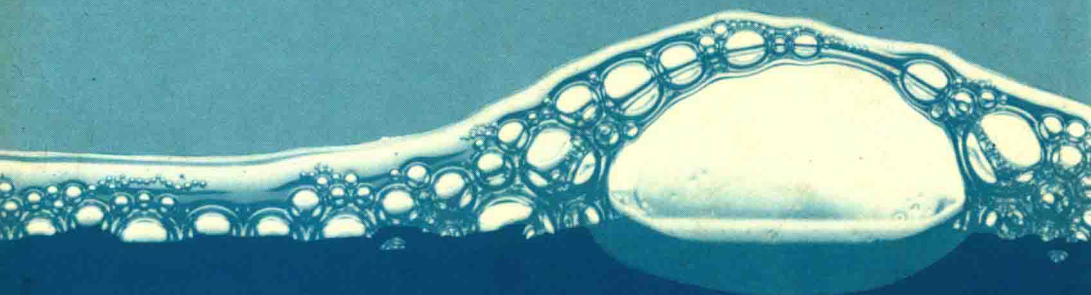


*A S Davidsohn & B Milwidsky*



# **SYNTHETIC DETERGENTS**

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*seventh edition*

# SYNTHETIC DETERGENTS

Seventh Edition

A. S. DAVIDSOHN DIPL. CHEM.

and

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# Synthetic Detergents

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# Preface to the Seventh Edition

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In presenting this seventh edition we have maintained the guidelines of the sixth edition, but have shifted the emphasis somewhat.

From reports reaching us it has become apparent that the book is being used, in addition to a reference book for the industry, as a training manual for workers new to detergents and as a textbook for the study of a typical chemical process industry.

With this in mind, we have described in somewhat greater detail basic synthetic processes to give the reader a working understanding of the raw or intermediate products he will encounter, including the new materials coming into use. Sight has not been lost of our original aim to cater for the practical man.

The world is also preoccupied with the diminishing reserves of fossil fuels. We have also indicated possible sources of organic intermediates not based on petrochemical sources.

Formulations have been updated to cover the latest trends in the industry, including the newer raw materials becoming available and the latest state of the art plant being manufactured.

The chapter on analytical procedures has been omitted as there are separate works on this subject and it cannot be covered, other than very sketchily, in a single chapter.

*A. S. Davidsohn  
B. Milwidsky  
January 1986*

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# Preface to the Sixth Edition

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This is a work by practical men for practical men. It is not intended to be simply a collection of formulae and recipes, nor does it set out to list all the materials which could conceivably be used as basic ingredients. Excellent books of that kind do exist but, in our view, they are often so comprehensive that it becomes difficult to distinguish between references to literature and practical work. We have purposely confined our text to those details which the practical manufacturing chemist will require and, we hope, will be able to use.

Our intention has been to cover all the operations that a manufacturer is likely to encounter in practice. All of the formulations have been critically selected so as to provide a useful starting-point; each manufacturing chemist can then use his own initiative to develop these formulations to suit his own local, or special, conditions. The reader may notice that we frequently quote from technical brochures issued by raw-material manufacturers. We make no apology for this, for the extensive research and development on which these publications are based often make them unique as sources of information for workers in this field.

During the past decade the detergent industry has seen a series of upheavals. It has seen a change from hard to soft detergents in the developed countries, and the large-scale introduction of enzymatic powders (and the subsequent problems). It has also had to face the attack on phosphates. In addition, the rate of growth of non-ionic detergents is increasing at the expense of anionic.

The alterations and many additions to this sixth edition reflect the new developments that have taken place as a consequence of these changes. However, the basic structure of the book remains the same, although, since this edition has been completely re-set, the opportunity has been taken to improve layout, to use metric units wherever practical, and to remove some inconsistencies that have crept in over the years.

Although this edition was prepared during a time of economic stagnation, the detergent industry has continued to develop from year to year. We have endeavoured to cover the technical aspects of this healthy trend and we hope that this book will continue to meet the needs of those in or concerned with the detergent industry throughout the world.

*A. S. Davidsohn  
B. Milwidsky  
November, 1977*

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\* The name Alfol is a registered trade mark of the Continental Oil Company, Oklahoma, USA.

† The name Epal is a registered trade mark of the Ethyl Corporation, Baton Rouge, LA, USA.

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# 1. Development of the Detergent Industry

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Although the start of the synthetic detergent industry is not shrouded in the veils of history as were the beginnings of the soap industry, it is nevertheless not easy to pinpoint exactly when the detergent industry, as such, came into being. The primary problem is to decide exactly what is being referred to as a synthetic detergent. The term itself leads to confusion. In the USA the words surfactant or syndet are being used, whilst in Europe the term 'tenside' (for tensio-active material) has come into fashion.

Many definitions of synthetic detergent have been proposed, all of which are very wide. *The Comité International de Dérivés Tensio Actifs* has after several years of deliberation agreed on the following definitions:

*Detergent*: Product the formulation of which is specially devised to promote the development of detergency. *Note*: A detergent is a formulation comprising essential constituents (surface active agents) and subsidiary constituents (builders, boosters, fillers and auxiliaries).

*Surface Active Agent*: Chemical compound which, when dissolved or dispersed in a liquid is preferentially absorbed at an interface, giving rise to a number of physico-chemical or chemical properties of practical interest. The molecule of the compound includes at least one group with an affinity for markedly polar surfaces, ensuring in most cases solubilization in water, and a group which has little affinity for water. *Note*: Compositions in general are usually mixtures of such compounds.

*Amphiphilic Product*: Product comprising in its molecule, at the same time one or more hydrophilic groups and one or more hydrophobic groups. *Note*: surface active agents are amphiphilic products.

We shall, however, in this book continue to use the term synthetic detergent for a material which cleans (or is used for cleaning), but in this definition we do not include soap.

Even so, this is still a wide definition, because, of course, it can refer to the active ingredient, or the solid, liquid, paste or powder compounded from this active matter. However, this should not lead to confusion, as the industry itself as yet makes no distinction in terminology between the basic material and the ready-for-use product.

The first synthetic detergents which fall into our definition of the term seem to have been developed by the Germans in the First World War period to allow fats to be utilized for other purposes. These detergents were of the short-chain alkyl naphthalene sulphonate type, made by

coupling propyl or butyl alcohols with naphthalene and subsequent sulphonation, and appeared under the general name of Nekal. These products proved to be only fair to moderately good detergents, but good wetting agents and are still being produced in large quantities for use as textile auxiliaries.

In the late 1920s and early 1930s long-chain alcohols were sulphonated and sold as the neutralized sodium salts without any further additions except for sodium sulphate as an extender.

In the early 1930s long-chain alkyl aryl sulphonates with benzene as the aromatic nucleus, and the alkyl portion made from a kerosene fraction, appeared on the market in the USA. Again, these were available as the sodium salts extended with sodium sulphate. Both the alcohol sulphates and the alkyl aryl sulphonates were sold as such as cleaning materials, but did not make any appreciable impression on the total market. At the end of the Second World War alkyl aryl sulphonates had almost completely swamped the sales of alcohol sulphates for the limited uses to which they were applied as general cleaning materials, but the alcohol sulphates were making big inroads into the shampoo field. An exception was Teepol, a secondary alcohol sulphate which remained popular for some years.

In common, however, with other chemical developments during this century, progress was not in one direction only. The limiting factor is always the availability of raw materials in a particular country. Concurrently with the above developments, there were developed, both in Germany and the USA, the Igepon type of compounds of which Igepon-T, the sodium salt of oleyl tauride is an example, and in Germany the Mersolates, which are alkane sulphates.

Each of these basic materials has its advantages and disadvantages, but in considering the feasibility of production the following factors must be taken into account:

- availability of raw materials;
- ease of manufacture;
- cost of raw materials;
- cost of manufacture;
- suitability of finished product.

We have purposely placed suitability last, as it is only too true that not always is the best material made available.

As a result of its ease of manufacture and versatility, the alkyl benzene sulphonate very quickly gained a foothold in the market, and after the last war the existing keryl benzene was very quickly replaced by an alkyl benzene made from propylene tetramer coupled to benzene (PT benzene).

This PT benzene very quickly displaced all other basic detergents and for the period 1950-65 considerably more than half the detergents used throughout the world were based on this.

To give an idea of the enormous rise in synthetic detergent production, Table 1.1 compiled from figures submitted by the American Soap and

# DEVELOPMENT OF THE DETERGENT INDUSTRY

Detergent Association and the German firm of Henkel KGaA shows both soap and detergent sales in the USA for various years.

TABLE 1.1

US Soap and Detergent Sales		
Year	Soap sales 1000 tons	Synthetic sales 1000 tons
1940	1410	4.5
1950	1340	655
1960	583	1645
1972	587	4448
1982	545	5090

These figures reveal that immediately after the Second World War synthetics started making inroads into the production of soap, which now seems to have settled down to a constant whereas synthetics have increased enormously.

By 1959 although the US per capita consumption had somewhat levelled out, total production was still rising as shown in Table 1.2 which has been compiled from the Henkel figures.

TABLE 1.2

Comparative Production Figures for Synthetic Detergents in the USA				
	1958 1000 tons	1963 1000 tons	1972 1000 tons	1982 1000 tons
Domestic detergents (solid)	1200	1425	2672	2763
Domestic detergents (liquid)	354	640	1773	2327

The broad picture that appears from Table 1.2 is that while solid detergents (among which of course powders are included) are making great strides forward, the liquid detergents are increasing at a much faster rate.

Looking at world production figures for the last few years (Table 1.3), it appears that although soap is growing, synthetics are increasing tremendously. This is because Third World countries are increasing the use of both synthetics and soaps. A random look at per capita consumption shows that in Western Europe in the years 1960 to 1980 per capita consumption rose from 9.7 to 18.9 kg per annum, in North America from 12.8 to 30.1 and in Asia from 1.0 to 2.1 (2.5 for China).

It can be seen that despite a tremendous growth in the past decades, the production of synthetics is still continuing to rise.

No review of the history of the detergent industry can omit mention of

## DEVELOPMENT OF THE DETERGENT INDUSTRY

TABLE 1.3

World Production of Cleaning Materials				
	1968 1000 tons	1970 1000 tons	1980 1000 tons	1982 1000 tons
Soaps	6493	6059	8218	8705
Synthetics	7369	8352	16,453	17,889
Other*	1015	1310	2380	2562

\* Includes fabric softeners.

the builders, without which the remarkable success outlined above could never have been achieved.

After the Second World War, when detergents started appearing in appreciable quantities on the retail market, it was noted that white cotton goods were not being washed as white as they should be. This was explained by the fact that although the active material was able to lift the dirt from the cloth it could not keep it in suspension. Hence small spots of dirt were being redeposited uniformly over the whole surface area of the cloth while in the wash-tub or machine, thus giving the cloth a grey appearance.

The sodium salt of carboxymethylcellulose (CMC) had been known to industry for many years and, in fact, a French patent had been applied for in 1936,<sup>1</sup> using CMC as an additive to washing materials. However, this patent was not developed extensively until the Second World War, when CMC was used in Germany on a moderately large scale, initially as an extender for soap which was in short supply, and then as an additive to the synthetic detergents being produced as a wartime substitute for soaps. When intelligence reports on the German industry were published, the use of CMC as an additive to synthetic detergent powders was noted and investigated and it was found that this addition eliminated the redeposition problem.

Despite the considerable advances made in the production of the active detergent matter, by the end of the Second World War progress in the use of detergents for heavy-duty (cotton) washing was still relatively slow, although they had already displaced soaps to a considerable extent in the field of fine laundering and dish-washing. To improve the heavy-duty washing properties, manufacturers turned for analogies to the soap industry. Soap for cotton washing had for many years been 'built' with alkaline materials such as carbonates, silicates, borax, and orthophosphates. All of these singly and in combination were tried with moderate success. Condensed phosphates had started appearing on the market in increasing quantities and from 1947 onwards heavy-duty detergent formulations were introduced, initially with tetra sodium pyrophosphate and then with sodium tripolyphosphate with startling success.

With the advent of CMC and tripolyphosphate builders the detergent industry established itself and has never looked back.

Production in the use of sodium tripolyphosphate rose from 100,000 tons in 1947 to over 100,000,000 tons in 1970. In that year environmental problems arose and production fell somewhat, concurrently a search for replacements was undertaken. To date no 'plug-in' replacement has been found.

Propylene tetramer benzene sulphonate held almost undisputed sway as the major ingredient used in washing operations till the early 1960s.

As early as 1952 in the UK, somewhat later in other countries, it was noted that sewage treatment problems were arising. The amount of foam on rivers was increasing and where water was being drawn from wells located close to household discharge points, the water tended to foam when coming out of the tap. This was attributed to the fact that propylene-based alkyl benzene sulphonates are not completely degraded by the bacteria naturally present in effluents, and was further narrowed down to the fact that it is the branched-chain formation of the alkyl benzene which hinders the attack by the bacteria. However, fatty acid sulphates were found to degrade very easily, and since all naturally occurring fatty acids from which fatty alcohols are produced are of the straight-chain variety (as also are the Ziegler alcohols which started appearing in commercial quantities at about this time), it seemed possible that a straight-chain alkyl benzene might be degradable.

Methods of test were developed and it was, in fact, proved that linear alkyl benzene is biodegradable. Germany introduced legislation prohibiting the discharge of non-biologically degradable material into sewer systems. In the USA detergent manufacturers agreed voluntarily to switch over from PT benzene to linear alkyl benzene by June 1965. In the United Kingdom a similar type of 'gentleman's agreement' was entered into.

The change to linear alkyl benzene (which can be considered as a return to a purified form of the keryl benzene in use twenty years previously) gave some rather surprising results. It was found that the detergency in a heavy-duty formulation using linear alkyl benzene sulphonate was approximately 10 per cent better than when using PT benzene sulphonate, solutions of the neutralized sulphonic acid had a lower cloud point, and pastes and slurries had a lower viscosity. The first two results were obviously advantageous and a lower viscosity in slurries had an advantage when the product was spray-dried to a powder, but when the LAS was sold as a liquid or paste detergent, this lower viscosity had to be overcome as sales appeal was lost. The manufacture of powders based on LAS posed some problems, however. Powders became sticky and lost their free-flowing characteristics, whether made by spray-drying or one of the other methods. Mausner and Rainer<sup>2</sup> have indicated that the actual isomer distribution of the linear alkylate has an effect on the stickiness of the powder, with the 2-phenyl isomer giving the greatest tendency to stickiness and the 5- or 6-phenyl isomer the least. Additives to overcome this tendency have therefore been developed (p 78).

The switch to linear alkyl benzene is not, however, complete. In many parts of the world where the problem of sewage treatment is not serious,

the PT benzene is still being used in ever-growing quantities. Also the Ziegler alcohols are now competitively priced with the linear alkyl benzenes, and alkane sulphonates are reappearing.

Despite the 'hardness' of propylene tetramer benzene, it is still used to a certain extent. For agricultural emulsifiers, due to administrative difficulties of reformulation and consequent registration, the PT version is still used, but in countries where sewage problems have not yet arisen, there is a switch to the linear product for economic reasons, in that with the fall in production of the one and the increase of the other, the linear product has become cheaper and problems of reformulation are not as complicated for detergent products as they are for emulsifiers.

Modern awareness of the danger of depletion of petroleum reserves has started a tendency to reconsider the use of materials based on oils and fats as the source of the hydrophobe, that is a reversion to the original type of detergents, as the sources are self-replenishing.

Having successfully coped with the problem of biodegradation the industry faced a new attack. It appeared that in certain lakes and ponds algae started reproducing at an unprecedented rate. This was blamed on the extensive use of phosphates which are a food for these organisms, and again the detergent industry became the whipping boy, because tremendous amounts of sodium tripolyphosphate are used and then discharged down the sewer. (The term eutrophication, meaning nutrition by chemical means, has been applied to this phenomenon.) It is not clear whether the blame should be taken solely by the detergent industry, as concurrently with the increase in the use of detergent phosphates there was an increase in the use of phosphate fertilizers, which also find their way into natural water systems. However, with the big international preoccupation with ecology the detergent industry is searching for an efficient substitute for sodium tripolyphosphate. To date a complete replacement has not been found but in the Scandinavian countries particularly, formulations of household powders are beginning to appear with appreciable portions of the phosphate replaced by NTA (nitrilo triacetic acid) which is a better sequestering agent than tripolyphosphate but has none of the other properties exhibited by the phosphate. We are inclined to fear that in time the extended use of NTA might bring new problems of this sort, as it contains nitrogen which is again a good fertilizer and nutrient for algae. We quote from an editorial comment in *Chemical Week* on this subject:<sup>2</sup>

'We caution the legislations and administrations against shooting from the hip. They may blast phosphate out of detergents, but they may not be very proud of their victory when the facts are finally in.'

The search is still going on for a phosphate substitute. NTA on its own will only partially replace phosphates. A mixture of NTA and borax has been suggested as a complete replacement but here again the borax might produce more problems than the phosphate is alleged to produce. Some of the hydroxy-polycarboxylic acids (see p 92) containing only carbon, hydrogen and oxygen are also being considered.



In 1977 the German firm Henkel patented the use of synthetic zeolites as a partial replacement for phosphates. These zeolites, sodium aluminium silicate of a particular lattice structure have the facility, although insoluble in water, of absorbing by ion exchange heavy metal cations. The reaction with calcium is rapid, with magnesium somewhat slower but this increases with rising temperature.

No claims for total replacement of phosphate are made by the proponents of the zeolites, it is emphasized that they be used in admixture with STP<sup>4</sup> as they only soften water and perform none of the other functions attributed to the condensed phosphates.

In actual fact, their use has not reached the actual totals predicted but in Europe a fair proportion of detergent powders contain zeolites, in the USA a somewhat lesser amount; this is despite the fact that there are no ecological or toxicological objections to their use.

The other candidate for a (partial) phosphate replacement is nitrilo triacetic acid. NTA has had a chequered career. In 1970 it was used in large quantities in the 'no' or 'low' phosphate powders as a chelating agent (to complex the hardness ions) but towards the end of that year the US Surgeon-General issued a report stating that it has been found that NTA caused birth defects in rats when their drinking water was contaminated with NTA and cadmium or mercury salts. The report had specifically indicated that the quantities under the test conditions were several tens of times the possible concentration that could be encountered in practice and that there was no health hazard. Use of NTA was immediately phased out in the USA, but Canada and Europe did not react to this report. Further work indicated the possibility of urinary tract and cancer problems, again in high concentrations. In 1980 the Environmental Protection Agency (EPA) in the USA cleared the material for use in detergents, but at the time of going to press, in New York State doubt is again being cast on the safety of the material, but nowhere else is the product considered suspect.

With the pressure on detergent manufacturers to reduce phosphate concentrations, work is being continued on a substitute. NTA is a probable contender but no experience is available of large-scale discharge of this nitrogen compound into large masses of water, it might well be that if the volume comes close to that of the phosphates, adverse effects will be noted. Note that it took some ten years of continual use of the hard alkylate till biodegradation effects were noted, and well over twenty years for the phosphates. Zeolites might only be a partial answer, but being insoluble in water they have no effect on detergency. On the other hand sodium tripolyphosphate not only softens water, but also has a positive effect on detergency (p 50).

Two important reviews have appeared on this subject. In *European Chemical News* (19 November 1976), under the title 'No case for a European ban on detergent phosphates', it is pointed out that, in parts of the USA, although the phosphate ban is in effect, deterioration in washing quality is being experienced with no appreciable improvement in quality of