7
5	15	Т	51.1	51.1	41.1	30.5
0	20	М	49.4	51.7	40.2	29.4
%						
hosphate	<u>% Citrate</u>	Detergent	80	133	330	550
0	20	М	49.4	51.7	40.2	29.4
5	15	N	47.0	50.7	44.9	34.3
10	10	0	42.7	49.5	51.1	41.6
15	5	Р	34.4	47.9	49.6	45.3
20	0	E	33.4	35.6	50.4	51.4

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Sphate Detergent P205) Detergent 0 I 5 Q 0 R 5 S 0 E	80 66.5 67.9 66.5 67.9 66.8	<u>133</u> 69.1 67.8 67.5 67.7 67.5	330 64.9 68.5 68.6 69.1	55.6 55.5
5 Q 0 R 5 S	67.9 66.5 67.9	67.8 67.5 67.7	68.5 68.6 69.1	53.6 55.6 55.5 64.4
0 R 5 S	66.5 67.9	67.5 67.7	68.6 69.1	55.5
5 S	67.9	67.7	69.1	
				64.4
0 E	66.8	67.5		
			68.4	63.9
				53.6
	80	133	330	550
				51.6
U C				53.5
5 Т				52.3
) М				49.7
()	0 I 5 V 0 U 5 T	0 I 66.5 5 V 66.9 0 U 67.3 5 T 69.2	0 I 66.5 69.1 5 V 66.9 70.1 0 U 67.3 68.8 5 T 69.2 69.6	0 I 66.5 69.1 64.9 5 V 66.9 70.1 67.5 0 U 67.3 68.8 65.6 5 T 69.2 69.6 64.7

/0						
Phosphate	<u>% Citrate</u>	Detergent	80	133	330	550
0	20	М	67.4	67.6	60.5	49.7
5	15	Ν	66.1	68.0	63.4	58.4
10	10	0	66.0	68.2	67.2	56.7
15	5	P	66.5	66.8	66.7	61.2
20	0	E	66.8	67.5	68.4	63.9

The results of the above tables are recorded graphically in Figures lc(I) to 6c(III).

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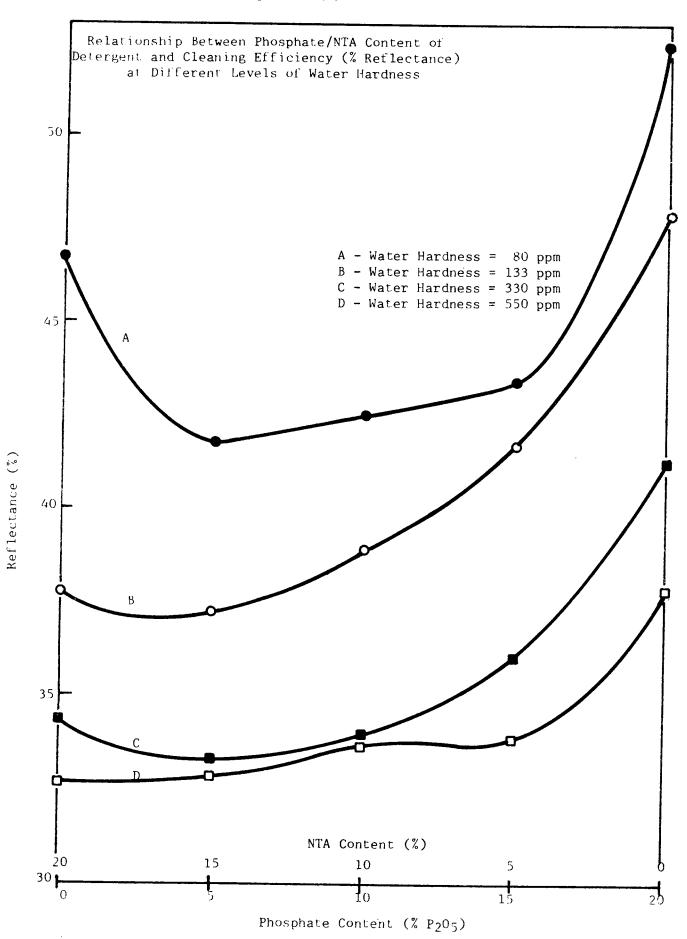
M.J. Williams Department of Textiles

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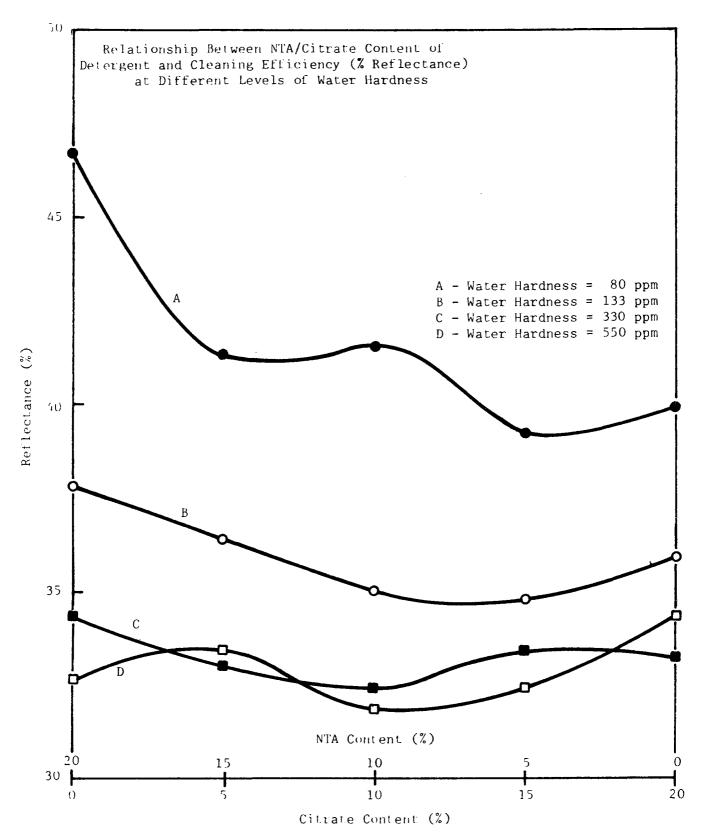
Table VIc

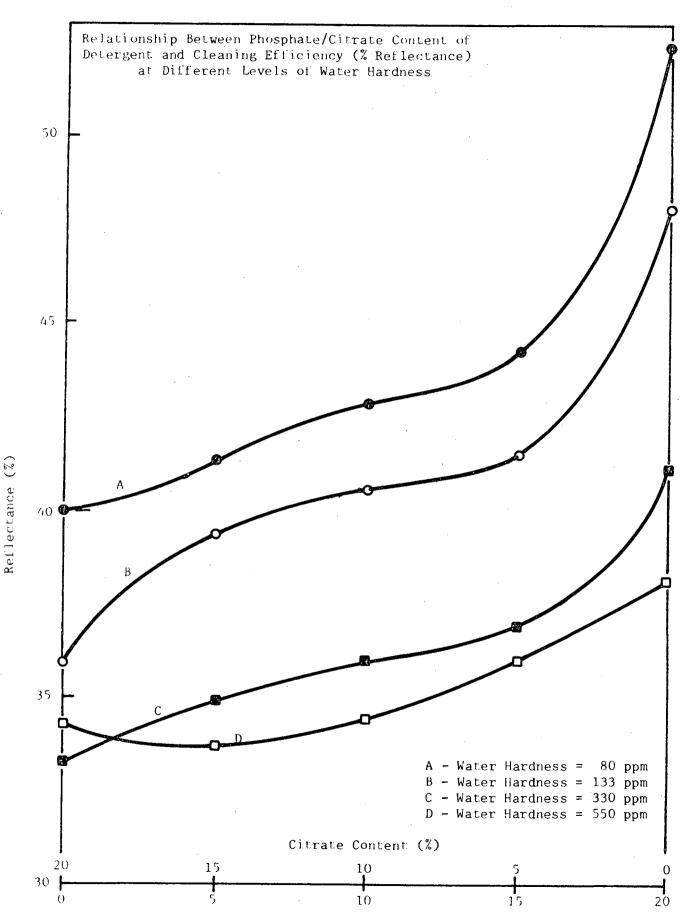
Reflectance Values for Orlon STC (Original Reflectance = 41.9%)

Figure 1c (1) COTTON CMS



- 51 -





Phosphate Content (% P_20_5)

- 53 -

COTTON CMS

Figure lc (III)

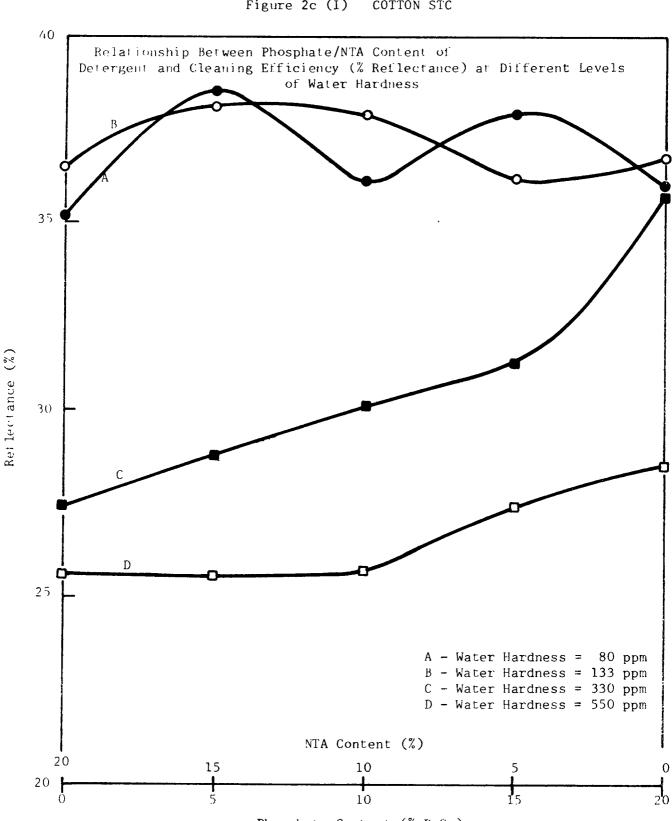
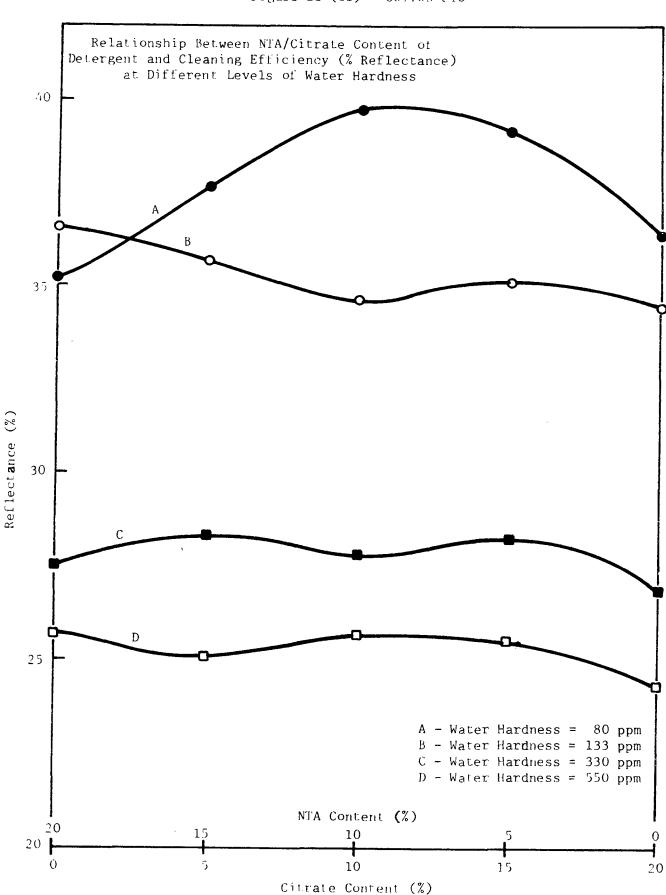
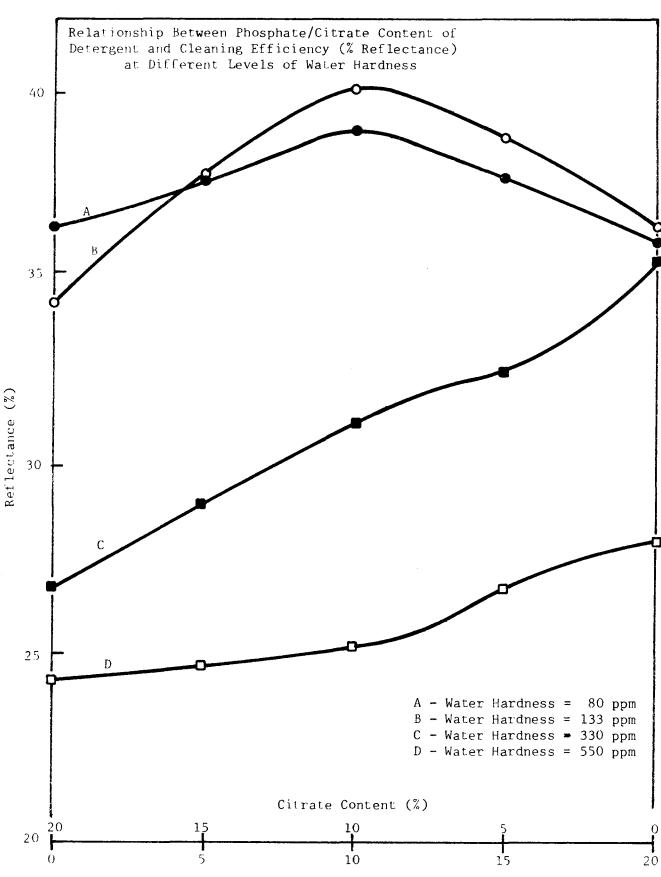


Figure 2c (I) COTTON STC

Phosphate Content (% P_20_5)



- 55 -Figure 2c (11) COTTON STC



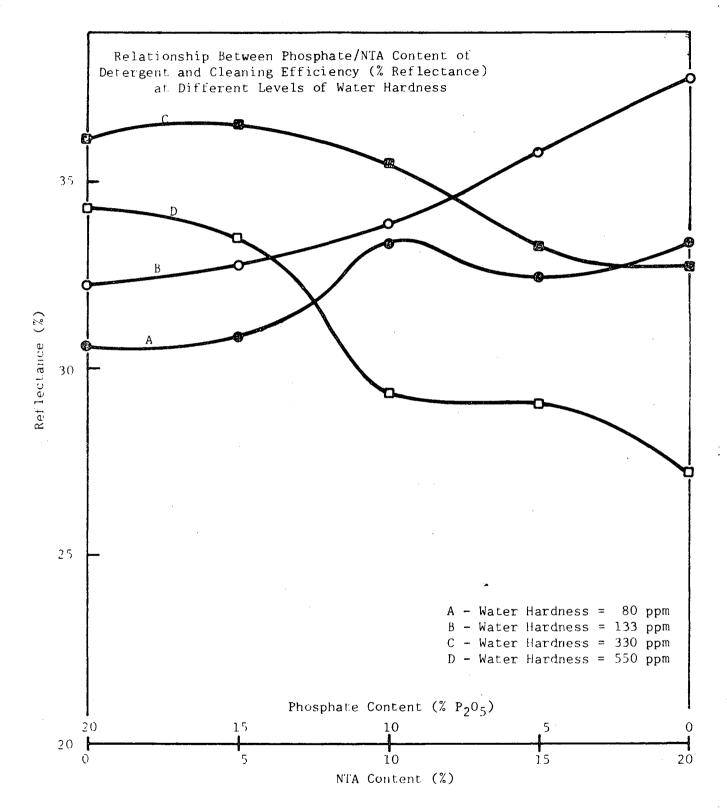
Phosphate Content (% P_2O_5)

- 56 -

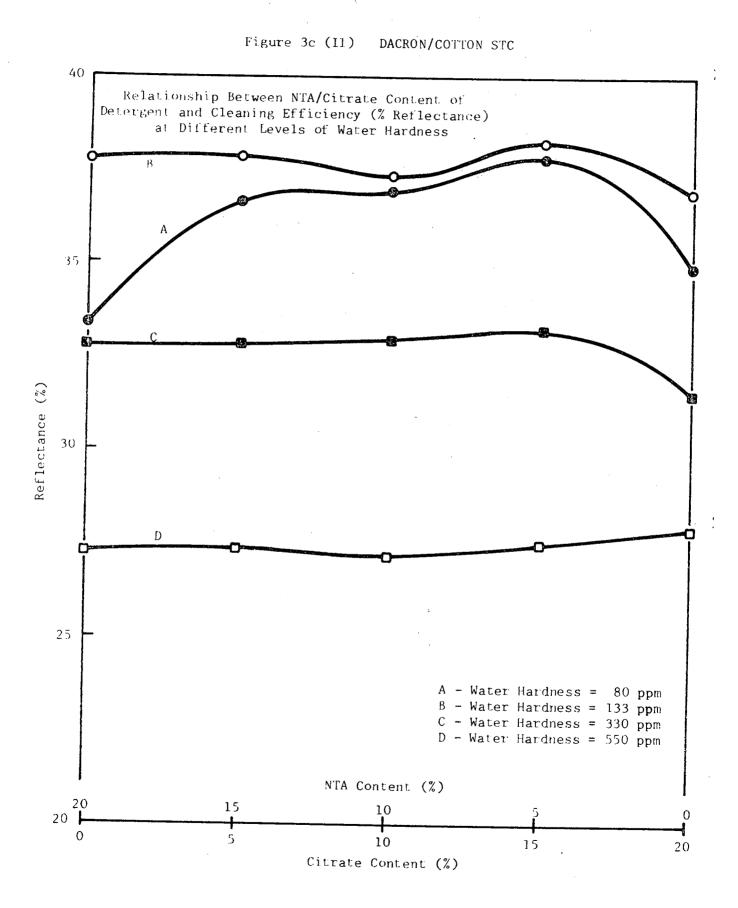
COTTON STC

Figure 2c (III)

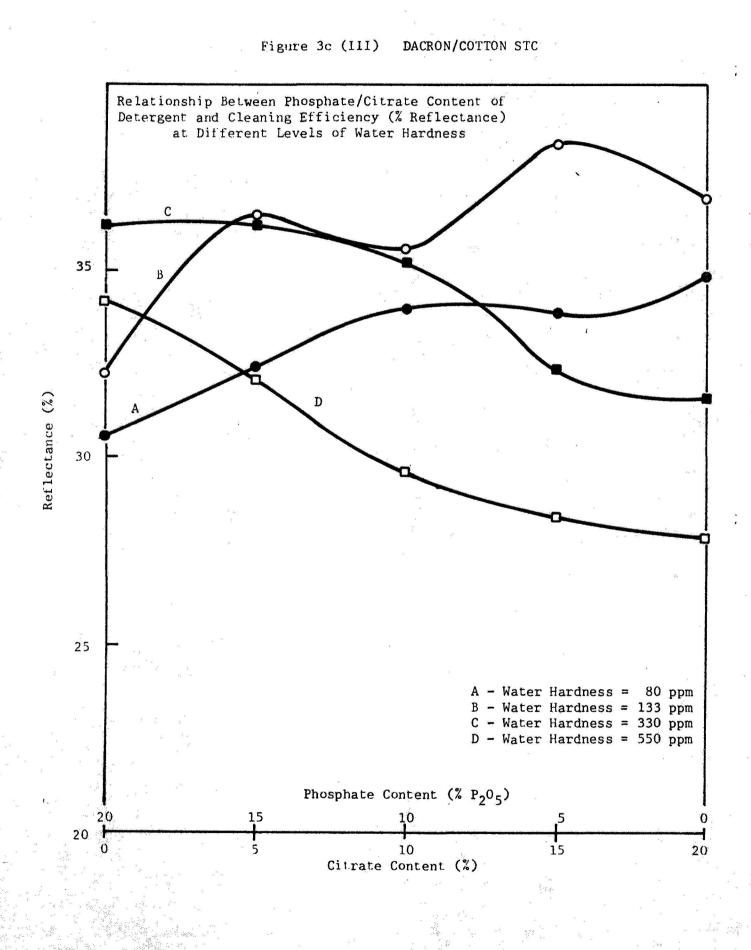


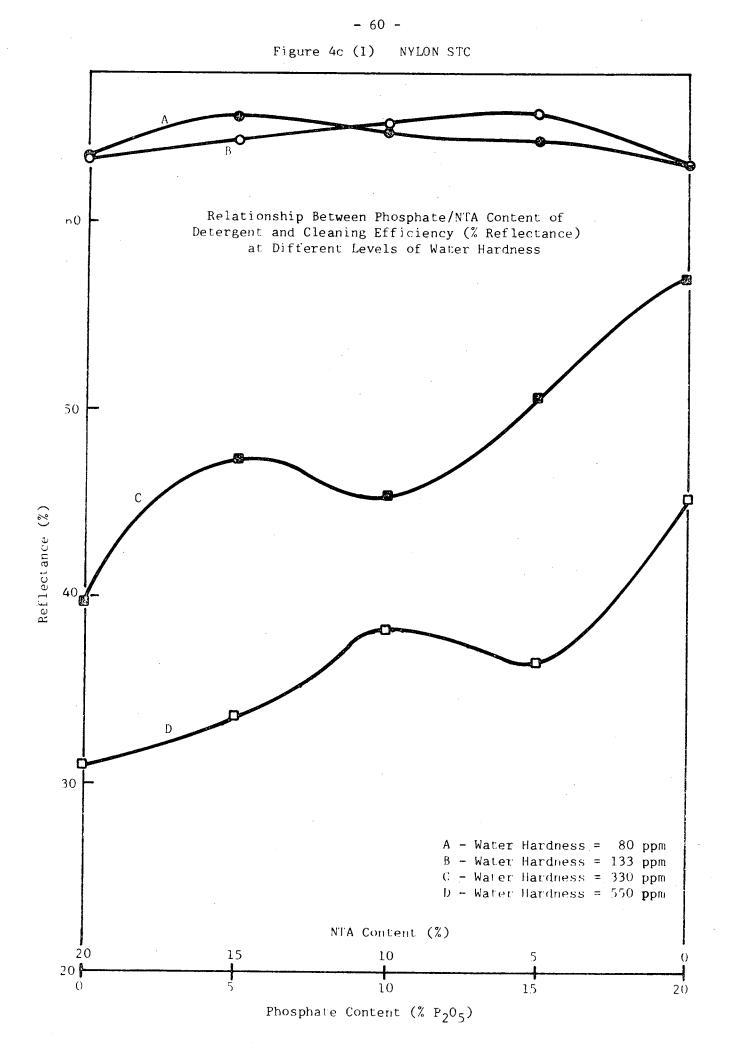


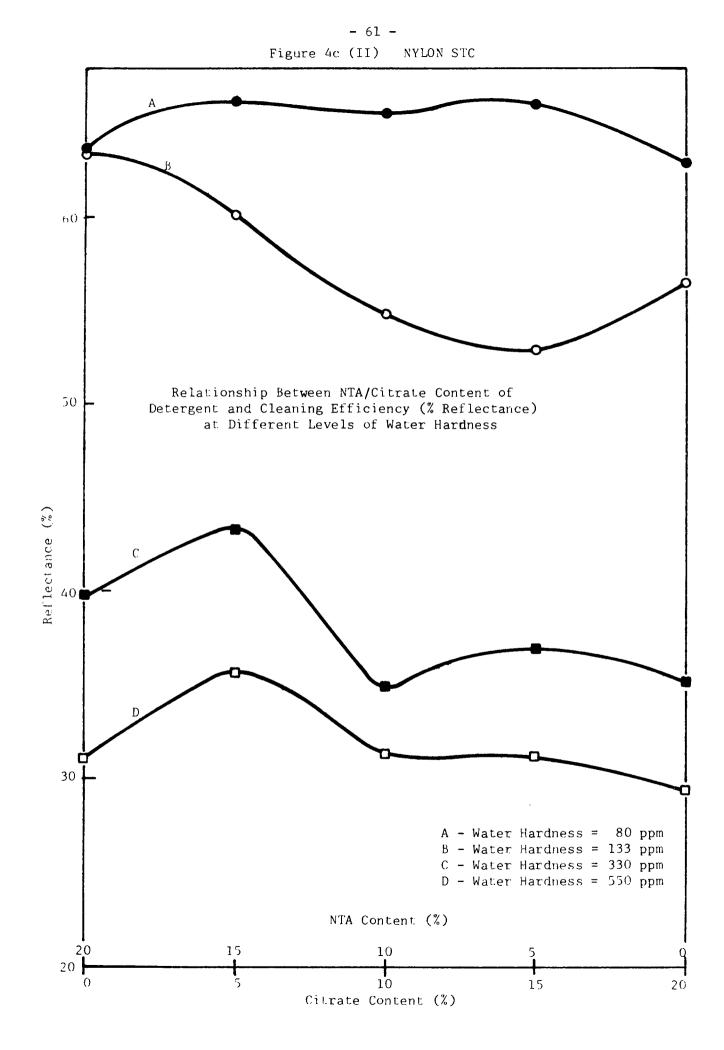
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- 58 -







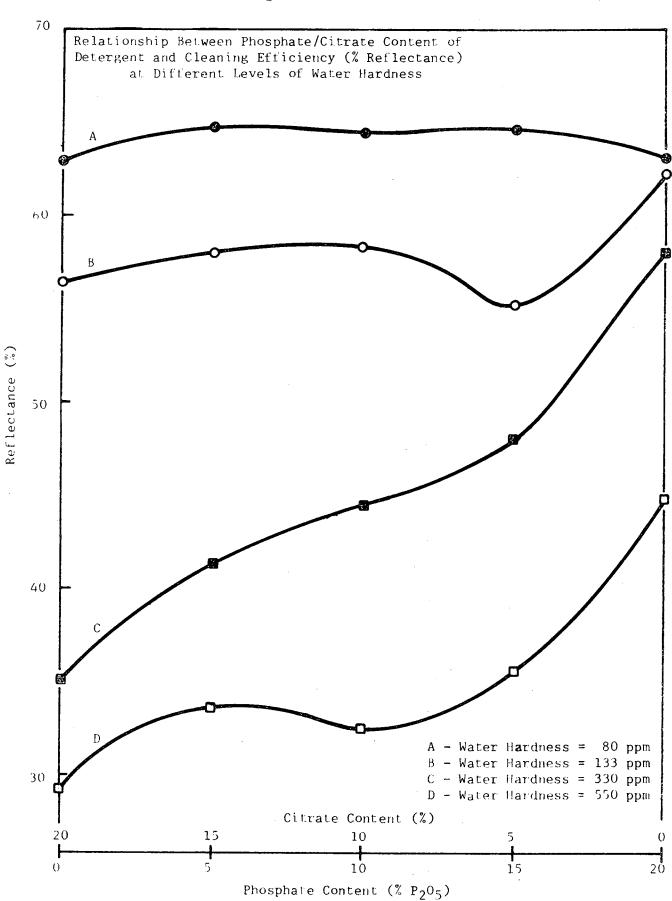
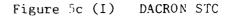
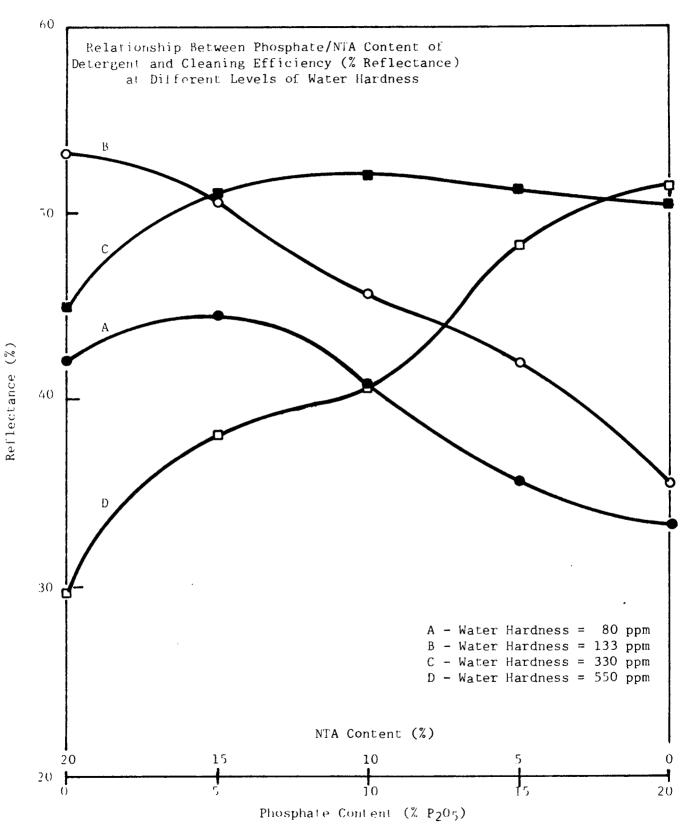


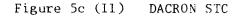
Figure 4c (III) NYLON STC

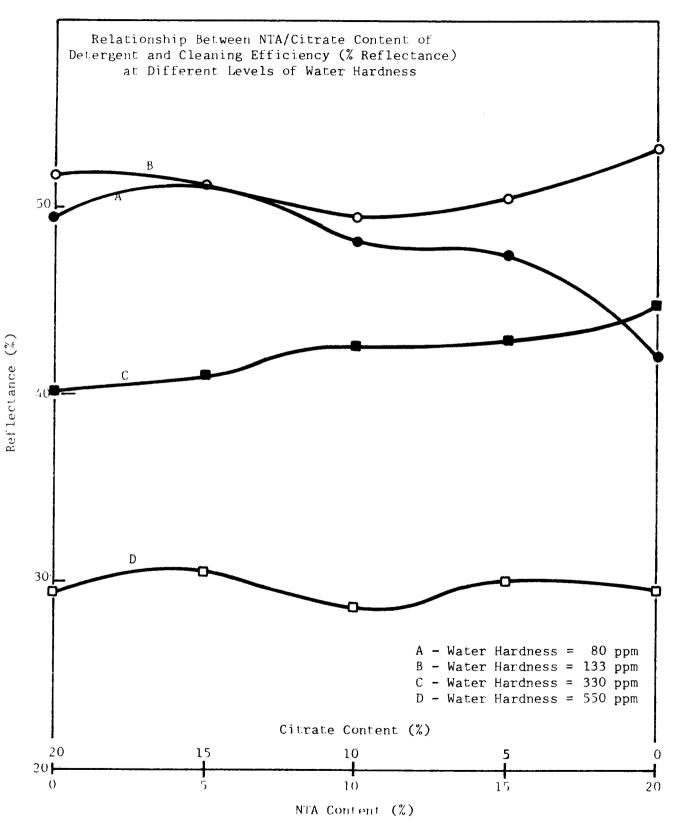
- 62 -

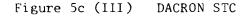


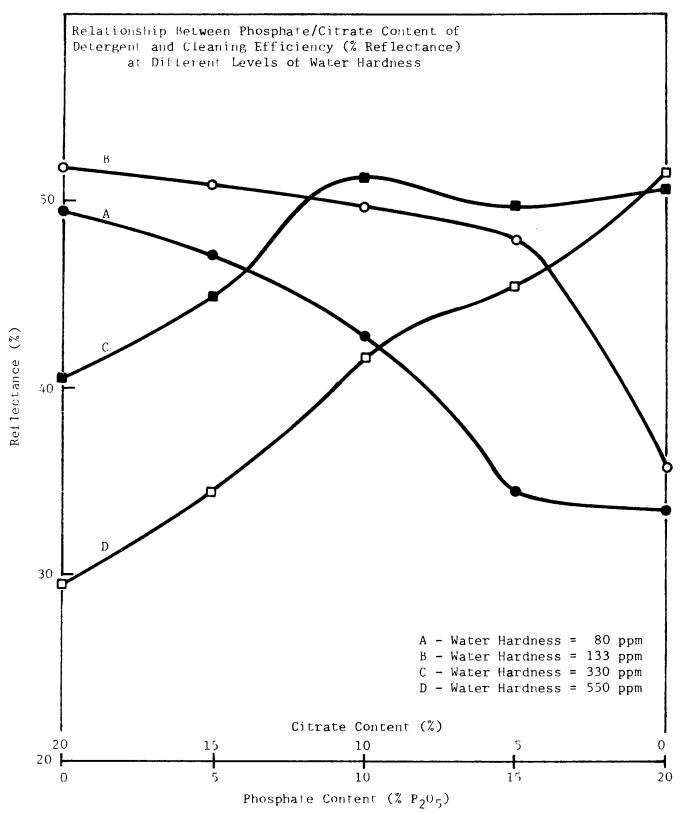


- 63 -

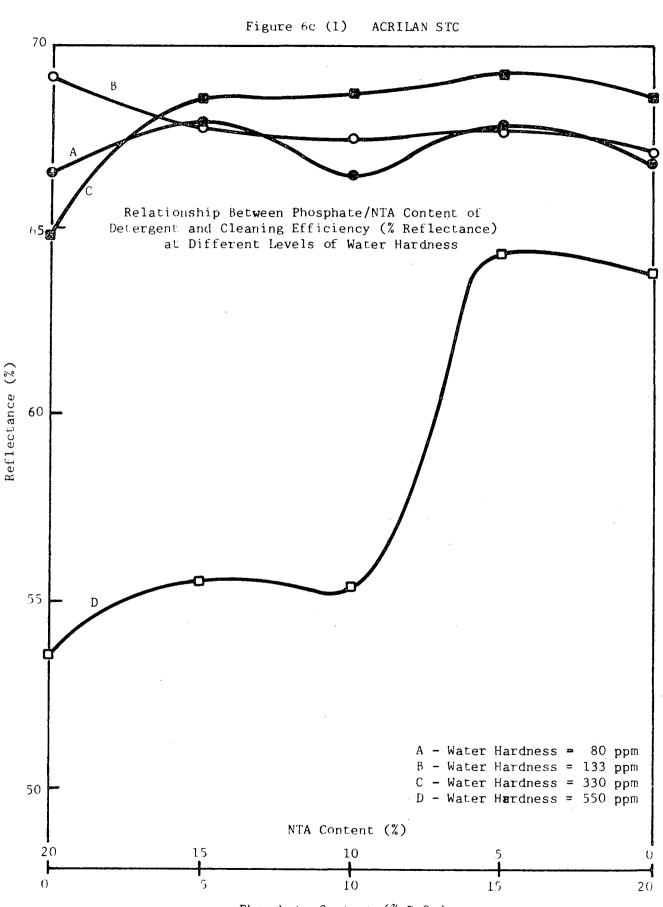






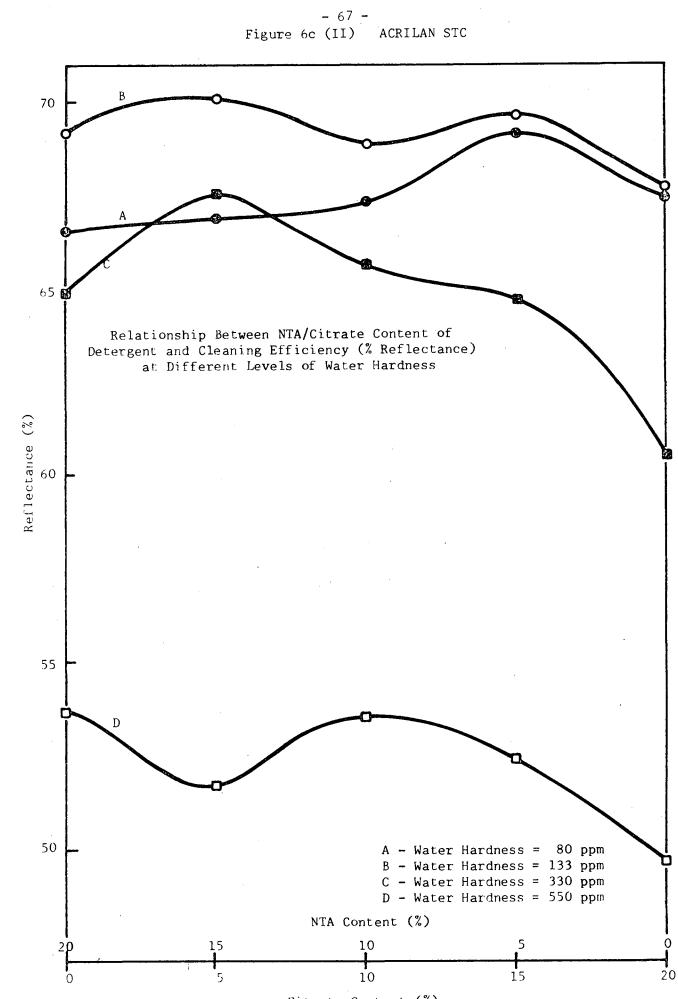


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Phosphate Content ($\% P_2 O_5$)

- 66 -



Citrate Content (%)

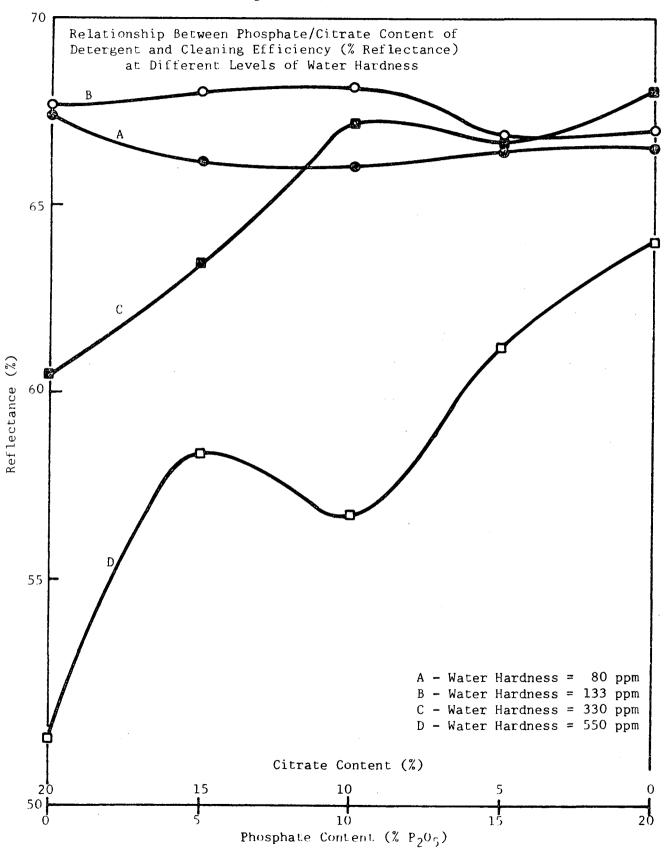


Figure 6c (III) ACRILAN STC

EVALUATION OF DETERGENT FORMULATIONS

Interim Report for Period April 29 - May 19, 1972

1.0 Summary

This part of the overall research program is concerned with the effect of variation in tricomponent blends of NTA, phosphate and citrate on detergency efficiency. Several detergents, based on CGSB specifications, were prepared containing between 5 and 15% of each component in the blend. Their cleaning ability was compared by laundering swatches of the soiled fabrics described in our interim report of February 17, 1972. The water supplies used, the specimens and replications, and the assessment of soil removal were also described in that report.

The seven formulations investigated in this phase of the program are described below.

		Detergent Identification					
	W	x	Y	Z	α	β	8
Cedepon S-85	25%	25%	25%	25%	25%	25%	25%
CMC D435	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Britesil C-20	10	10	10	10	10	10	10
Sodium Sulphate	38.3	26.5	26.5	30.2	30.2	22.7	22.7
NTA	7	5	15	15	10	10	5
STPP (asP ₂ 0 ₅)	7	10	10	5	5	15	15
Sodium Citrate	7	15	5	10	15	5	10

2.0 Results

The mean white light reflectance values after laundering the soiled fabrics with detergents of different NTA, phosphate and citrate content at different levels of water hardness are shown in Tables Id - VId.

	Reflectance V	alues for C	otton CMS (Ori	iginal Refl	ectance =	34.8%)	
% NTA	% Phosphate (as P ₂ 0 ₅)	% <u>Citrate</u>	Detergent	80	Water Har <u>133</u>	dness (ppm <u>330</u>	1) 550
7	7	7	W	42.2	38.8	35.2	33.3
5	10	. 15	х	44.8	40.9	36.8	34.8
15	10	5	Y	46.7	42.4	35.8	34.0
15	5	10	Z	44.3	41.0	35.0	34.5
10	5	15	α	44.2	41.1	35.2	34.2
10	15	5	β	45.5	44.9	37.9	34.2
5	15	10) /	44.7	43.4	37.0	36.0

TABLE Id

TABLE IId

Reflectance Values for Cotton STC (Original Reflectance = 18.1%)

%	% Phosphate	%		1	Water Har	dness (ppm	1)
NTA	$\frac{(\text{as P}_2 0_5)}{(\text{as P}_2 0_5)}$	Citrate	Detergent	80	<u>133</u>	330	<u>550</u>
7	7	7	W	40.6	37.9	28.3	25.1
5	10	15	Х	41.8	38.9	33.2	27.9
15	10	5	Y	42.5	38.4	33.7	26.4
15	5	10	Z	39 .6	41.5	30.6	26.5
10	5	15	α	39.9	39.3	30.3	25.7
10	15	5	β	41.8	41.0	37.0	29.0
5	15	10	3	41.0	41.8	37.2	29.2

Ref	lectance Valu	es for Dacr	on/Cotton STC	(Original	Reflecta	nce = 20.52	%)
%	% Phosphate	°/			Water Ha	rdness (pp	m)
NTA	$(as P_2 O_5)$	Citrate	Detergent	80	133	330	550
7	7	7	W	33.8	37.6	35.3	28.2
5	10	15	Х	32.9	35.6	38.3	31.5
15	10	5	Y	31.8	34.5	38.3	30,5
15	5	10	Z	33.2	36.1	36.0	29.0
10	5	15	α	33.6	35.9	36.1	29.6
10	15	5	β	32.8	32.5	37.4	34.6
5	15	10		33.5	33.2	37.1	33.3

Т	AB	LE	IVd	

Reflectance Values for Nylon STC (Original Reflectance = 24.2%) % Water Hardness (ppm) % % Phosphate 80 133 330 $(as P_0)$ Citrate Detergent NTA 7 5 15

7	7	7	W	63.8	63.5	40.4	29.7
5	10	15	х	63.7	63.7	51.2	36.4
15	10	5	Y	63.8	64.2	55.3	33.1
15	5	10	2	64.6	64.2	45.5	35.2
10	5	15	α	65.1	63.6	46.2	33.5
10	15	5	β	65.1	63.0	55.4	38.8
5	15	10		65.0	63.3	56.8	40.7

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TABLE Vd

Reflectance Values for Dacron STC (Original Reflectance = 25.0%)

%	% Phosphate	%		<u></u>	Water Hai	cdness (pp	: m)
NTA	$(as P_2 O_5)$	<u>Citrate</u>	Detergent	80	133	330	550
7	7	7	W	43.2	50.6	52.2	38.2
5	10	15	х	37.2	44.6	52.9	44.7
15	10	5	Y	32.9	39.4	54.4	42.7
15	5	10	Z	43.3	47.5	54.3	39.8
10	5	15	α	46.2	50.3	51.9	39.6
10	15	5	β	36.3	35.5	54.4	51.5
5	15	10		36.6	41.3	54.0	50.2

TABLE IIId

	Reflectance	Values for	Orlon STC (Ori	.ginal Ref	lectance =	41.9%)	
% <u>NTA</u>	% Phosphate (as P ₂ 0 ₅)	% Citrate	Detergent	<u></u>	Vater Hard	ness (ppm) 330) <u>550</u>
7	7	7	W	67.4	68.3	64.2	54.4
5	10	15	х	66.4	67.3	68.3	61.8
15	10	5	Y	67.6	67.4	68.6	59.3
15	5	10	Z	67.2	68.0	67.7	60.1
10	5	15	α	67.6	67.5	68.7	55.7
10	15	5	β	67.3	68.6	69.5	66.4
5	15	10	5	67.0	67.9	69.2	65.1

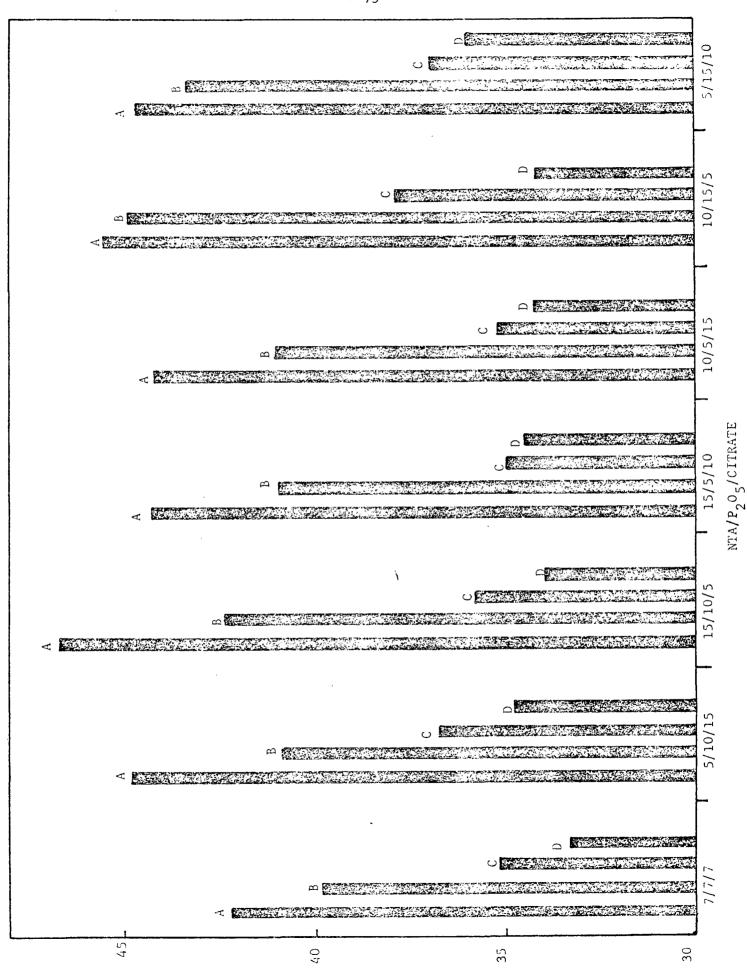
TABLE VId

The results of the above tables are recorded graphically in Figures ld to 6d.

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M. J. Williams Research Scientist Department of Textiles

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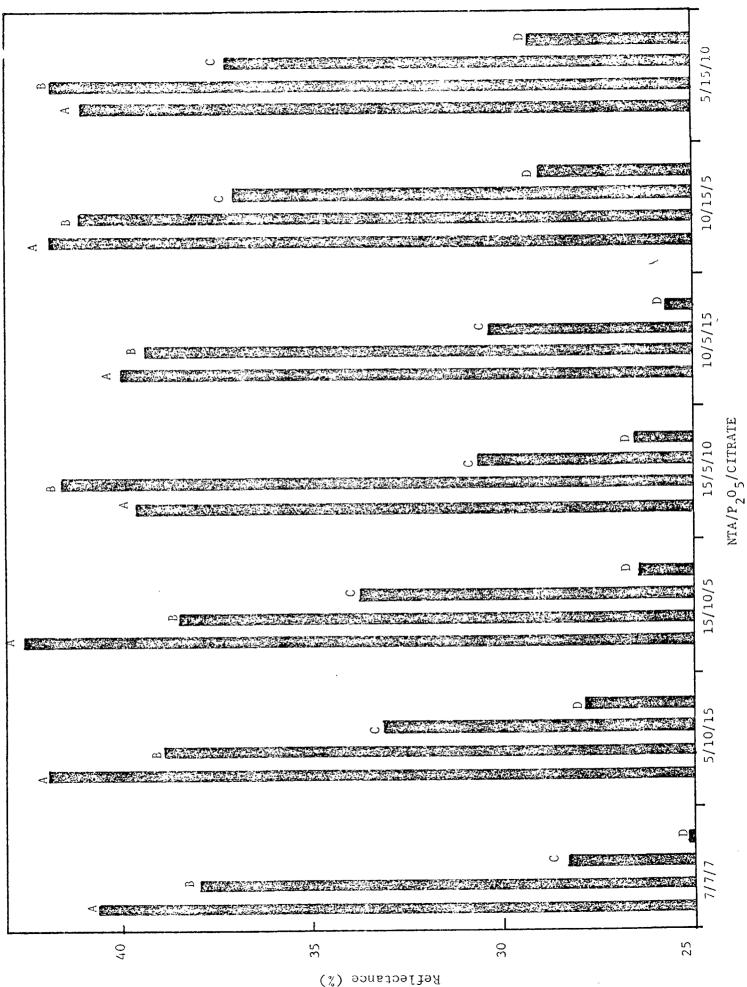


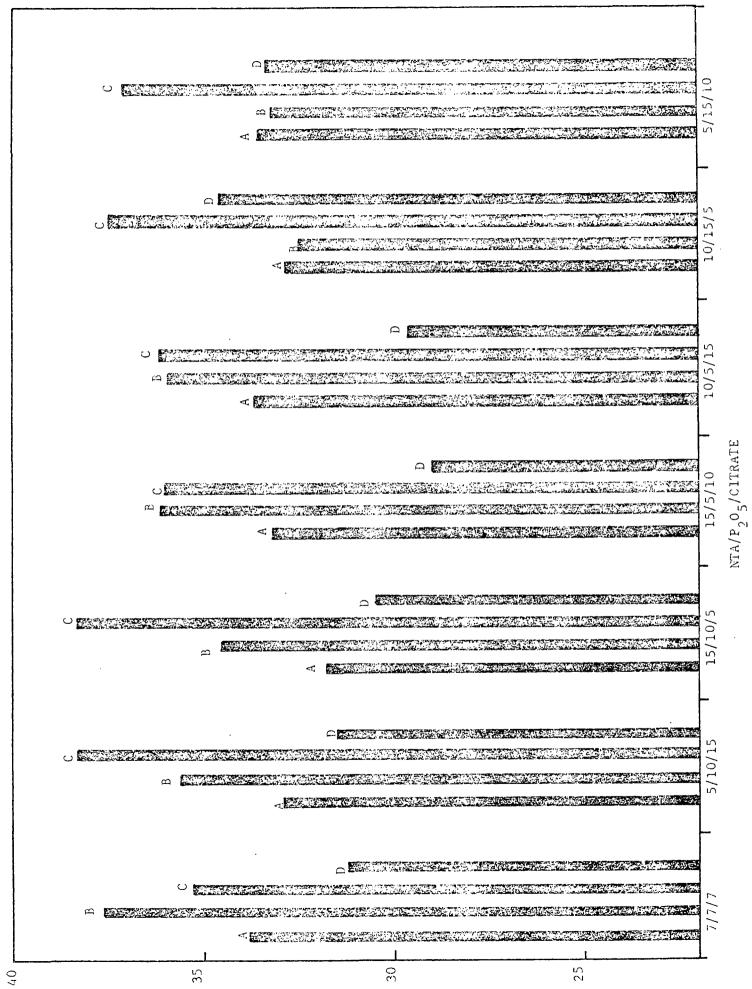
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FIGURE 3d ACRUNSTC

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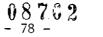
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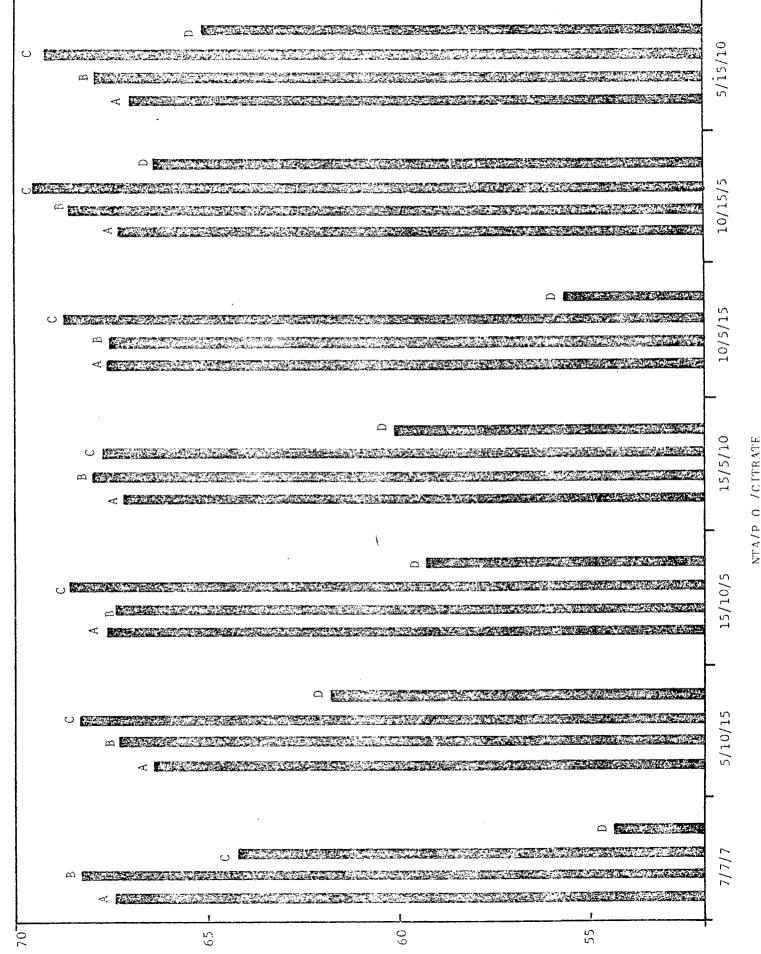
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Reflectance (%)