

provide only a rough indication of the nature of the main variables, and leave many doubtful points to be cleared up later. The experiment has shown that 32 tests are sufficient to distinguish the main effects of the four variables from the variance due to error, and has also shown that detergent concentration is a better variable than detergency quantity, and the pilot experiment has thus served its purposes. But it has done much more than this and, exceeding all expectations, has established all the main effects at significance levels all better than 0.01, and shown all interactions to be non-significant. The largest interaction variation has not even reached a significance of $p = 0.2$, and as no effects are left as "probably significant" the investigation of the importance of the variables is complete.

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WHITE OILS

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The early history of white oil manufacture is outlined, examples are given of its current wide use and reference made to the most recent spectroscopic technique for determining its stability.

THERE ARE many formulations of the cosmetic industry which contain white oil; that is, hydrocarbon oil of petroleum origin, and it is this raw material which is the subject of this talk. White oils are, however, comparatively new entries into an art or science which has, at times, claimed the attention of priests, and even politicians, and of which there are relics dating back to 5,000 years ago. The petroleum refining industry is by contrast in its infancy.

Research shows that there are very few aids to beauty to-day which have not had their counterpart in earlier centuries, but apparently it was not until the early 1900's that cosmetic compounding became really scientific. Strangely, or perhaps one of the reasons for the development, was the introduction of white mineral oils about the same time. To-day, of course, the cosmetic industry of this country holds a very important part in the national economy. It engages a large number of people, directly and indirectly, in its manufacture and distribution, and it has a rapidly increasing output both on the home and on export markets.

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The introduction of white oils came just before the turn of the century. Liquid paraffin was first introduced in the British Pharmacopoeia in the 1898 edition. About 1887, a Russian chemist, J. Markownikoff, devised a process for the manufacture of white oils from Russian crude or distillate, and eight years later an oil refinery, specialising in white oils, was started at Riga, at that time in Russian territory. The refinery was under the name of Oelrich & Co., and it apparently kept going in spite of wars intervening until about the 1930's. It is solely because of this Russian origin that white oils came to be known as Russian oils ; in fact, even to-day the name still sticks in one or two isolated cases.

The manufacture of white oils quickly spread westward to such places as Germany, Belgium and France, and up to the outbreak of the first world war there were probably half a dozen such refineries processing Russian distillates shipped from the Black Sea into white oils. America, up to a few years before 1914, imported all its white oil requirements from these refineries and, of course, so did this country. The 1914-1918 war put a stop to all exports from Europe and so the United States went ahead with its own production, until to-day its production capacity takes good care of all domestic requirements and even leaves room for exports.

It should be mentioned here that the demand for the so-called "Russian" oils from America did not die out until 1939, although up to that time the demand was getting smaller and smaller, year by year.

In England, a small refinery was built at Snodland in Kent during the 1914-1918 war, and that ran for a few years before closing down some time in the late 1920's. There was also a very small plant introduced in Tottenham or Edmonton, under the guidance of a specialist, believed to be of Russian birth, who was certainly responsible for increasing the popularity of the use of white oils. However, that plant closed down after a few years and the United Kingdom had to rely on imports from a number of countries, Germany, France, Belgium, Holland and America being the main suppliers. In 1938 a refinery was started in the North of England to make such oils, and thus to assist in the national economy and strategy at a particularly opportune time.

To-day, in 1955, the picture has completely changed. Other refiners have started here in this country, and there is now an adequate capacity to satisfy not only the demands of the home market, but to meet a large and ever-growing export market.

The advantages of home production of white oils to the cosmetic and to other industries are obvious. White oil production is a highly skilled and scientific job. Quality control and consistent supply are of vital importance in all industries, and not the least of the advantages is the fact that the white oil producer and the user can pool their research and development work to produce a tailor-made product for the particular application. The impor-

tance of this co-operation cannot be over-emphasised; experience has shown its value in many instances throughout the numerous industries using these highly refined oils.

The basic or physical properties of white oils were at one time determined by the type of crude oil employed. For example, a naphthenic crude oil yielded a white oil of high gravity and viscosity, whilst a paraffinic crude gave white oil of lower gravity and viscosity. Although that was the case in years gone by, when Markownikoff started his researches, to-day the science of modern petroleum refining is such that the type of crude does not play so important a part in the finished product.

In the same way, the constitutional analysis of petroleum products has made enormous progress in the last few years and is continuing to progress, like all scientific methods.

It is not appropriate in this short review to go into the details of the methods of production of white oils; the technique varies somewhat according to the type of crude selected and the final product required. Broadly speaking, the refining methods involve the use of sulphuric acid or oleum to remove all the unsaturated, aromatic and hydro-aromatic hydrocarbons. In other words, the unsaturated hydrocarbons are regarded as impurities which must absolutely be removed from the oil. This is a very expensive process, not only because of the amount and value of the chemicals used, but also because of the losses of oil during refining. The chemically treated and neutralised oil is then washed and filtered and "polished," and at all stages a very careful control must be exercised to ensure a correctly finished product.

Even when the finished product is obtained; that is, when the plant process engineer has received approval from the control laboratory, the oil is most carefully handled to preserve its particular characteristics right up to the user. That can amount to, say, a 16-oz. bottle of liquid paraffin BP that may or may not have been displayed in a chemist's shop for many weeks before it is sold to the final consumer. The care and attention paid to medicinal liquid paraffin applies equally to all grades of white oils and their compounds.

Before enlarging on this very brief indication of the technique and care required to produce satisfactory white oils, it may be interesting to examine some of the uses to which white oils are put in order to have a better measure of the need of control at all stages to ensure the particular characteristics required. The uses of white oils are so extensive that no single person can be aware of them all, and therefore anything said on this aspect should not, by any means, be accepted as comprehensive. Further, it will be seen that the uses or at least quite a large percentage of them call for special skill and knowledge of quality and particular applications.

Among the more conventional uses are those of the cosmetic industry and of the pharmaceutical industry. In the latter, the oils are used as internal

lubricants, for carriers for drugs, for skin applications either directly or in the form of ointments. Nasal drops and nasal sprays call for oils of light viscosity, and so does the manufacture of anti-biotics such as penicillin. Here, of course, the oil is used as a de-frothing agent. The pharmaceutical industry lays down its quality standards in the British Pharmacopœia, but in addition some individual firms also stipulate special gravities, viscosities, cold and acid tests, etc.

These are among the more conventional uses but there are many others covering a wide range of industries such as foodstuffs, insecticides, veterinary medicines, cattle sprays, electrical equipment (e.g., X-ray instruments and television lenses), refrigerators, textile machinery and yarn processing. A brief description of some of these will illustrate the variety and skill involved in the applications.

In food manufacture, every piece of machinery handling food is an example of the use of white oils. Automatic baking ovens are usually lubricated with white oils and so are dough dividers, sweet pans for confectionery receive a thin coating of white oil to prevent the sweets sticking to the pans, moulds for bottle caps, both the metallic and plastic caps, are lubricated by white oils.

Egg preserving by dipping in white oils is in regular use in some overseas countries such as Denmark, but here, although it has received the approval of the Ministry of Food, only a small quantity of eggs is preserved in this way.

Impregnation of apple wrappers, lubricating machinery for macaroni and spaghetti manufacture, the polishing of rice, the treatment of dried fruit to prevent sugar-crystallisation, the maturing of wine are some of the other uses coming under the category of food manufacture.

In the field of agriculture and horticulture, where it is essential not to injure delicate plant life, special types of insecticidal sprays also use highly refined white oils.

In cattle and animal husbandry the oils are used as in the pharmaceutical industry, as carriers for drugs and medicaments, as well as direct internal lubricants. Fluke disease in sheep gives trouble in countries like Australia, where artificial irrigation is prevalent, and here white oils are used as a carrier for a drug to eject the actual fluke from the liver of the sheep. The hide of pigs, both dead and alive, is dressed with white oils; for show purposes for the dead meat and as a skin medicament for live pigs.

X-ray instrument filling calls for a highly refined white oil of low refractive index and specially dehydrated.

Because of the diversity of usage in the manufacture of textiles, it deserves some special mention. It is not only used for lubricating the yarns and cut staple man-made fibres to assist winding and weaving, but also as a direct aid to processing of cotton and these other fibres. It is probably safe to say

that 95 per cent of the continuous-filament fibre produced in this country is lubricated by a high grade white oil.

To give some idea of the importance of this application, statistics are available showing total production in this country is 37.3 million lb. per month of filament and cut staple fibre combined during 1954. That is, about 200,000 tons per year.

Last year, in July, it became compulsory for mule spinning mills to use white oils for the lubrication of the mule spindles. The Ministry of Labour published data showing a big reduction of dust in mill card rooms where the cotton is impregnated with white oil, and in fact such methods, amongst others, are officially recommended for the prevention of byssinosis in the cotton industry. Mule spindle lubrication by white oil is urged to obviate the possibility of mule spinners' cancer. These mule spindles are running at 9,000 to 12,000 revs. per minute, and so it will be appreciated that white oils are capable of lubricating just as well as the more conventional types of mineral oils. Both byssinosis and mule spinners' cancer are notifiable and compensatable diseases.

Much more could be said on the uses of white oils, but it is time to return to the question of quality control. Practically all uses have their individual purity standards or specifications. Some are considered as very high, particularly those from the cosmetic and textile industries, so that a complex technique and much care are required to produce satisfactory white oils.

Firstly, the crude oil is checked on arrival at destination; during distillation hourly samples are drawn by the operator of the distillation plant and submitted for laboratory test and approval. At the next stage of refining, the raffinate is again hourly controlled until it arrives at that condition ready for processing into white oils. From that time to its finished state several days may have elapsed, and during the processing samples are drawn for checking by the control laboratory. At its final stage comes the critical examination by the men specialised in white oils and, no matter how urgent a delivery, these are the men on whom an organised refinery must rely for the maintenance of quality standards.

To anyone not versed in the needs for strict control, it must be exasperating to have a delivery held up because, say, the viscosity is up or down two points on the specification, but nevertheless it has to be done. That is quite apart from other characteristics that are required in the various industries.

The oil is at this stage only in storage or run-down tanks. It has yet to be transferred into drums, small or large, road or rail-tankers, and here it passes to staff experienced in filling into containers. No package is filled until it has been individually examined by the operator, who has a special lamp which he inserts into the drum. For tanker loading the same procedure

takes place, but before such a load is cleared a sample is drawn from the delivery side and carefully examined in the laboratory.

These remarks give some idea of the amount of care that is taken at all stages of production and delivery and it is certainly true that the resulting home-produced white oils compare most favourably with any offered by competitive sources.

This talk would be incomplete without some mention of individual, national and international standards of purity. This is a very complex subject and it really requires a special study, but one or two points are mentioned by way of illustration.

It is well known that the most generally recognised standard of purity in this country to-day is that laid down in the British Pharmacopœia. It stipulates, amongst other things, a minimum gravity and viscosity, the maximum amount of solid paraffins and the degree of carbonisable substances. It goes to a great length to explain how these carbonisable substances are determined and measured.

The Statutory Order No. 1545 covering the Mule Spinning Special Regulations for Health states that "white oil" means a hydrocarbon oil of petroleum origin which has been drastically refined with sulphuric acid and conforms to a specified colour and viscosity. The specified colour is Plus 30 on a Saybolt colorimeter and that means a water-white oil.

Colour, or rather lack of colour for this class of oil is a good guide to standard of purity. It is, however, remarkable that so far there are no standards laid down on an official scale for stability, which is a very important characteristic for such oils not only in the cosmetics industry but in many others. For that matter it applies equally importantly to many other commodities. A good deal of research on these points has been done in recent years, and the time seems ripe for some drastic change in standard of measurement of purity along spectrophotometric lines.

Each national pharmacopœia stipulates, for medicinal liquid paraffin, a sulphuric acid test. This test is a relic from the days when refinery procedure was less drastic and stringent than it is to-day. The purpose of the acid test was to ensure, in the days when Russian crude oil was processed, that there should be no perceptible quantities of aromatic hydrocarbon molecules left in the refined material. Investigations during recent years have shown that the sulphuric acid test is no quality criterion in this respect and that, in fact, it will fail extremely highly refined products if the conditions are such that there are no aromatic bodies for the sulphuric acid to sulphonate so that, instead, the acid dehydrogenates some naphthenic molecules which, in their turn, then become sulphonated.

Most medicinal liquid paraffins show a characteristic band structure in the near ultra-violet region of the spectrum with a maximum at about 2710 Å. a minimum at about 2490 Å. and often a subsidiary maximum at

about 2780 Å. A great deal of work has been done to show that the quality and stability criterion for medicinal liquid paraffin can be based on such spectrophotometric data. The gist of this work was published in the 1951 volume of the *Journal of Pharmacy and Pharmacology*. The criterion is briefly this: for medicinal liquid paraffin to be stable, the ratio of the absorption intensities of the maximum at about 2710 Å. and the minimum at about 2490 Å. must be at least 2, and the absorption intensity of the maximum not larger than $E_{1\text{cm}}^{1\%} = 0.1$. When a liquid paraffin shows very little absorption in this spectral region, the absorption intensity at 2710 Å. must be smaller than $E_{1\text{cm}}^{1\%} = 0.006$ for the material still to be stable and of good quality when its spectrum does not show any band structure in this region.

Thus, a quantitative assessment is given of the quality and stability of medicinal liquid paraffin by two figures derived from the ultra-violet spectrum of the material; its absorption intensity at 2710 Å. and the ratio of the absorption intensities at 2710 Å. and 2490 Å.

For other white oils similar types of criteria are easily possible.

From this brief description of one branch of research, it will be realised that the manufacture and use of white oils is a progressive business. Research and development work in this group of specialised products has the continuous attention of chemists and physicists not only in the oil industry but in the research laboratories of the many industries and scientific institutions concerned with their use.

RECORDS AND COSTINGS IN THE PERFUMERY AND COSMETIC INDUSTRY

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A straightforward method for the easy control of stock and the calculation of costs is described, and specimen record cards are given.

INTRODUCTION

THERE IS no need to stress the value of keeping records and calculating costs in an industrial concern. It is only by making use of these that such an organisation can run on a satisfactory basis, and the advantages are of especial value in times of depression or of severe competition. There is also the possibility that certain lines are being sold at a loss—or have such poor sales that the financial return is not worth the trouble involved or the amount of capital tied up.

Admittedly there are some manufacturers—including compounders and cosmetic manufacturers—who run at such a high rate of profit that there is

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