# JOURNAL

#### OF THE

# OIL AND COLOUR CHEMISTS' ASSOCIATION





#### September 1966

#### FOUNDATION LECTURE

The decoration of polyolefines for packaging

An investigation of the ammonia and heat tests for the yellowing of white paints

Coloured pigmentation for electrodeposition

Prefabrication primers for structural steelwork

\* 8TH AUSTRALIAN CONVENTION REPORT \*

For fuller details of contents and page references see page xxiii

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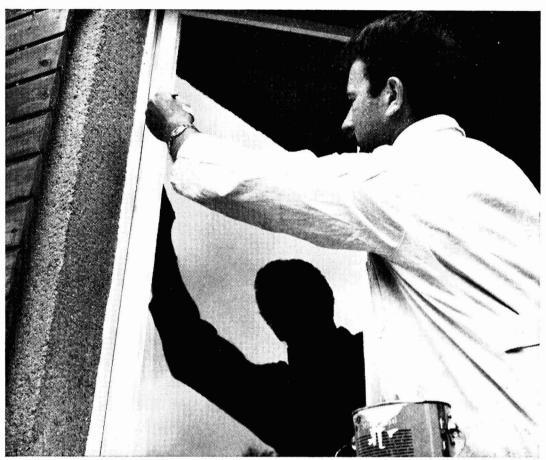
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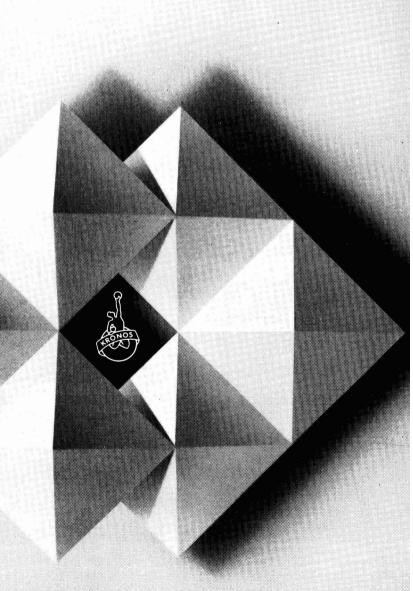


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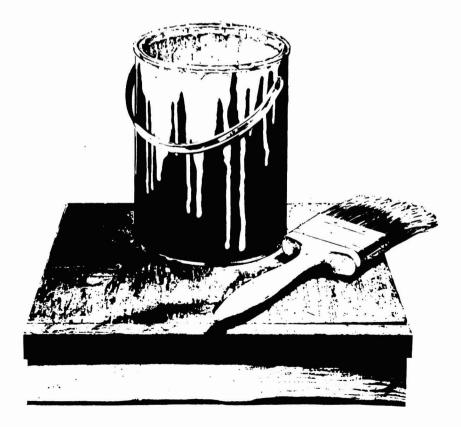


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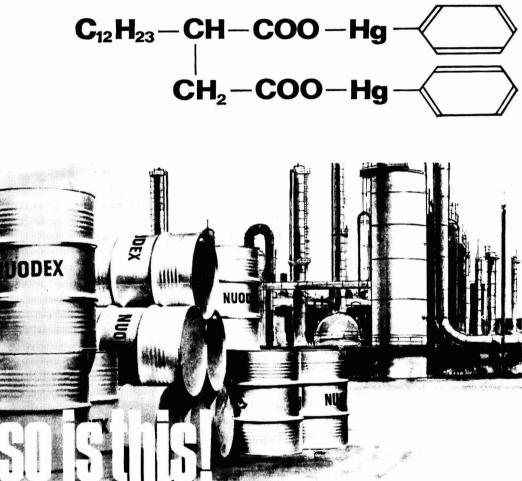


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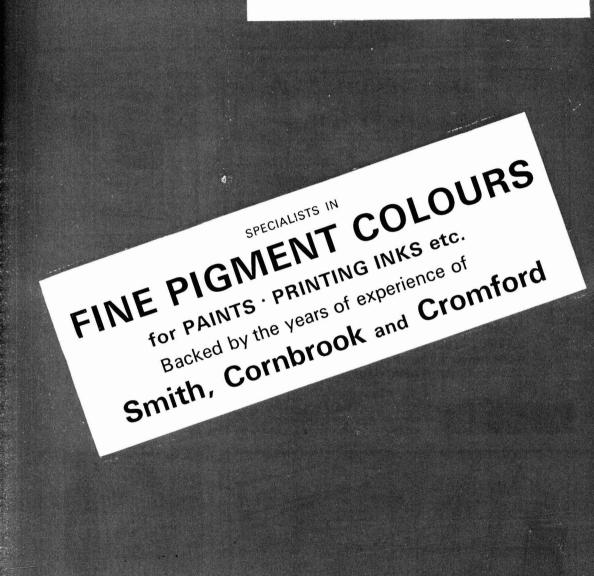
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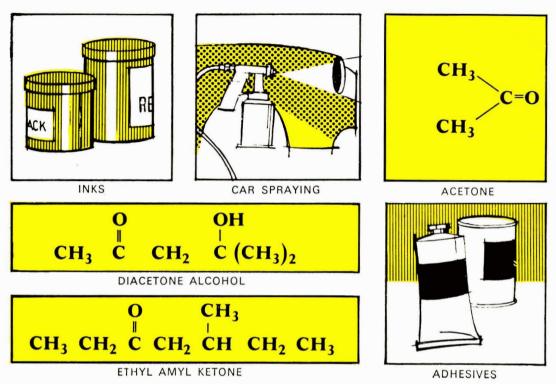




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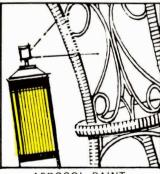


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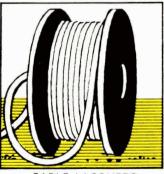
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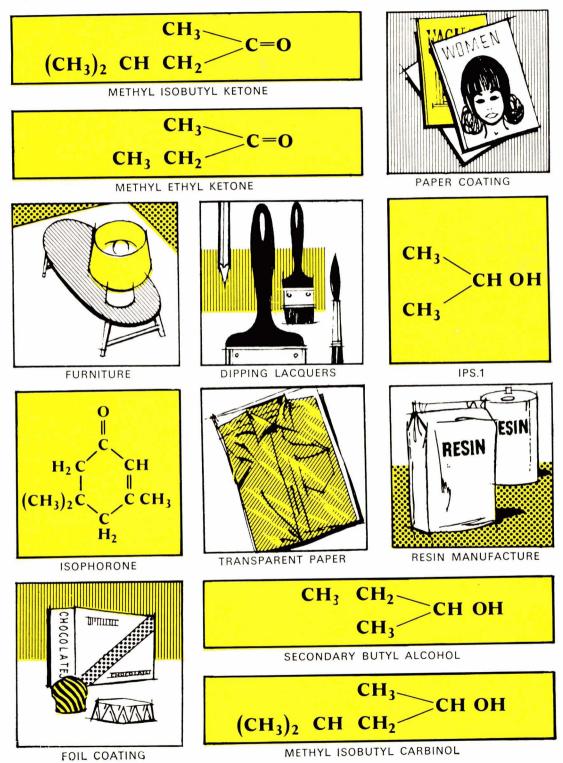


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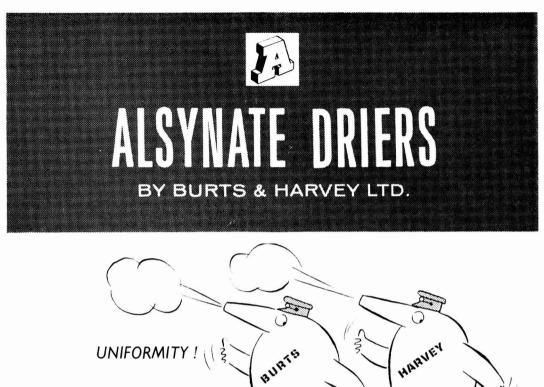
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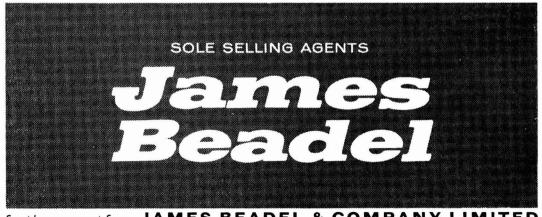
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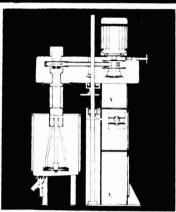
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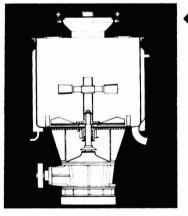
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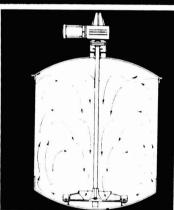
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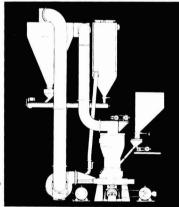
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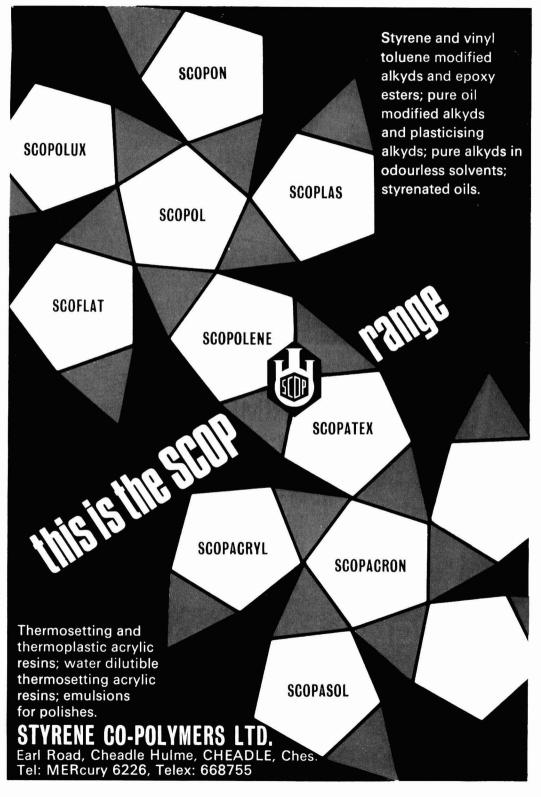
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Director of Research of the British Ceramic Research Association and Chairman of the Committee of Directors of Research Associations.

#### Mr. President, Master, Ladies and Gentlemen,

You have conferred upon me today the privilege of delivering your Association's Foundation Lecture. To have refused your invitation would have been unthinkable, yet to meet the occasion worthily seems impossible. You will know, of course, that I am not, scientifically speaking, of your company, but, in another and wider sense, we are indeed of one brotherhood, for it is our task to serve, in our various ways, the national economy by providing industry with scientific services so that it may rest securely upon a sure base of science rather than upon the quicksands of empiricism and expediency.

I have chosen as the title of this lecture a provocative generalisation made by George Herbert over 300 years ago, for my main theme will be co-operation. The very existence of your Association, Mr. President, shows at once that you have an investment in, and a feeling for, co-operation in your own field, and indeed, it would be easy to multiply such instances. But I shall speak of cooperation in wider terms—of co-operation in industrial research, which is a reality, and of co-operation in training, education and the provision of manpower, which is still largely a dream.

#### Co-operative research in Britain

I propose now that we should look at co-operative research activity in the United Kingdom. It is an activity which was originally peculiar to ourselves, but one which now appears, in one form or another, in many other countries. It is in fact the activity represented by the existence of 48 industrial, grant-aided research associations, covering about 70 per cent of British industry, measured by product value, employing some 6,000 scientists, engineers and technologists, and deploying a total income of about £12m a year, three-quarters of which, approximately, derive from industry itself and about one-quarter from government.

Many of you will be familiar with the history and origin of these bodies, but I hope you will bear with me if, for the sake of completeness, I give you a brief historical background. Until the turn of the century most research in this country was carried out in universities, and so little was done for or in industry that when Kelvin visited USA in the 1880s he was driven to remark on the use that the industry of that country was already making of science and scientists, particularly in its steel industry. In the declaration of the State's interest in applied research we were anticipated both by Germany and by USA for the PTR was established in 1887, and the Bureau of Standards a few years later, but it was not until 1902 that our own NPL was formally opened by the then

<sup>\*</sup>Presented to the Association at Painter Stainers Hall, London, E.C.4, on 30 June 1966.

Prince of Wales. Still, events then moved on fairly quickly, spurred of course by World War I, during which DSIR was founded (1917). I well remember a remark made by the late Sir Richard Glazebrook in a lecture he gave to the NPL in 1933. He said: "The years that followed the conclusion of the Franco-German war saw a great expansion of industry in Germany, an expansion supported by von Helmholtz and Werner von Siemens on the scientific side. They knew that science could give help of the greatest value to technical endeavours, and they pointed out the methods by which this could be done." The result, as I have said, was the foundation of the PTR, and it is curious and interesting that 40 years had to pass before a similar reaction, stimulated by the stresses and crises of war, was felt in this country.

DSIR in 1918 was given an allocation of  $\pounds 1m$  to enable it to give assistance to those industries which were sufficiently forward-looking to help themselves to put their research and development affairs in order. There was a ready response, with some non-starters, but the whole operation was well under way by 1920. There are now 48 research associations, with incomes ranging from under  $\pounds 20,000$  p.a. to over  $\pounds 1m$ .

It could perhaps be argued that the research associations represented an attempt to bridge the gap between university and state research on the one hand and such large private industrial laboratories as there then were on the other. The smaller firms could not effectively take advantage of either and, short of groupings and take-overs, which were much less fashionable then than now, the solution seemed to be to co-operate in research—to have a stake in a laboratory and its facilities which would be beyond the resources of any one small firm. It was argued that in this way the small firm would survive as a viable economic unit and that all could share equally in research and development. I have sometimes reflected that in the research associations we have an excellent answer we did not expect to a question which was incorrectly posed! That the research associations make a massive contribution to British industry is undeniable: that the small firms really benefit as they were expected to do seems questionable, and for a very obvious reason-development to the level of successful commercial exploitation is, in general, a much more expensive exercise than research. After over 40 years of research associations, there has to be some rethinking of their role if for no other reason than this. Whether by design or circumstance, research associations started their existence by setting out to help industry as it stood at that particular moment in time : some are still locked in this stasis by the sort of negative feed-back imposed on them by their industries and industrial committees, but others are striving, as eventually all must, to turn their endeavours more and more to the problems of industry a generation hence. And implicit in this, of course, is the concept that research associations are oriented to serve industry, and not to serve *individual firms*. This may have a hard ring to it, and before a less liberal audience I might well have to make the point more diplomatically, but it is an inescapable concept and carries with it some rather uncomfortable economic implications.

Research associations continue to derive their funds very much in the pattern laid down 40 years ago. Industry supports them according to various formulae, and government offers a block grant conditional upon a minimum investment from industry, plus an incentive grant based on income over this minimum, to a set ceiling. This equation is reviewed quinquennially for every research association, and it is an exercise in extrapolation, not to say crystalgazing, for a director to forecast with conviction, and argue with compulsion, the case for continued expansion from one quinquennium to the next. There is, indeed, a good deal of financial bargaining of one sort or another, and this contributes to some of the problems of running an RA.

I have already indicated briefly the financial size of research associations. On the last available figures, which refer to 1964, the smallest had an income of just about £13,000 and the largest 100 times this—a very broad spectrum indeed. Four research associations enjoy—if that is the "mot juste"—a statutory levy : this requires a united demand from industry and parliamentary action. The rest depend largely for their industrial income upon agreed subscriptions or upon levies related to volume or gross output of product, or to number of operatives or to the wages bill. It seems likely that some sort of ad valorem formula is the most equitable. There is, of course, a fine field for economic dispute here, but it can be argued that the cost of research must ultimately be borne by the customer, and so it should be built by a proper cost structure into the price of the product : the price in turn will change with the changing economic climate, so that levies should be self-adjusting in regard to inflation.

Government contributions, now standing, as I have said, at about one quarter the total income, have, on this average figure, diminished relatively over the years. This is not to be taken as a contentious statement, nor simply accepted at its face value. Government contributions do in fact range from a "low" of about 10 per cent to at least one case of over 100 per cent of the industrial income. We should note too that this revenue grant, as it is called, is not in any way controlled as to its use : its deployment is entirely a matter for the Director and his Council. In this respect Britain remains unique in the partnership between government and industry in the field of civil science, and in spite of this government investment, the research associations are completely autonomous.

There are no formal links between the various associations, but there is an increasing amount of collaboration. This has been fostered by the formation of the Committee of Directors of Research Associations, and is manifest in the operation of certain joint working groups, such as the Industrial Automation Group, the Building Materials Working Party and a Public Relations Group, and by the operation of joint projects in certain fields. CDRA has no formal status, but it is increasingly recognized by the government as a body which can be usefully consulted on matters relating to industrial research. CDRA has recently established fruitful contacts with the Confederation of British Industry and we feel we are now entering upon a new phase in the history of co-operative industrial research in Britain. When the first research associations were formed, they were committed wholly to the interests of the industries they served, but by a process of natural growth and evolution, most of them find themselves now involved in tasks which transcend their original boundaries, and committed to a national, as well as a local, role. Those of us who have the responsibility of directing the British research associations feel that we are part of an integrated national network, as well as being the servants of our own industries : this is indeed the result of partnership with government.

Now, after this factual and, I hope, moderately enthusiastic account of the British research association system, it turns out that we are confronted by a very disturbing statistic. It appears that research association activities represent probably something under 2 per cent of the research and development effort of British industry. Can this really have a significant effect upon our affairs? Well, first we have the measure of confidence in the system displayed by British industry : as I have said, some 70 per cent of British industry is involved. The industries outside the system are mostly in the modern, science-based group, which are weak in regard to co-operative work because they generally consist of large companies which are technically self-sufficient. But even this is too facile a generalisation : if we take the "top 300" industrial and commercial concerns in Britain, we find that in the years 1960 to 1966 large company membership has increased from just over 1,000 to over 1,300. Some of the large firms not involved with the grant-aided research associations are, of course, concerned in another form of co-operation through private—that is, nongrant-aided-organisations, of which there are, interestingly enough, about 12.

What then, does this 2 per cent mean? It clearly does not mean that industry has no confidence in the system : it could well mean the recognition of a quite large amplification factor resulting directly from co-operation. This amplification factor has in fact been put as high as 100: my colleague Dr. Douglas Hill, addressing the Parliamentary and Scientific Committee in 1961, said that on a conservative estimate the direct benefit to industry today from the work of the research associations is something in excess of £100,000,000 a year. This is indeed an amplification factor of around ten : I have quoted the higher figure just because it in turn has been quoted from time to time.

How have the research associations achieved this? There are obvious advantages in co-operative laboratories which I need not specify-economy in man-power, equipment and real estate. But, additionally, as a matter of historical development, the British research associations have such close contacts with the industries they serve that they are indeed deeply involved in their problems, so much so that their outstanding achievement over the past 40-odd years has not been so much in the achievement of the spectacular "breakthrough "-although instances are not lacking-but in completely changing the climate of the industries they serve, so that productivity, efficiency and quality are continually advancing. The concept of the "breakthrough" as an index of performance is misleading and almost dangerous. A " breakthrough " has a powerful news value and is, instantaneously, at least, very impressive, but it cannot, and must not, break our commitment to the continued evangelism for a belief in a better understanding of the scientific background and the deployment of scientific techniques. I do not know that one can even plan for a "breakthrough," but we can and must plan for the unremitting advance of our scientific and technological frontiers, and this in the main is what the research associations have done, and have done well.

Looking back over the years since the end of World War I, it is difficult to see how industry, particularly traditional industry, could have sifted through the massive volume of technology it had inherited and could have placed it on a good scientific foundation without the research associations. It is never very fashionable to applaud government in this country, but it has been a remarkable piece of non-political government over the years to have provided an external stimulus—indeed, a nucleating force—which has enabled industry to face this situation. This perhaps does not mean so much to those industries which sprang, fully-armed, from the laboratory bench—chemical and petrochemical industries, pharmaceutical, communications and the like—but to those industries which started far back in the fields, like yours and mine, it has proved a most effective catalyst, and as I have said, the main return has been the change in industrial climate and the continued revitalising of those basic industries which are still our economic strength. Part of this exercise has been the encouragement, overt or covert, by the research associations, of the growth of scientific and technical man-power within the industries they serve, and the help they have given in setting up laboratories, however modest, in the firms themselves. Indeed, any research association must stake a claim in the wider field of education and training in and for its industry, or it will be operating in a vacuum.

The research associations have carried out a good deal of objective basic research which has been, on the one hand, beyond the scope of individual firms and, on the other, of a kind outside the interest of university schools. The sort of work they have to do is dictated by the necessity of looking ahead in their chosen field, and has to be tackled irrespective of how difficult it may be and whether it corresponds to any particular field of academic interest. Numerous important techniques have emerged from research association work, one of the most notable being partition chromatography, developed at the Wool Research Association.

A little-publicised but immensely valuable part of the work of the research associations is the contribution made to health and safety in industry and in the community. Again, it is difficult to see how this contribution could have been made quickly and effectively other than by co-operative effort. In my own industry we have successfully developed protection for the operative against the fearful hazard of silicosis: we are now involved in a joint enterprise with the Cast Iron and Steel Castings Research Associations directed to an attack over a wider front on the mineral dust hazard. Safety devices of every kind, hygiene in food factories and laundries, radiation hazards—to all these and many others research associations have made massive contributions. And in doing this the research associations have done magnificent liaison work between the Factory Inspectorate and the industries themselves.

There is in some quarters an inclination to regard research associations as trouble-shooting agencies. I insist that this is only a subsidiary role, but it represents still a formidable force. It is of benefit to the smaller firms, but it is even more valuable to the larger firm with some scientific or technical staff of its own. There is no doubt that this sort of service contributes immensely to the establishment of the right climate for development, and it is peculiarly a product of co-operative effort because it depends utterly upon the accumulated know-how and expertise of the research association staff, which in turn is based on their wide and peculiar experience of their industries' problems.

Another contribution made by research associations is in the establishment of standards and specifications, and especially in dealing with foreign specifications which could bear heavily on export trade. A single firm, or

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even a trade association, could well find itself powerless here, and the research association can provide the protection and advice necessary to place the homemade product correctly against its foreign competitor. Whether Britain formally enters the European Common Market or not, there is a great amount of technical interchange and movement towards standardisation with our European friends, and again a research association is certainly the best, and in some cases the only suitable, agency for this work.

#### Co-operative research in other countries

Before we go on to consider the reverse of this attractive coin I have been spinning before you, it may be worth while to comment upon the scene elsewhere: the co-operative research organisations in other countries. There are about 180 research associations in Europe, one third of them in Western Germany, and about 130 other forms of co-operative research organisation, not receiving government support. We in CDRA have during the past year made occasion to visit Europe to see at first-hand how these organisations operate. There is a great diversity in operating philosophy and in financial structure and, of course, a historical background quite different from that of the British prototypes. Unique amongst world organisations is the Netherlands TNO structure, which is a body set up by government and underwritten by government, charged with developing industrial research. It has discharged this responsibility by setting up a number of research institutes, some of which operate as research associations, with trade associations rather than individual firms as members. Indeed, TNO could, in principle, function without industrial support, but it would not wish to do so. Nevertheless, the total industrial support of TNO is much less, proportionally, than in the United Kingdom. A somewhat similar system operates in Norway under the Royal Norwegian Council for Scientific and Industrial Research. Both the Central Organisation TNO and the Norwegian Council are voted a sum of money by the respective parliaments to deploy as they think fit. In Norway, the Council has set up a Central Research Institute, which does a good deal of sponsored research in the field of applied physical sciences, while Norwegian industry has set up its own research associations independently. Now the Central Council is beginning to consider support to these research associations as their financial burdens increase.

In Germany, almost 80 per cent of the co-operative bodies are connected in some way with the Technical Highschools. They receive money both from the Federal and Länder governments, but only against approved projects, and this is distributed by a body called the Arbeitsgemeinschaft Industrieller Forschungsvereinigung e.V. (AIF) which acts as a negotiating body between the Institutes and the governments. The German associations have as great, if not a greater, stake in training for industry as in applied research, and indeed this close partnership between education and research is one which we should examine closely ourselves.

In France, many of the co-operative "Centres Techniques" have been established under the law of 1948, which provides for a statutory levy, raised as a tax within the Annual Finance Act. Thus government has no direct investment in these organisations, but has provided the legislation whereby they have their being : the French government, however, reserves the right to certain appointments on councils and has certain powers of veto.

To British eyes the continental scene presents, perhaps somewhat meretriciously, a picture of financial stability which we might envy. What it does not show, however, is that close contact with industry which, as I have said, is our great strength at home. Indeed, even in America, where I know some co-operative bodies in my own area of interest, I am not persuaded that this intimate involvement in industry is really apparent. Curiously enough, in spite of these differences and in spite of the different historical backgrounds. it is clear when we talk to our colleagues overseas that we have all just about contrived to reach the same problems at the same time by vastly different routes. These are basically problems of manpower and money : they derive from the circumstance that industry's desire for scientific services and investment in research now appears, everywhere, to go beyond industry's ability to pay. There, you note, I have deliberately said *ability*, not willingness. This is a hard economic fact : research, even at the level of scientific services, is a monumentally expensive exercise, and the time is fast approaching when expenditure on this essential element of our modern life has to be rationalised. As I have said elsewhere, all rational research is good, but only some is timely, and it is increasingly the responsibility of those concerned with science, either at a national or lower level, to realise the truth of this before we have lost the balance between what is intellectually exciting and what is economically necessary. Again, I hope I have chosen my words carefully: I have spoken of balance. In no circumstances could a nation such as ours not afford to invest in the pursuit of knowledge for its own sake: without this, not only would our own technology fail to advance, but we could no longer avail ourselves of advancing technology elsewhere. But equally we must advance our technological frontiers, and the plain truth is that at the moment we do not seem-and we are not alone in this—to have either the men or money to do both on the scale we should like. These are the vital raw materials of research and they must be conserved and rationally deployed.

#### Investment in research

How much money is spent on research? While absolute figures are impressive, if only for their astronomical size, they are meaningless unless related to some index of economic activity. One such index is the value of the gross national product : another is based on the investment per head of population. The figures I am about to quote are taken from a recent issue of the OECD *Observer*, a journal to which I make my acknowledgements. The figures refer in fact to 1962 : there is always a time lag in producing reliable statistics of this sort.

In the UK, with a GNP of about  $\pounds 28.5 \times 10^9$ , our gross expenditure on R and D was  $\pounds 634m$  or 2.2 per cent. Of this, about  $\pounds 250m$ , representing some 0.9 per cent of the GNP, was spent on R and D for defence purposes, so that about 1.3 per cent of the GNP was spent on civil science, representing about  $\pounds 8$  per capita. In the rest of Western Europe, the total expenditure was around 1.5 per cent, the civil element being about 1 to 1.2 per cent, while in America the figures are 3.1 per cent and 1.6 per cent, so that here nearly one half the R and D effort is directed towards defence and space research. As far as civil R and D

is concerned, the American expenditure per capita was about £16. It is believed that for USSR the total figure may be as much as 3 per cent. While these statistics are interesting and give one an appreciation of the magnitudes involved, we also have to remember that research costs, in which the principal element is likely to be research salaries, differ from country to country, and it has been estimated that US costs are at least 50 per cent greater than in Western Europe, where costs again are about double those in USSR.

As to manpower, the total investment in R and D in America is 1.1m people, just about twice that in Western Europe and five times that in UK. Setting these figures against working population, in America about 1 per cent of this body is involved in R and D compared with 0.6 per cent in UK and about 0.4 per cent in Western Europe. Russia must be somewhere between UK and USA and probably nearer the latter.

I think these figures focus our attention upon the need to distinguish between percentages and absolute values. Suppose we have a small sector of industry worth about £5m and suppose this undertook to contribute to a co-operative research venture at the rate of 0.2 per cent, which is somewhere near the mean for British research associations. This yields £10,000, which would pay for no more than three or four qualified men with support staff. It is a reasonable contribution but yielding a scarcely viable working unit. In the same way, a small country like the Netherlands investing 4.5 per cent of its manpower in R and D can only produce a total R and D force of 33,000, of which about one third are qualified scientists and engineers. Higher investment in scientific activity, however statistically beguiling and however alluring, must inevitably react upon other vital aspects of the economy, notably management and teaching. Once again we face the problem of balance, and to achieve this feat of equilibration we have to ask how far economic growth is really related to investment in science. That it is related is manifest : in almost every aspect of our life, the situation has been transfromed, for good or ill, within the lifetime of most of us, simply because more and more science has been injected into the system of living. But the hard fact is that although we believe we see this relation, it is by no means a simple linear association. Indeed, Professor Bruce Williams, who has put in a good deal of work in this field, is on record as saying that there is no significant positive relationship between the research and development rate and the economic growth rate, and he points out that Britain and America. clear leaders in the former, are near the bottom of the table in industrial growth. You may argue that Britain and America had reached a point on the curve where the slope was falling off, and that the others in the race were so far behind that they are still enjoying the region of high gradient. Be this as it may, it is an uncomfortable fact to face. Bruce Williams has in fact offered some cogent comments on possible reasons, but by far the most powerful, in my view, is that in which he says that the important part of the whole process is not the overall expenditure but the scientific manpower at the back of it.

#### The manpower conflict

We see, indeed, in the problem of manpower, craft pitted against craft once more, and making no living. I spoke earlier of the risk of losing the balance between what is intellectually exciting and what is economically necessary : it is easy to translate this problem into the balance of manpower. Not only is this in short supply, but urgent demands are put upon it from all quarters. The more we put into research the less, as I have already pointed out, will be available for other vital activities. In speaking of the German scene, I noted that a substantial effort went into training men for industry : the men so trained actually go into industry, and indeed it seems very likely that the Germans use a higher proportion of their qualified scientists and engineers in productive industry than we do. Again, Bruce Williams suggests that there is a vast amount of evidence that, could we but deploy more scientific manpower in production and related processes, then would we bring about a faster and more extensive application of science to industry, and with it a greater economic growth rate. You may well point to USA and remind me that there it is always argued that productive industry employs a high proportion of scientific manpower, and yet is lagging behind in economic growth rate. Well, this is a fact, but we do not have all the data : is it once again the distinction between percentages and absolute values?

I am now going to make a point which may well shock you. I was a few years ago a lone voice, but now I believe I have many supporters. The observation with which I shall shock you is that I believe our investment in education has gone adrift and that the balance between investment in universities and the rest of our requirements has been seriously disturbed. I believe that too many people are going to universities for the wrong reason and that far too many people are remaining at universities to do research which, however good, is not necessarily timely, and too much of our scarce manpower is being sequestered in areas remote from our real economic needs. I have felt this for some time : it was sharply focused for me when recently I interviewed a young man for appointment at my laboratory. He was eminently suitable : he could have joined at a salary not far short of £1,000 p.a. and he could undoubtedly within three years have made a valuable impact on a problem which is not merely the concern of my industry, but which is of some significance nationally. He has opted to accept a research grant and to stay on at university to work for a Ph.D. This he will undoubtedly get : he will complete a research which, as I have said, however good is not likely to be particularly timely; he will have cost the country perhaps £10,000 and at the end of it all he will not necessarily be better or worse fitted for a task in industry than he is at present. Is this a good investment in manpower?

A recent White Paper gives the estimated government expenditure on research and development for various sectors. In the current year, 25 per cent goes to universities, 9 per cent to the Ministry of Technology and 16 per cent to the Science Research Council. The Ministry of Technology is indeed the low man on the totem pole : 20 years ago DSIR had 50 per cent more than the university investment.

In putting these views before you I am not in any way condemning our universities. I am urging that in this moment of history we have our balance wrong and that we are not really earning enough as a nation to spend our money in this way. How then do we redress the balance? I think we begin by saying that the universities, particularly those of great tradition, must be sustained as the nurseries of pure science and that there must be a minimum national investment in this. A chosen few of the best graduates must remain to

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carry on the torch of learning and discovery—I have indeed said earlier that without this we could not advance the frontiers of technology. Some of the others must be encouraged to go directly into industry, to industrial laboratories and to research associations. If they too have their sights fixed upon a Ph.D, then today the means are to hand. London has always offered this opportunity, and some other universities now permit a master's degree to be taken externally. But now we have the Council for National Academic Awards, that catholic successor to the National Council for Technological Awards—The Hives Committee—which can under its charter award the degree of Ph.D for what is essentially extramural work.

It is worth reflecting for a moment on the history of this, because it develops from a partnership between industry and government in the field of technological education. In 1955 the National Council for Technological Awards was set up by the then Ministry of Education under the chairmanship of Lord Hives. By a skilful integration of much of what had been known and tried before. the now familiar pattern of co-operative sandwich courses was established, leading then to the Diploma in Technology, now, under CNAA, to a bachelor's degree. In my view this has been the boldest and most imaginative educational exercise of this century. The success of the scheme lies not so much in the detailed tactics, for these had in some sense been explored over half a century or more. but rather in its enlightened strategy, for it seeks to build a man into his industrial career from the moment he leaves school. To do this it canvasses the assistance of industry both in selecting students and in providing schemes of training. Industry has responded well, although the burden is heavy. But one is impressed by the confidence of those colleges which, in their new life in the Robbins era, continue to pursue the pattern laid down by Hives, and by the appreciation expressed by the employers of those who have graduated under the scheme.

Here we have a new source of trained men for industry : here we see, in the newer colleges of technology, opportunities for co-operation in research and training which might well recognise some indebtedness to the German scene.

The research associations have, inevitably, been the outposts of what it is now fashionable to call the interdisciplinary sciences. It has been their especial task to produce a race of converts from the classical disciplines of science to tackle the regrettably unstylised problems of industry, and to do this some excursion into education has been inescapable. The most obvious task is that of securing a recognition of industrial problems and training needs, and a first step here has been taken over the years by the appointment to university staffs as honorary lecturers of people from research associations which are either geographically or technologically closely placed to the university. It is, indeed, very much the ambition of the principal of one of our famous colleges of technology to see this idea extended to actual secondment and interchange of staffs. In addition to this sort of link, many research associations sponsor work at universities and colleges of technology, and provide occasional lecturers for short courses. The essential return from these exercises is not so much the completed research report, but rather the continuing awareness in the university of the problems of industry and of the possible avenues of employment for its graduates.

Research associations have been quick to support the Dip.Tech. scheme, both by providing training places—and here your own Paint Research Association has an impressive record—and by helping to establish new courses.

As technical colleges develop, perhaps into Colleges of Advanced Technology, and as CATs themselves assume a steady state, we are made aware again of the philosophy which argues that good teaching at high level cannot proceed without good research schools. In this transient phase, it is not always easy to see how a coherent research policy can emerge in a college : staffs are new, and are anxious to retain their individual interests, while their conditions of service often leave only marginal time for research effort. Research associations are surely in a strong position to help establish growing points for research in these developing colleges, and perhaps to encourage a more generous outlook in the allocation of staff time to research.

Quite apart from their conjoint efforts with established centres of education, research associations do an immense amount of teaching by "direct approach." There can be few, if any, research associations which do not hold regular schools and conferences within their own walls, ranging from simple exercises in training in techniques for the younger people in industry to high level internationally-based symposia. That the research associations will have an important role as ITA develops is certain.

One positive move of very great interest is the recent re-establishment of the Hydromechanics Research Association on the campus of the College of Aeronautics at Cranfield. The crystallisation of the fascinating possibilities of this situation will deserve close attention and one would hope to see a prototype pattern emerge which might form the basis of new conjoint institutes.

### Conclusion

"Craft against craft makes no living." Have I indeed in this survey of some of the characteristic economic, industrial and educational conflicts of this present age, contrived to establish this thesis? You will decide, but I at least am persuaded that, in presenting to you the story of the British research associations, I have shown that craft moving with craft here has been a most profitable exercise. What I have not ventured to say is whether craft against craft is good or bad when we move from research to development—when we step across the development gap which separates the laboratory from the production unit. It may well be that here the thesis is not wholly tenable: the normal forces of industrial competition will always drive the livelier, more efficient firm ahead, even though all start with the same advantages of research.

But the first of our real conflicts is not this : it is the conflict between the urgent need to provide science for industry and the ability of industry to pay. How far should government support develop? That the research associations have a national responsibility I believe I have demonstrated to you : this alone provides sufficient case for government involvement, but it tells us nothing about the magnitudes involved. Government is to diversify the activities of some of its own laboratories : how far can this be made a real investment in industrial research? Do we simply replan research programmes, or do we re-deploy staff?

The manpower conflict is perhaps more formidable even than this financial conflict : inevitably, the two are mutually entangled. The present position is that

the universities and SRC together spend more than four times that spent by the Ministry of Technology. The concomitant aspect of this is that more people are being consumed by the universities than the nation can at present afford. I have already suggested how the balance might be redressed even if the conflict is not fully resolved. More graduates should be persuaded to go directly into industry on graduation and perhaps return later for doctorate studies or to take advantage of the CNAA post-graduate schemes.

Finally, how far is training or education a responsibility of co-operative research bodies? There is an obvious acknowledgement of this responsibility in the present exercises of many research associations—can this conflict be finally resolved by conjoint institutes?

I must leave you with these questions : if I have engaged your ear and perhaps beguiled your minds with my observations upon our present scene you will perhaps allow that I have discharged the agreeable task which has been laid upon me today.

### **Transactions and Communications**

# The decoration of polyolefines for packaging<sup>\*</sup>

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### Summary

A survey is made of the various methods used for the pretreatment of polyolefine surfaces to ensure satisfactory adhesion of printing inks and other surface decoration. This is followed by a consideration of the various printing processes, particularly dry offset and silk screen, and the necessity for the use of barrier lacquers to decrease the permeation of certain types of lacquer and comparisons made of their effectiveness in reducing the permeability characteristics of a series of chemical compound such as are used in, for example, adhesives, cosmetics, medicaments, etc. A comparison is also made of the permeability characteristics of a series of typical commercial products which could be packaged in low density polyethylene containers. Consideration is given finally to two methods of decoration not involving the use of fluid decorating materials, i.e. hot foil stamping and a hot transfer process.

### La décoration des polyoléfines destinées aux emballages

### Résumé

On passe en revue les méthodes diverses propres au traitement préparatoire des surfaces de polyoléfine pour assurer l'adhésion satisfaisante des encres d'imprimerie et des autres types de décoration superficielle. Puis on considère des procédés d'imprimerie, surtout l'offset à sec et le pochoir sur soie. La nécessité de d'utiliser des vernis-barrières afin de faire réduire la pénétration de certains types de composés chimiques est soulignée. On pose des exemples de l'emploi des types divers de vernis et l'on fait des comparaisons de leur efficatité pour réduire la perméabilité d'une série de composés qui se trouvent, par exemple, aux adhésifs, cosmétiques, et médicaments, etc. On fait également une comparaison de la perméabilité d'une série de commerce que l'on pourrait mettre en emballages de polyéthylène de densité faible. Enfin on considère deux procédés de décoration qui ne renferment pas d'utilisation des matériaux liquides de décoration, c'est-à-dire, l'estampage à chaud de feuille plastique et un procédé de décalquage à chaud.

### Das Verzieren von Polyolefinen für Verpackungen

### Zusammenfassung

Ein Überblick über die verschiedenen zur Vorbehandlung von Polyolefin-Oberflächen üblichen Verfahren, welche eine genügende Haftung von Druckfarben und anderen Oberflächendekorationen sicherstellen sollen, wird gegeben. Die Betrachtung der verschiedenen Druckverfahren schliesst sich an, insbesondere des trockenen Offset- und Siebdrucks, wobei das Erfordernis Isoliergrundlacke anzuwenden, um das Durchschlagen gewisser Arten chemischer Verbindungen herabzusetzen, betont wird. Es werden Anwendungsbeispiele für verschiedene Lacksorten gegeben, und deren Wirksamkeit bezüglich Herabsetzung des Durchschlagens einer Anzahl chemischer Verbindungen, die z.B. in Klebstoffen, Schönheitsmitteln, Medikamenten etc. vorkommen, verglichen. Ebenfalls werden die Durchschlagscharakteristika

\*Presented to the London Section, 16 September 1965.

einer Anzahl typischer Handelserzeugnisse, die sich für das Verpacken in Behältern aus Polyäthylen von niedriger Dichte eignen, verglichen. Schliesslich werden noch zwei weitere Verzierungsmethoden, die keine flüssigen Mittel erfordern, betrachtet nämlich das Aufdrucken heisser Folien und ein Abziehbildverfahren, bei dem Hitze angewandt wird.

### Декорация полиолефинов в упаковках

#### Резюме

Выполняется обзор различных методов употребляемых в предварительной обработке полиолефиновых поверхностей с целью обеспечения удовлетворительной адгезии печатных чернил и других поверхностных декораций. Затем следует обсуждение различных печатных процессов, в частности сухой офсет и шёлковый экран, и подчеркивается необходимость применения преграждающих лаков для уменьшения проницаемости некоторых типов химических составов. Приведены примеры применения различных типов лаков и сравнивается их эффективность в снижении характеристик проницаемости ряда химических составов которые применяются в производстве, например, клеев, косметических и медикаментных изделий. Сравниваются также проницаемые характеристики ряда типичных торговых продуктов годных для упаковки в низко-плотных полиэтиленовых сосудах. Наконец рассматриваются два метода декорации в которых не применяются жидкие декоративные материалы, т.е. штамповка на фольге и процесс горячего переноса.

### Introduction

The packaging industry uses both thermosetting and thermoplastic materials but, as the bulk of the former are used primarily as closures for containers, the thermoplastic materials are those of principal interest so far as decoration is concerned. Of these, the greatest usage is to be found in the polyolefines. These polymers are used as extruded films for wrapping purposes and for the production of bags, as blow mouldings for bottle manufacture and squeeze tubes, and as extrusions for the bodies of composite containers, occasionally in combination with metal ends and sometimes with injection mouldings for the manufacture of squeeze tubes. Polystyrene is also fabricated by injection moulding to produce such packages as vials for cosmetics and pharmaceutical products; polyvinyl chloride has recently been blow moulded to produce both rigid and squeeze bottles, and polyacetals have been blow moulded to produce aerosol containers. All these types of packages require to be decorated to identify the contents, brand names, user instructions and the name of the manufacturer and/or merchandiser. In this paper it is proposed to concentrate primarily on the methods adopted to decorate polyolefines when used for moulded containers.

The efficiency of the adhesion of the decoration is dependent to a very great extent on the treatment to which the moulded article or extruded film has been subjected before the decoration is applied. This pretreatment is essential in the case of the polyolefines to ensure the permanence of adhesion of inks, lacquers and paints, and is necessitated by the fact that the articles to be decorated have smooth, glossy surfaces which afford little scope for the mechanical keying and bonding of the surface coatings, and the polymers themselves tend to be insoluble, chemically inert and non-polar. Pretreatment processes, therefore, are concerned with surface modification of the plastic and five methods have been proposed for this purpose, viz. (1) Solvent immersion, (2) Oxidation by chemical treatment, (3) Irradiation processes, (4) Electrical processes, (5) Flame treatment.

### Pretreatment

In the method of solvent pretreatment the plastic is exposed to suitable solvent or solvent vapours, when the surface is deglazed and roughened. Solvents used for this purpose include trichlorethylene, perchlorethylene and toluene, and the usual time of exposure is 15-30 seconds. The disadvantages of this method are the necessity to prevent the spread of toxic or inflammable solvent vapours and the fact that the treated surface so produced may only have a limited life. To improve the length of life, particularly in the case of polypropylene mouldings, a suitable resin is sometimes dissolved in trichlorethylene<sup>1</sup> and the article to be treated immersed in the resin solution for about 20 seconds when, on slow withdrawal, a thin resinous film is left on the surface of the plastic. This process is seldom used for film or thin walled containers such as bottles or tubes and this restriction also applies to the oxidation treatment involving chemical reaction.

A suitable oxidation process for polypropylene involves the use of saturated potassium permanganate solution<sup>2</sup> to which has been added 2.5 per cent of concentrated sulphuric acid, the treatment being carried out at 65-70°C for 4-5 minutes. Another solution which has been recommended comprises chromic acid and a typical composition contains one part by weight of potassium dichromate, 20 parts by weight of concentrated sulphuric acid, and 1.6 parts by weight of water. Both these chemical processes suffer from the disadvantage that the treated plastic has to be washed carefully to free it from residual acid and then has to be dried before any decorating process can be applied. Other disadvantages are the relatively lengthy time involved and the hazards attached to the use of highly corrosive liquids.

Another oxidative process, patented by Du Pont<sup>3</sup>, employs an aqueous solution of a halogen, hydrogen halide, hydrogen peroxide, nitrous acid, alkaline hypochlorite, concentrated nitric acid or mixtures of concentrated nitric and sulphuric acids at 25-95°C. The use of ozone has been patented<sup>4</sup> where the presence of an accelerator such as a halogen or nitrous oxide preferably in ultra-violet light is also claimed.

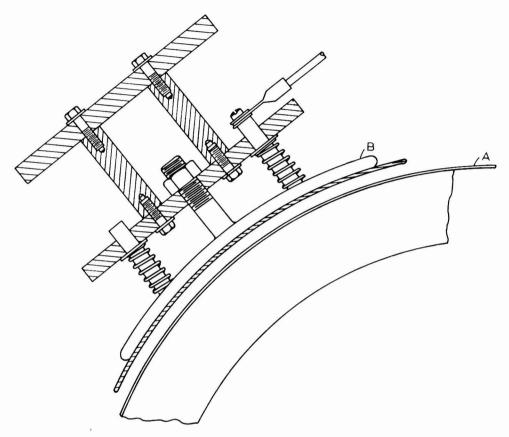
Ultra-violet irradiation has been claimed in another patent<sup>5</sup> where the exposure is to the radiation from a mercury vapour tube, but this requires an exposure time of about 20 minutes, which makes it quite uneconomical for practical purposes.

Another irradiation process proposed is that using  $\gamma$ -radiation produced from Cobalt-60<sup>6</sup>, but this process can lead to marked changes in the physical properties of the polymer due to cross-linking and there is also the disadvantage of the precautions which have to be adopted when handling radio-isotopes in addition to the cost of such materials.

The fourth group of treatments embraces the electrical methods, some of which are used on a considerable scale, particularly in the USA, for treatment of polyolefine films and blow moulded containers. One of these processes, developed by the Visking Corporation<sup>7</sup>, subjects the surface of the film to a high voltage electrical stress accompanied by a corona discharge. The film is fed on to a wheel, which is earthed and acts as a movable electrode, and

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### Fig. 1. The electrode arrangement

fixed electrode. Voltages between 10,000 and 30,000 volts are used and these are produced by built-in transformers from the mains supply. The exposure time is quite short as may be judged from the film speed, which is of the order of 300-360 ft per minute. The higher the speed of travel the longer must be the electrode to achieve the necessary exposure time. The distance between the fixed electrode and the dielectric around the wheel may be between  $\frac{1}{4}$  and  $\frac{1}{2}$  in for films of thicknesses 0.01 in. Similar processes using different electrode forms have been claimed in several other patents<sup>8</sup>. A disadvantage of this method is that pinholing can arise due to arcing between the electrodes, particularly when treating very thin films. A process claimed<sup>9</sup> to eliminate

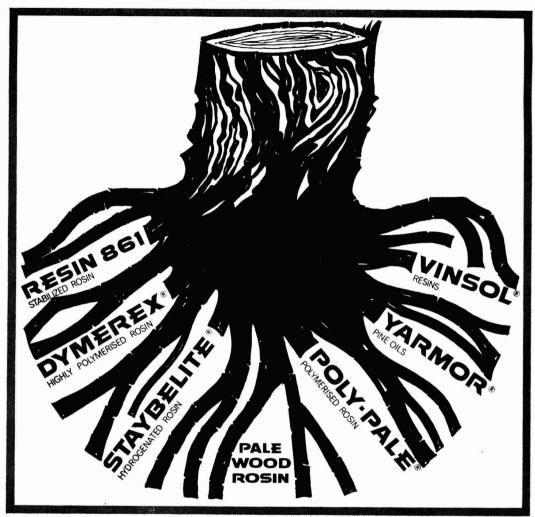
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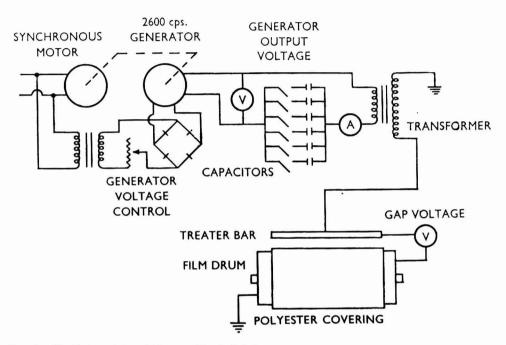
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The electrical process which is most widely used in practice is that based on the corona discharge principle and known as "D" treatment, the circuit diagram for which is shown in Fig. 2. It is usual to treat films by this method



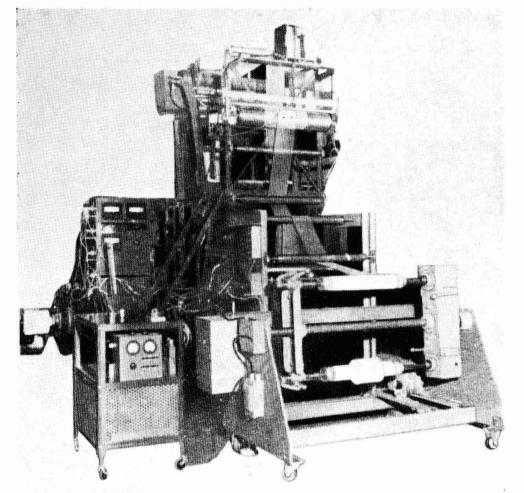
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Fig. 2. The circuit diagram of the "D" treatment machine

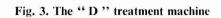
in-line, i.e. immediately after extrusion, in order to minimise the effects of additives contained in the film. The fixed electrode is usually in the form of a metal blade held in an insulated hinged holder as shown in Figs. 3 and 4, and the distance between this and the insulated roller, which is the movable electrode, is of the order of  $\frac{1}{16}$  in.

The effects of changes in the major process variables on the degree of treatment by this method were studied by Wechsberg and Webber<sup>10</sup>. It was found that the degree of treatment was roughly proportional to the voltage applied across the electrode gap up to a limiting voltage above which there was a tendency for the adhesive properties to decrease. This is shown in Fig. 5. A similar tendency to reduction in adhesive properties was also found if film speed was increased, i.e. the time of treatment was decreased (Fig. 6).

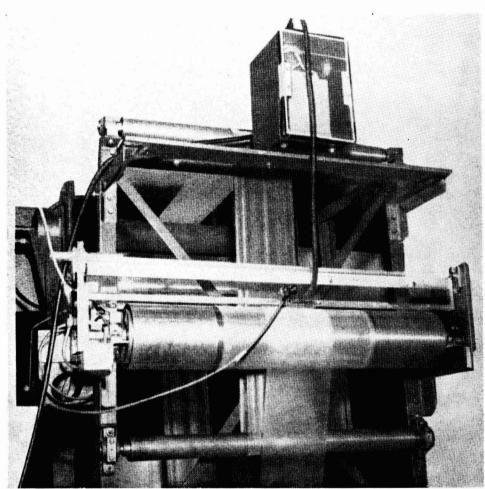
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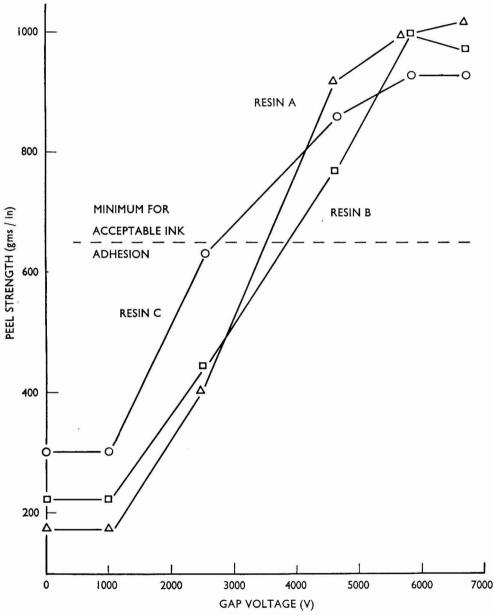


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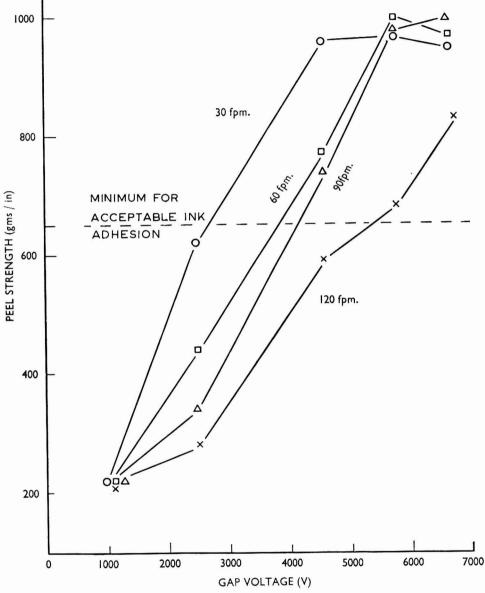
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Fig. 4. A close up view of the electrodes

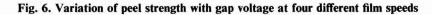




### Fig. 5. Variation of peel strength with gap voltage for three different resins



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Intensification of treatment either by voltage increase or the use of multiple treaters can permit decreased time of treatment provided that the voltage increase does not reach the limiting values where adhesion decrease takes place. A further point to be noted with this method of treatment is that marked reduction in heat sealability occurs with increasing intensification of treatment. This is shown in Table 1.

Table 1

nulled voltage		Heat seal strength (g/in)		
pplied voltage —	120 C	150 C	180 C	
0	1,770	1,820	1,950	
1,000	1,640	1,730	1,770	
2,500	1,550	1,550	1,680	
4,000	1,370	1,370	1,500	
5,800	1,180	1,320	1,370	
6,700	1,180	1,140	1,180	

The presence of lubricants in the polymer necessitates an increase in intensity of the pretreatment by this method in order to obtain adequate adhesive properties as is shown in Table 2.

Applied voltage		Peel strength (g/in)	
Applied voltage	No lubricant	0.04% oleamide	0.04% ethylene bis-stearamide
0	180	180	180
1,000	180	220	220
2,500	400	220	250
4,500	900	380	630
5,750	1,000	430	770
6,750	1,020	950	740

Table 2

Minimum peel strength for acceptable ink adhesion is 650 g/in.

The effectiveness of slip agents is decreased by delay in treatment following extrusion as may be seen for the figures for coefficient of friction in Table 3 where the additive was 0.04 per cent oleamide.

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Applied voltage	Treated in-line	Treated 24 hours after extrusion
0	0.29	0.29
1,000	0.29	0.28
4,600	0.22	0.31
6,700	0.18	0.39

Table 3

Base resin without additive has a coefficient of friction greater than 1.

The effectiveness of antistatic additives is generally markedly improved by this method of treatment and this is a very important advantage when printing is envisaged.

A more detailed study of the effects of corona discharge on polyethylene was presented to the AIEE summer general meeting in 1958<sup>11</sup>, but this paper was mainly concerned with the deleterious effects of corona discharge and is interesting from the present point of view only in showing the dangers inherent in this method on overtreatment. Discussions on the electron bombardment and corona processes have also been published by Bloyer<sup>12</sup> and by von der Heide and Wilson<sup>13</sup>.

An electrostatic technique has also been reported<sup>14</sup>, in which the polythene film is led, under pressure, over a friction producing body, thus producing an electrostatic charge. Some distance from this point the opposite side of the film is discharged through an earthed electrode.

The final method of pretreatment which should be mentioned is that in which heat is used. Several different techniques have been claimed in which the heat has been applied electrically by a heating element, by hot air or by oven heating<sup>15</sup>, but that which has gained most popularity is the application of a gas flame<sup>16</sup>. This flame treatment consists basically of the application to the surface of the polymer of a hot oxidising gas flame for a time of 1-3 seconds. The equipment is comparatively simple and can usually be fully automated. It is important to remember that in order to achieve adequate pretreatment, and hence satisfactory adhesion for surface coatings, it is necessary to control the process very closely, particularly in respect of distance of the moulding from the burner, time of exposure to the flame, type of flame and composition, i.e. air/gas ratio of the flame. Either town gas or bottled gas may be used and it should be fed with compressed air in order to produce an oxidising flame hot enough to allow for adequate treatment in as short a time as possible. A normal injection of atmospheric pressure air as in the Bunsen burner is generally inadequate. The flame should be quiet and steady and preferably produced by a burner having a multiple line of small flame holes rather than a single flame of low intensity<sup>17</sup>. A survey of the method and comparison with the electronic methods has been given recently by Buchel<sup>18</sup>. The process is said to give a treated surface which will still show satisfactory adhesion properties up to at least six months, so that it is not necessary to decorate immediately after treatment, although for several obvious reasons this is desirable

As with the electronic process, overtreatment tends to reduce the adhesive properties of the surface, but it has been reported that an overtreated surface can be recovered to a satisfactory state by a further light treatment. A further similarity with the electronic process is the antagonism of adhesive properties with heat sealability. In both cases, therefore, it cannot be stressed too highly that where a satisfactory compromise of the two properties has been achieved with any particular grade of polymer the conditions found should be maintained very closely. Further, it cannot be assumed that the conditions found with one grade of polymer will necessarily be the same as those applicable to another grade of the same polymer even from the same source of supply, and experimental work will have to be carried out to determine the optimum conditions of treatment when any change of polymer identity is envisaged. So far as change of polymer type is concerned, a rough guide is that the ratio of treatment times in the flaming method for low density polyethylene, high density polyethylene and polypropylene is 1: 2: 3.

In the light of the necessity for the experimental approach to optimum treatment conditions, it is equally necessary to apply test methods which will indicate a satisfactory surface condition of the treated polymer. Chapman<sup>19</sup> described a test method using a printing ink as an adhesive between two treated surfaces. Boxler et al.<sup>17</sup> described methods of improving the reproducibility of this test, but it has not gained widespread acceptance. The two tests in common use today are neither of them quantitative measures of treatment efficiency, although modifications designed to that end have been proposed to both. Both tests are extremely simple in performance and, therefore, admirably suited for use in a production department by unskilled operators. The first of these, which was foreshadowed in the work described by Wechsberg and Webber<sup>10</sup>, where the peel strength of a pressure sensitive tape applied to the treated surface under standard conditions was measured and correlated with ink adhesion properties, involves the application to the dried printed pretreated surface of a piece of pressure sensitive tape and the subsequent rapid removal of the tape. Should the treatment be inadequate the tape removes the ink film. This simple qualitative method can be applied to film, blow mouldings or extrusions, but recently a British Standard<sup>20</sup> has been introduced which uses a double surfaced adhesive tape which is applied to a metal backing plate and to which is applied by its untreated surface a sample of the film to be tested. A length of standard pressure sensitive tape is then applied to the treated surface of the film under standardised conditions and the assembly mounted in the jaws of a suitable tensometer which pulls off the standard tape at a fixed rate of 1 ft per minute. The load required for this operation is recorded as a quantitative measure of the level of treatment.

The other simple assessment method is to dip the cooled treated surface into a vessel containing clean cold water when, if the treatment has been adequate, the surface will be uniformly wetted and the water film will persist unbroken on the surface for up to 30 seconds on removal from the vessel. If the surface has been under- or over-treated the water film will form globules immediately on removal from the water vessel. A similar test has been suggested using a solution of a dyestuff, e.g. Victoria Pure Blue Lake 0.4 per cent w/v in nitroethane<sup>21</sup>. This has the advantage that the tested article is permanently stained by the dyestuff and can be filed as a reference standard for correlation with treatment conditions. More quantitative tests based on the fact that the contact angle between the surface and a drop of water is reduced by pretreatment have been put forward. Allan<sup>22</sup> showed that the contact angle between distilled water and polyethylene was related to ink adhesion and McLaughlin<sup>23</sup> developed this basic principle into a practical test by measuring, by means of an inclining platform tester, the angle at which a polyethylene surface just fails to hold a

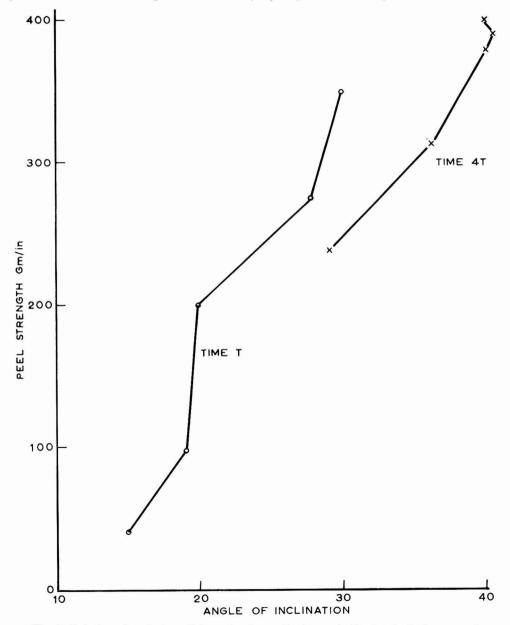


Fig. 7. Relation of peel strength to water drop adhesion on inclined polyethylene sample

water drop. Correlation of the angle of inclination with ink adhesive properties of the treated surface was made by comparing results obtained with the Chapman peel test<sup>19</sup> and these are shown in Fig. 7. These curves also illustrate the effect of time and extent of treatment on the adhesion properties, for each point represents a different gap voltage.

It may be thought that considerable space has been devoted to this subject of pretreatment, but it must be emphasised that it is an extremely important prerequisite for satisfactory decoration particularly in the case of the polyolefines.

Before starting on any decorating process, in addition to the pretreatment already described, it is necessary to take cognisance of the fact that all plastics are electrical insulators, with the result that they inevitably pick up static electricity during fabrication. This means that the plastic surface to be decorated, being charged, attracts any dust or dirt present in the atmosphere and no great imagination is required to understand the way in which this can impair the final appearance of decoration particularly the application of ink films. Immediately before any decorative process, therefore, some device to discharge the plastic must be employed. In some cases this is done by the incorporation of chemical agents, e.g. long chain amine salts, into the plastic before fabrication. In other cases a surface coating of an agent designed to produce a leakage path for the charge is applied. Both these methods suffer from the disadvantage that they affect the surface of the plastic which is to be decorated and might impair the application of the decoration. A more satisfactory method of removing the charge is to use an ionised air flow over the surface which neutralises it. This airflow must be applied up to the time of application of the decoration to prevent a reaccumulation of static which can occur very rapidly.

### Printing

Three principal methods are used for decorating plastics by printing processes; flexographic or photogravure being largely used for continuous film printing, silk screening being used for bottles, particularly those of body shapes other than cylindrical, and off-set letterpress, sometimes known as "letterset" or "dry offset" due to the fact that no water is required as the printing plate used is in relief.

It is not proposed to describe these various processes in detail as this has been covered adequately in a series of recently published papers<sup>24, 25, 26</sup>. In all these cases, however, it has to be remembered that the surfaces to which the printing ink has to adhere are substantially non-absorptive and, in the case of the thermoplastics which are the principal plastics being considered here, the distortion temperatures are comparatively low so that stoving types of ink such as those normally used for metal decoration cannot be used. Furthermore, as the process is normally required to be carried out by a mass production method on a continuous basis and not in batches, in order to restrict the plant to reasonable dimensions it is necessary to restrict the drying time of the ink quite severely. It could be said that, at the best, a forced air drying technique is suitable although occasionally, particularly with silk screen printing, air drying inks are used. Conveyor type ovens are usually used through which the printed objects pass on a series of pegs attached to a continuous chain. Typical stoving conditions used in such equipment are 10-20 minutes at 65-70°C in the case of low density polyethylene and 5-15 minutes at the same temperatures with polystyrene. A further restriction is applicable where the printed objects are designed for use in the packaging of foodstuffs as both media and pigments or dyestuffs must be selected such that there will be no contamination of the foodstuff to be packaged. This is particularly important in relation to the use of flexographic inks on thin packaging films where the ink ingredients may well permeate the thin plastics films used. There must also be no residual odour from the inks used for printing packages designed to contain cosmetics, pharmaceuticals or food products.

In the case of off-set printing it is necessary to formulate an ink which will neither dry on the rollers nor be absorbed by the transfer rollers or blanket in order to obtain adequate printing definition and yet at the same time be capable of the extremely fast drying properties noted a little earlier. Satisfactory inks have been produced using media drying by oxidation and reduced for application with high boiling slow evaporating solvents. Two-pack inks have also been applied satisfactorily using both epoxy and polyurethane bases, but these have been used more in silk screen printing than in off-set, as with the off-set roller set-up it is necessary to use rollers covered with a synthetic rubber of the butyl type to prevent the absorption of the medium which is liable to occur with such of the more common coverings as natural rubber, acrylonitrile or chloroprene rubbers. Flame drying of some of these types of ink has also been satisfactorily used, a new technique which has materially assisted in the solution of the problems of obtaining the necessary rapid drying associated with adequate application properties.

One type of ink which has been put forward for off-set printing and which is claimed to be applicable to unpretreated polyethylene and to give good adhesion is of interest. This is a thermal diffusion type of ink. Printing properties on a dry off-set machine are certainly very good and there is no doubt that, even with the restricted drying conditions permissible with low density polyethylene, the print has good adhesion to the base plastic even to the extent of satisfying the adhesive tape test. Unfortunately the disadvantage which, so far at least, has restricted the acceptance of this type of ink is that which makes it dry so satisfactorily, namely the diffusion properties, for whilst the print obtained initially is extremely clear, after drying the edges are quite blurred due to the diffusion into the plastic. In consequence it is at the moment quite impossible to print letterpress, particularly the fine type so often required on cosmetic and pharmaceutical packages, so that a legible result is obtained. It might be applicable where large rather diffuse designs are required, but these are so few and far between that at the present stage of development this type of ink is only a curiosity.

### Lacquering

Whilst many of the inks used today on plastics will give a high gloss film, it is usually necessary to consider the application of an overprint varnish in order to produce a uniform finish to the container and to protect the ink film

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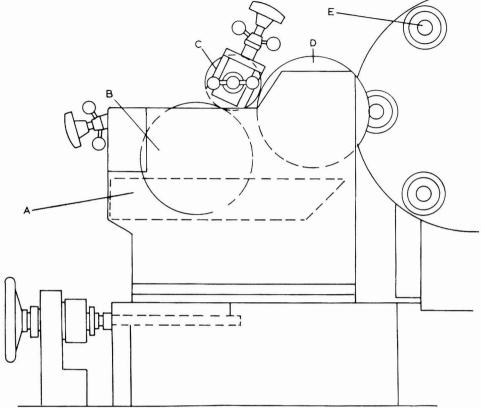
from damage by scuffing or abrasion. In the case of low density polyethylene in particular, there is, however, a further reason because one of the unfortunate intrinsic properties of this particular plastic is the tendency for certain products to permeate through the walls of the container without any obvious chemical or physical deterioration of the polymer. Amongst these danger materials are hydrocarbons and other organic solvents such as might be incorporated into adhesives, etc.; essential oils and perfumes as contained in cosmetics. One has only to smell a polyethylene tube of a hand cream, for example, to become aware of this fact. Permeation of these ingredients from the packaged product can result ultimately in a severe reduction of the contents or even a change in the properties of the product. Further, certain gases also will permeate through polyethylene and here one should mention particularly oxygen. Again, permeation of oxygen from the air can cause deterioration of the product inside the container. Table 4 shows some of the figures published for permeability of low density polyethylene compared with other polyolefines used for packaging.

		cc/sq cm	Permeability at 20°C s/sec/cm Hg/cm thickne	ss of film
		Low density Polyethylene	High density Polyethylene	Polypropylene
Oxygen		0.28×10 <sup>-9</sup>	1.1 ×10 <sup>-10</sup>	0.10×10 <sup>-9</sup>
Carbon dioxide		$1.22 imes10^{-9}$	4.7 ×10 <sup>-10</sup>	0.30×10 <sup>-9</sup>
Hydrogen		0.8 ×10 <sup>-9</sup>	3.5 ×10 <sup>-10</sup>	0.64×10 <sup>-10</sup>
Nitrogen		0.1 × 10 <sup>-9</sup>	0.36×10 <sup>-10</sup>	0.05×10 <sup>-9</sup>
Methane		$0.34  imes 10^{-9}$	1.1 ×10 <sup>-10</sup>	$0.05 imes10^{-9}$
Water vapour		1.26×10 <sup>-9</sup>	24 ×10 <sup>-10</sup>	0.05×10 <sup>-9</sup>

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It is necessary, therefore, to apply an overall coating of a suitable barrier lacquer to reduce this permeation problem. It has to be admitted that to date no barrier coating has been found which will completely eliminate the problem, but a great deal of work has been carried out, and various types of surface coatings have been put forward ranging from solutions of other lacquer-type polymers, e.g. vinyl co-polymers and polyvinylidene chloride, to conversion coatings of the alkyd, alkyd/amine and epoxy ester types, to the two-pack epoxy/amine or epoxy/polyamide types.

Amongst the properties necessary in a commercially satisfactory barrier lacquer, in addition to the intrinsic barrier properties, are (a) application properties, i.e. viscosity, solids content, viscosity stability under working conditions, (b) film properties, i.e. flexibility, gloss, initial good colour, colour retention, flow. In many cases the application is by roller coater and a typical example of such a machine is shown in Figs. 8 and 9. The lacquer is placed in the tray A into which dips a steel pick-up roller B. The lacquer film thus picked up is doctored off by an adjustable doctor/transfer roller C and the doctored film is then transferred to a rubber blanket D which in turn comes into contact with the object to be coated which is carried on a rotating mandrel

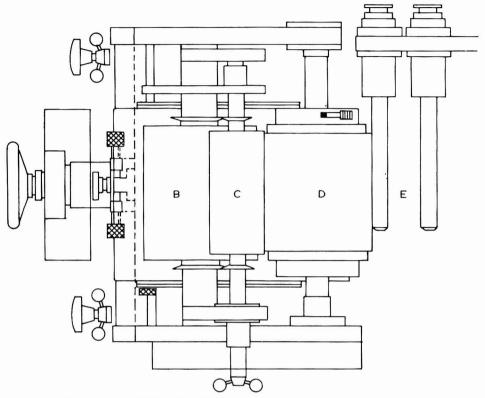


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E. In this way a film of constant wet thickness can be applied to the object. This description of the operation of the machine also explains the importance of the consistency in application properties of the lacquer, particularly the viscosity stability. The continuous rotation of the pick-up roller in the lacquer causes a considerable degree of agitation of the lacquer resulting in aeration which can cause marked variation of viscosity of the lacquer over a continuous period of operation by both solvent evaporation and the chemical effect on the ingredients of the lacquer.

As the film thickness which can be applied is limited by the necessity for the film to flow out to an even thickness on the coated object without running or tearing, it is obvious that the solid content of the lacquer has to be fairly high and this generally rules out such film forming polymers as vinyl and vinylidene resins where the molecular weight necessary to give good film properties invariably places a restriction on the solid content which can be attained with a reasonable application viscosity. Such polymers also usually require powerful solvents in order to obtain stable solutions and this factor can have an adverse effect on the films of printing ink to which the lacquer is applied. The use of two-pack lacquers has already been mentioned, but

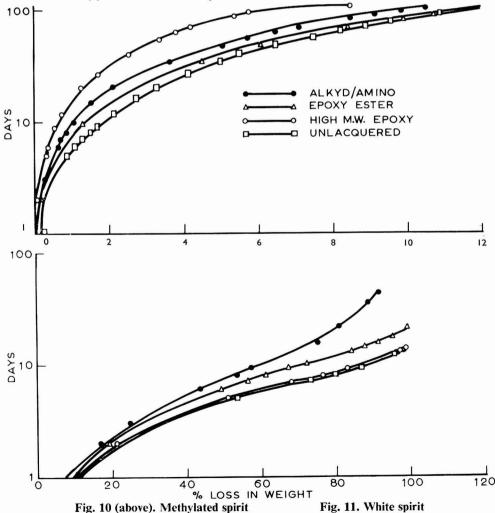


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Fig. 9. View from above of the barrier lacquer application machine

these have the great disadvantage of comparatively short (3-4 hours) pot life if the films produced are to have satisfactory drying properties; in fact it has been reported that it is necessary when using this type of lacquer to throw away the contents of the lacquer tray at least twice per day. This is obviously a very uneconomical procedure, particularly when it is remembered that the initial cost of the lacquer is comparatively high. The necessity for high solid content in the lacquer is also to some extent governed by the gloss requirements of the finished film which should be of a high standard in view of the importance of this property to the final appearance of the coated object which, in many cases, particularly in the field of cosmetic packages, can be a vital factor in successful marketing. A minimum figure for gloss as determined by a 45° standard gloss meter is 60-65°. Initial colour is also of extreme importance in this respect when, as so many of the packages are based on white plastics and the colours of the inks are frequently very carefully chosen to produce artistic effects, a lacquer giving a slightly yellow finish would be completely unacceptable. Colour retention of the film is of importance for the same reasons, but the major causes of discolouration in this field are usually the products which are being packed and in particular the ingredients of those products which can permeate the plastic and attack the lacquer from beneath. Many otherwise excellent barrier lacquers have proved to be unacceptable because of the yellowing caused by attack of, e.g. perfumes, in the product. This same problem is frequently encountered also in the choice of inks where, although the ink may appear to be resistant to smearing by the product on the outside surface, extended storage of the package with the product inside will cause discolouration. As examples of these effects, certain polyethylene tubes known to be overlacquered with an epoxy two-pack lacquer showed decided yellowness after storage when containing various types of hand cream, and a polyethylene tube decorated with a metallic ink and overlacquered with an alkyd/amino type of barrier lacquer, whilst showing no discolouration of the lacquer film, showed definite greening of the metallic ink when stored containing a certain type of shaving cream.

As was stated before, no lacquer has yet been produced which has succeeded in completely eliminating permeation through low density polyethylene, but results obtained on long-term storage of various products in tubes coated with various types of barrier lacquers are shown in Figs. 10 and 11.



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The tests shown in these figures were carried out by filling a known quantity of the product into a 1 in diameter polyethylene tube and weighing it at intervals during storage in a temperature controlled oven. The rapid permeation of hydrocarbons is shown by the complete loss of white spirit, in 13 days in the case of an unlacquered tube. Lacquering with an alkyd/amino type lacquer extends this period to something over 60 days. An epoxy ester lacquer gives extension to 21 days, but a high molecular weight epoxy resin lacquer allows complete permeation in much the same time as the unlacquered tube. Similar tests have been carried out using ethyl acetate, carbon tetrachloride, benzene, xylene, acetone, water and methyl salicylate. The test procedure was also carried out at room temperature when the permeation rate was considerably lower. Results obtained in these tests are shown in Tables 5 and 6.

### Table 5

Room temperature permeability Per cent loss of weight of product

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Solvent	Alkyd/amino lacquer	Epoxy ester lacquer	High MW epoxy lacquer	Unlacquered
Ethyl acetate .	. 32.2	33.4	35.01	39.39
Acetone	. –		43.4 (48 days)	15.24
Carbon tetrachloride	100 (28 days)	100 (28 days)	100 (27 days)	100 (27 days)
Methylated spirit .	. 1.36	2.74	0.84	1.10
White spirit	. 49.92	62.0	90.1	100 (76 days)
Benzene	. 100 (20 days)	100 (20 days)	100 (20 days)	100 (20 days)
Xylene	. 100 (20 days)	100 (20 days)	100 (20 days)	100 (20 days)
Methyl salicylate .	7.5	8.1	8.28	11.24
Water	0.29	0.25	0.25	0.25

One thing which seems to arise from these figures is that it may prove necessary to choose a barrier lacquer in relation to the product being packed in order to obtain the optimum in permeability reduction. This view is supported by the figures for weight loss shown in Tables 7, 8, 9 and 10, which are for a series of commercial products such as might be packed in polyethylene tubes.

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Solvent	Alkyd/amino lacquer	Epoxy ester lacquer	High MW epoxy lacquer	Unlacquered
Ethyl acetate	52	50	48	44
Acetone	95.4% (104 days)	98.6% (104 days)	104	5
Carbon tetrachloride	10	10	9	9
Methylated spirit	10.6% (105 days)	12.2% (105 days)	8.98% (105 days)	
White spirit	63	21	14	13
Benzene	9	9	9	9
Xylene	9	9	9	. 9
Methyl salicylate	56.7% (104 days)	64.3% (104 days)	66.9% (104 days)	87.9% (104 days)
Water	3.0% (105 days)	2.35% (105 days)	2.26% (104 days)	3.68% (104 days)

Table 6 Permeability at 40°C Number of days for complete loss of product

Table 7
Permeability from hand creams
A. Per cent weight loss of product at room temperature

		Α			в			С		1	D			Е	
Barrier							Day	s' stora	age						
lacquer	50	100	150	50	100	150	50	100	150	50	100	150	50	100	150
Two-pack epoxy	.18	.36	.52	.17	.38	.53	.18	.34	.47	.17	.30	.41	.12	.24	.34
Alkyd amino	.25	.45	.63	.20	.41	.57	.24	.40	.58	.22	.39	.50	.16	.32	.45
Epoxy ester amino	.21	.40	.57	.20	.41	.58	_	_	_	_	_	_	_		_
Epoxy ester	.19	.38	.53	.18	.39	.54	_				_	_	.13	.25	.36
No lacquer	_		_	-	-	-	-	-		.21	.37	.50	.16	.30	.42

		Α			в			С			D			Е	
Barrier		Days' storage													
lacquer	50	100	150	50	100	150	50	100	150	50	100	150	50	100	150
Two-pack epoxy	1.04	1.96	1.58	1.04	2.02	3.08	1.44			1.35	2.51	3.70	1.21		-
Alkyd amino	1.04	2.87	3.06	1.09	2.87	3.11	1.55	2.87	4.18	1.40	2.62	3.87	1.39	2.64	-
Epoxy ester amino	1.00	1.86	3.04	_	_	_		_	_			_	_	_	-
Epoxy ester	.99	2.03	3.10	1.01	2.00	3.07	-			-		-	1.19	2.29	3.44
No lacquer	-	-	-	-	_	-	-	_	-	1.35	2.51	3.70	1.04	1.96	-

B. Per cent weight loss of product at  $40^{\circ}C$ 

	Α			В		С		
			Da	iys' stora	ige			
50	100	150	50	100	150	50	100	150
.06	.11	.15	.05	.09	.12	.23	.50	.76
.08	.14	.18	.06	.11	.14	.86	1.25	1.49
	_	-		_	-	.88	1.28	1.56
	_					.39	.65	.91
.07	.12	.15	.05	.09	.11	-		
	.06 .08	A 50 100 .06 .11 .08 .14 	A 50 100 150 .06 .11 .15 .08 .14 .18 	A Date of the second se	Days' stora           50         100         150         50         100           .06         .11         .15         .05         .09           .08         .14         .18         .06         .11	A         B           Days' storage           50         100         150         50         100         150           .06         .11         .15         .05         .09         .12           .08         .14         .18         .06         .11         .14	A         B           Days' storage           50         100         150         50         100         150         50           .06         .11         .15         .05         .09         .12         .23           .08         .14         .18         .06         .11         .14         .86              -         .39	A         B         C           Days' storage           50         100         150         50         100         150         50         100           .06         .11         .15         .05         .09         .12         .23         .50           .08         .14         .18         .06         .11         .14         .86         1.25               .88         1.28              .39         .65

Table 8Permeability from toothpastesA. Per cent weight loss of product at room temperature

### B. Per cent weight loss at 40°C

	А			В			С			
Barrier lacquer	Days' storage									
	50	100	150	50	100	150	50	100	150	
Two-pack epoxy	.50	.97	1.48	.44	.83	1.28				
Alkyd amino	.53	.96	1.42	.49	.92	1.42				
No lacquer	.44	.80	1.17	.36	.82	1.17		-	—	

Table 9Permeability from shaving creamsA. Per cent weight loss of product at room temperature

	А		H	3	С				
Barrier lacquer	Days' storage								
-	50	100	50	100	50	100			
Two-pack epoxy	.28	.38	.11	.25	.08	.16			
Alkyd amino	.31	.42	.15	.26	.11	.14			
Epoxy ester amino	.29	.39	.23	.43	_				
Epoxy ester			.14	.28					
High MW epoxy	.32	.46	.18	.34	.13	.27			
No lacquer	.28	.39							

### DECORATION OF POLYOLEFINES

		· · · · · · · · · · · · · · · · · · ·			a second s		
	ŀ	4	E	3	С		
Barrier lacquer			Days' s	storage			
	50	100	50	100	50	100	
Two-pack epoxy	1.44	2.95	1.50	2.38	.93	1.80	
Alkyd amino	1.01	1.13	1.20	2.18	-		
Epoxy ester amino	-		1.71	3.14		-	
Epoxy ester			1.30	2.53	_	—	
High MW epoxy	_		1.57	3.00	—	—	

### B. Per cent weight loss of product at $40^{\circ}C$

	Table 10
	Permeability from hair shampoos
A.	Per cent weight loss of product at room temperature

		А			В			С		
Barrier	Days' storage									
lacquer	50	100	150	50	100	150	50	100	150	
Two-Pack epoxy	.15	.28	.41	.07	.18	.39	_	_		
Alkyd amino				.18	.29	.39	.21	.36	.48	
Epoxy ester				.09	.17	.24		_		
No lacquer		_		.17	.28	.36		-	_	

B. Per cent weight loss of product at $40^{\circ}C$	
	-

		Α			В			С		
Barrier lacquer	Days' storage									
	50	100	150	50	100	150	50	100	150	
Two-pack epoxy	1.04	1.94	2.70	.67	1.84	2.98		_	_	
Alkyd amino		-		1.22	2.11	2.98	5.90	-	_	
Epoxy ester				.87	1.83	2.71			_	

### Hot decorating processes

There are other decorating processes which can be applied to plastics without pretreatment, because the decoration is applied by processes which soften the surface of the plastic during the actual decoration. Probably the most widely used of these processes is that known as hot foil blocking. This has the advantage of being a dry process in which the decoration is complete and adherent to the plastic without subsequent drying or overlacquering. It consists in pressing, by means of a controlled temperature die of the required design, a foil on to the surface of the plastic to be decorated. The foil consists of four main parts : (a) a carrier, usually a polyester, acetate or cellulose film, (b) a release layer, which is a waxy substance under the carrier and which melts at a predetermined temperature under the hot die, permitting the coating immediately beneath the die to separate from the carrier and adhere to the plastic surface, (c) the coating, which may be a metallic foil or a pigment layer, and (d) a sizing layer, which is a thermosetting adhesive which bonds the print to the surface. Descriptions of the process have been given by Bingham<sup>27</sup> and Harben<sup>28</sup>. As has been noted this process has the advantage of being completely dry and complete in itself, but it is rather slow in comparison with the off-set printing process (which can operate at up to 5,000 prints per hour), being limited to about 2,000-2,500 prints per hour. Another disadvantage is that it is only possible to apply one colour per pass, so that if multicolour decoration is required it is necessary to have multiple application. A still further disadvantage in relation to the decoration of cylindrical containers is that the extent of coverage of the surface by the print in one application is a maximum of approximately 60 per cent of the area, so that if print is required on both sides of the container, double application is necessary or two machines in tandem have to be used with some means of turning the container between applications. A typical machine designed for printing tubes is shown in Fig. 12. Recent developments in machines, however, are tending to remove some of these disadvantages, and there is now on the market a fully automatic machine which is claimed to print at 7,000 per hour and to cover the complete surface of a cylindrical container in one application, but it is still only possible to apply one colour at a time. The main advantage of this process is the possibility of the application of metallic decoration with the brilliant finish associated with a continuous metallic coating as opposed to metallic ink. The opacity of the pigmented foils is also generally superior to that which can be achieved with normal printing processes with the possible exception of the silk screen process. In the application of this process care has to be taken to support the object to be printed fairly rigidly in order to ensure (a) no distortion of the plastic and (b) an even, clear and sharp print. It is also frequently very necessary, particularly when using this process in combination with a barrier lacquered plastic, when the foil blocking is normally carried out after the lacquering, to select the foil extremely carefully in order to obtain satisfactory adhesion of the print, this adhesion being tested in the same way as normal printing, i.e. by the adhesive tape test.

Another process which has been developed recently, and which has found fairly wide acceptance in the USA, is that due to the Dennison Company and called "Therimage." Basically this is a transfer labelling process in which the design to be applied to the container is first gravure printed on to the heat release coating of a patented label paper. This transfer paper then passes between the rotating preheated container and a heated platen which presses the transfer on to the container. The process is completed by passing the transferred container through an oven where a glossy finish is imparted to

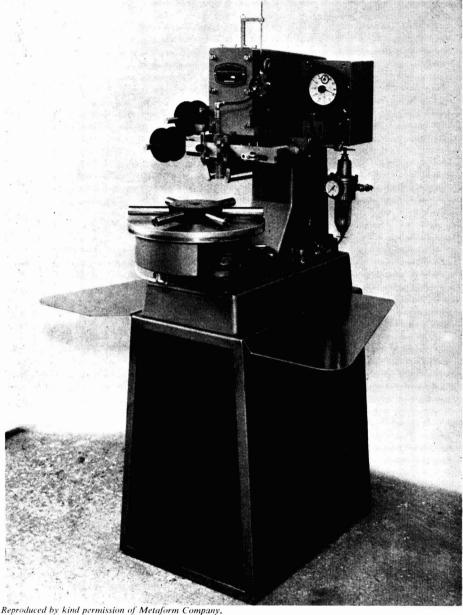
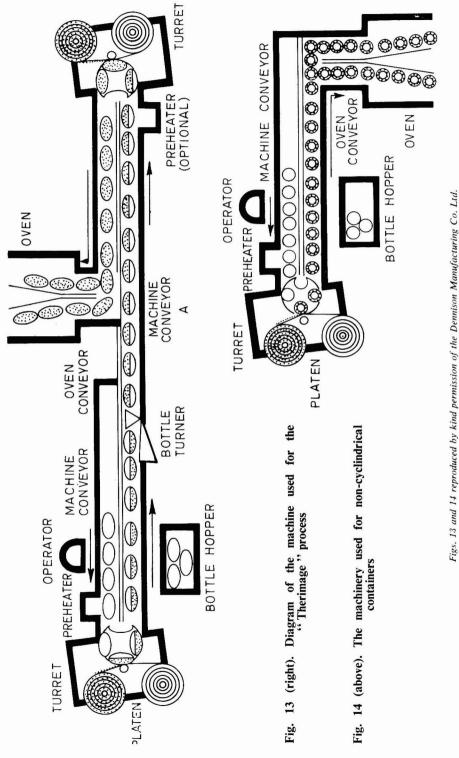


Fig. 12. The tube printing machine

the print. On a fully automatic line as shown in Fig. 13, for which the process is claimed to be ideally suited, printing speeds of up to 100 containers per minute have been achieved. The advantage of this process over the previously described hot foil stamping is that multi-colour prints can be applied completely round a cylindrical container in one operation, although where the container is non-cylindrical it may be necessary to use two platens in tandem (Fig. 14).



The process is said to give equally good results on all the polyolefines. polystyrene, polyvinyl chloride and urea/formaldehyde surfaces. In the case of polystyrene and polyvinyl chloride no post-heating is necessary to produce a glossy well adherent print. The process has been described and discussed in more detail by O'Connell<sup>29</sup>.

Other methods of decoration which are widely applied but which are outside the scope of this paper are electroplating and vacuum metallising, both of which are restricted generally to the more rigid types of plastic.

### Acknowledgments

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### Discussion

MR. E. L. FRENCH said that it had been shown that the bond strength of printing ink or paint increased with the applied voltage up to a maximum value. It was known that an electrical discharge in air produced ozone. Was the limiting value of adhesion due to the rate at which ozone was liberated (one would expect this to reach a maximum value), or was the physical change produced in the surface of the plastic due to electrical stress ?

Considering the lacquers which are used for coating plastic materials, had it been established whether the molecular weight of the resin used in the lacquer had any bearing on the bond strength of the coating to the plastic substrate ?

MR. C. R. PYE considered that the limitation was due to the ozone, or rather the resulting oxidation of the surface and not to the electrical stress. He had no personal experience of this particular work, but the evidence tended towards the oxidation theory.

Mr. Pye said that he had not done any work in which the specific effect of molecular weight was studied.

MR. T. R. BULLETT asked whether the lecturer had an explanation for the fall in adhesion properties claimed when electrical pretreatment was prolonged? Possibly such treatment would produce a degraded and therefore mechanically weak surface layer, but this would not affect the water-wetting qualities which were also said to deteriorate.

MR. Pye had no explanation to offer on that particular point; as was shown on the graph, the angle of inclination increased steadily.

MR. A. R. H. TAWN said that it had been stated that a disadvantage of the wet oxidation pretreatment was that the surface had subsequently to be washed and dried. Had any progress been made in the use of water-based inks which, by analogy with metal priming, should be applicable to wet surfaces ?

The action of flame, chemical or electrical pretreatments could be ascribed to surface oxidation. This did not seem to be consistent with the light re-treatment of an over-treated surface described by the speaker. Again, had not pretreatment by purely mechanical stressing been suggested? Was it not possible that oxidation was not the sole effect, but that other factors, such as re-orientation of surface molecules or even the creation of "living" polymer fragments in the surface, might play a part in surface activation?

MR. PyE said that he knew of no use of water-based inks for this purpose.

Concerning the pretreatment process, he shared the doubts expressed by Mr. Tawn. It had generally been accepted, and, in fact, various papers had been published commenting that oxidation was the prime result of these pretreatment processes. He had never been fully in support of this viewpoint and felt that while some oxidation was involved as a result of the presence of ozone, there was also some re-orientation of the molecules on the surface of the polymer. On the other hand, the polymers were basically non-polar and most of the inks were polar, so that there was a basic incompatibility and for the pretreatment process to be successful some chemical change must be involved.

MR. J. S. MARSH said that Mr. Pye had mentioned that the flame treatment gave six months' life under ideal conditions. Was a similar degree of permanence obtained by other pretreatments? What were the possible mechanisms of loss of the effects of pretreatment?

MR. PYE said that some re-orientation probably occurred and it was difficult to establish definite times for stability. It had been reported that corona discharge had a long length of life and figures of 4-6 months had been quoted. The oxidation-only theory could not account for a lack of stability, but there was the observed fact that the longer a plastic was left before decoration the lower the bond strength became.

MR. MARSH said that in many cases the bond strength decreased suddenly rather than gradually.

MR. PYE agreed that this could occur.

MR. BULLETT asked whether the Bikerman theory of diffusion to the surface of low molecular weight components could explain the falling off in adhesive properties after pretreatment?

MR. Pye agreed with this possibility. A plastic was not a single entity, but a mixture of various molecular species. If there were lubricants in the polymer they also might come to the surface at a later stage and be the cause of the reduction in adhesion.

MR. P. G. RICHARDSON asked, with reference to flame and other pretreatments, whether the significant effect of such primarily oxidative processes could be to remove compounds such as lubricants and slip aids by oxidative degradation, and that they were subsequently replaced by migration to the surface from the body of the plastic?

MR. PyE said that could well be so.

MR. P. J. QUORN asked which would become the more popular of the metal coating techniques, dry foil coating or vacuum metallising?

MR. PYE said that in the case of rigid plastics probably vacuum metallising would maintain its present position, but it had not been found possible to vacuum metallise polymer film or the softer thermoplastics, such as low density polyethylene, at a commercial rate.

MR. QUORN asked further whether vacuum metallising was more restricting for mass production ?

MR. PYE said this was not necessarily so.

MR. G. C. HURST commented that foil stamping, which might involve metallic or coloured markings, was far more practicable if the decoration involved any design including fine detail. Vacuum metallising would be preferred for overall marking only.

MR. V. F. JENKINS suggested that containers might have the barrier coating applied under the ink rather than above it.

MR. PYE said that this had been done and there were certain barrier lacquers of the epoxy ester type which, applied under the ink, were claimed to bleed through the ink film on thermal drying and produce an overall protective glossy finish. He had had some experience of this process, but without any significant success. He believed that a claim had been made recently for the use of a 7-ply laminate to produce tubes by the spiral winding process. As one of the plies was a metal foil this should undoubtedly reduce permeation, but as he had not seen any of these tubes he could not comment on their efficiency as squeeze tubes, and he thought that the production of an integral shoulder and nozzle might cause some problems. Another process which had been suggested was to use two extruders and extrude two materials through one die so that the barrier layer was welded to the main tube plastic in fabrication. So far as he knew this process, developed in France, had not yet attained any commercial significance. Suitable selection of the plastics for this process could be expected to produce tubes with very low permeability characteristics.

MR. J. F. BARTON asked, concerning the barrier lacquer, whether it would not be better to apply this internally, thus protecting the printing from outward permeation?

MR. PYE said that this was an interesting suggestion. He believed that at the present time there was only one company in the world which regularly produced internal coatings on polyethylene tubes. The difficulties were mechanical, in putting the coating on the tube. With metal tubes, however, the situation was quite different due to their greater rigidity, and the coatings could be cured at temperatures up to 300°C with aluminium tubes, while with polyethylene it was not possible to exceed 70-75°C without distortion. At the same time it had to be remembered that there was no problem of permeation with metal tubes, the main problem being reactivity between product and metal.

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MR. R. N. WHEELER, referring to the use of high molecular weight epoxy protective lacquers, said that presumably such materials were based on uncured high molecular weight resins, which might be expected to show a high solvent vapour permeability. Also, an epoxy ester was compared with an alkyd-amino system. Had an epoxy ester-amino resin system been examined, since this might well be superior?

MR. Pye said that generally speaking the weight losses were not less, and indeed, slightly higher than for an alkyd system, which was rather surprising, but, as he had mentioned, this depended on the identity and composition of the product being packed.

## An investigation of the ammonia and heat tests for the yellowing of white paints

#### By C. A. Chaplin and R. A. Fish

Scientific Branch, Greater London Council, County Hall, London, S.E.1.

#### Summary

Measurements have been made of the yellowing of white alkyd gloss finishing paints under conditions of natural exposure in central London, and these results have been compared with the results of accelerated yellowing induced by ammonia tests and heat tests. No consistent correlation has been found between these natural and accelerated tests and it is concluded that predictions of yellowing based on such accelerated tests are not valid for the London area.

#### Une investigation sur des essais pour déterminer le jaunissement des peintures blanches par le moyen de l'ammoniaque et la chaleur

#### Résumé

Observations ont été efectuées du jaunissement des peintures blanches brillantes aux alkydes sous conditions d'exposition naturelles au centre de Londres. Les résultats ont été comparés des résultats obtenus par le jaunissement accéléré produit par l'exposition à l'ammoniaque et à la chaleur. On n'a pas trouvé de corrélation reproductible entre ces essais. On n'a pas trouvé de corrélation reproductible entre ces essais naturels et accélérés et on conclut que des prédictions de jaunissement dérivées de ces essais accélérés n'ont pas de validité en ce qui concerne la zone Londonienne.

## Eine Untersuchung der Ammoniak- und Hitzeprüfungen auf das Vergilben weisser Anstrichfarben

#### Zusammenfassung

Das Gelbwerden weisser Glanzemaillen auf Alkydharzbasis wurde unter den bei natürlicher Exponierung in Zentral-London herrschenden Bedingungen gemessen. Diese Ergebnisse wurden mit denen mittels der Ammoniak- und Hitzeprüfungen erzeugten beschleunigten Vergilbung verglichen. Es konnten jedoch keine übereinstimmenden Beziehungen zwischen den Prüfungen unter diesen natürlichen und beschleunigten Bedingungen festgestellt werden. Es wird daher die Schlussfolgerung gezogen, dass Voraussagen für den Vergilbungsgrad auf Grund solcher beschleunigten Versuche für die Gegend von London wertlos sind.

### Исследование ожелчения белых красок посредством аммиачных и термических испытаний

#### Резюме

Измерялось ожелчение белых, алкидных, блестящих, конечных красок в условиях атмосферного выветривания в центральном Лондоне, и эти результаты сравнивались с результатами ускоренного ожелчения обусловленного аммиачными и термическими испытаниями. Не обнаружено постоянной корреляции между натуральными и ускоренными испытаниями и сделано заключение что предсказания ожелчения, основанные на таких ускоренных опытах, не справедливы в районе Лондона.

JOCCA

In 1950, the Baltimore Club<sup>1</sup> published results comparing various accelerated yellowing tests with natural indoor exposure on eight white paints (two alkyd media, each with four different pigmentations). Very good correlation with natural exposure was obtained in tests exposing painted panels at room temperature to an ammonia atmosphere prepared by placing an open beaker of 28 Bé ammonia in an  $18 \text{ in} \times 18 \text{ in} \times 18 \text{ in}$  glass cabinet. Five hours of such exposure gave results equivalent to seven months of normal indoor exposure.

In 1963, Valentine<sup>2</sup> gave qualified approval to this type of test, particularly with regard to alkyd paints, and said that exposure of films over either a 2-5 per cent aqueous solution of 0.88 s.g. ammonia or solid ammonium carbonate was equally effective. He suggested that such a procedure might simply be intensifying the prime factor responsible in normal yellowing, i.e., ammonia or nitrogenous bases in the atmosphere. The present authors consider that although this may well be true in some locations (e.g. in kitchens, toilets, etc.) it is unlikely to be true generally of London and similar large urban areas. In rural atmospheres free ammonia in the atmosphere may arise from farmyards or other nitrogenous concentrations, but in urban areas it probably only arises appreciably from the destructive distillation of coal and, possibly, from sewage treatment works. The GLC Scientific Branch carries out regular determinations of atmospheric pollutants sampled at 12 points in the London area. No measurements of ammonia concentrations are made, as acidic components of the air are always in excess in towns and the rainfall in London can have as low a pH as 4.0. In the London atmosphere, the average sulphur dioxide content is about 0.1 part per million by volume and may reach 10 times that amount during fog conditions. Compared with these figures any concentration of free ammonia in London's atmosphere must be negligible. Before adopting an ammonia test technique in the paint approval system of the London County Council (now the Greater London Council) it was, therefore, considered desirable to establish its validity for the purpose. This was done as follows :----

For estimating the yellowness of the panels a reflectance spectrophotometer was used with a head carrying a tri-stimulus wheel. Reflectance figures were obtained through each of the three filters (x, y and z) in turn compared with reflectance from a standard magnesium carbonate block. Yellowing was expressed by the formula  $\frac{100 (x-z)}{y}$ . The authors have found this over a considerable period to be a reliable method and throughout these tests the results were consistent with visual inspection.

Eight white alkyd gloss finishing paints, chosen to cover an expected wide range of yellowing properties, were applied by brush to identical clear glass substrates to give an estimated wet film thickness of 60 microns. As only one exposure cabinet was available not all the panels were coated on the same day, but after seven days drying each panel was measured for yellowness and then exposed.

The exposure cabinet consisted of a cylindrical glass container,  $11\frac{1}{2}$  in. in internal diameter, 6 in high and with a ground rim carrying a normal desiccator-type lid. Supports were placed in the cabinet so that 16 panels could be accommodated at one time. A 600 ml beaker in the centre contained 200 ml of a dilute ammonia solution as under :—

Duplicate panels were exposed

for 3 hours to 2% of ammonia (0.88 SG)

,,	5	••	,,	2%	,,	,,	••
••	2	days	,,	5%	••	••	••
••	5	,,	,,	5%	,,	,,	••
,,	8	,,	,,	5%	"	,,	,,
,,	15	••	,,	5%	,,	"	••

Not all panels could be placed at equal distance from the source of the ammonia and duplicate panels were therefore mounted at varying distances but, as might be expected, no clear differences arose between duplicates. (In the five-day, eight-day and 15-day exposures, panels generally developed some degree of blistering.)

Duplicate panels of all the paints were left to yellow naturally in a room in reasonably good light and measured at ages of 12, 18, 24 and 27 months. As the panels were of glass they were always read over a black tile to eliminate any effect of the colour of the support.

Results are given in Tables 1 and 2, and the means of the duplicate pairs are shown in histogram form in Fig. 1 (p. 753). (The paints have been designated A-H in order of increasing degree of yellowing on 18 months' interior exposure.)

Original Paint		iginal 12 months		18 months		24 m	onths	27 months		
raint	(a)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	
A	-1.5	1.0	2.5	2.0	3.5	1.5	3.0	2.5	4.0	
В	-1.5	1.0 1.5	2.5 3.0	2.0 2.0	3.5 3.5	2.0 3.0	3.5 4.5	2.5 3.5	4.0 5.0	
С	-3.0	1.5 0.5	3.0 3.5	2.0 1.0	3.5 4.0	3.0 1.5	4.5 4.5	3.5 1.0	5.0 4.0	
D	-1.0	0.5 2.5	3.5 3.5	1.0 4.0	4.0 5.0	1.5 3.0	4.5 4.0	1.0 3.5	4.0 4.5	
		2.5	3.5	4.0	5.0	3.0	4.0	3.5	4.5	
E	0.5	3.5 4.0	3.0 3.5	6.0 6.0	5.5 5.5	4.0 4.0	3.5 3.5	4.5 5.0	4.0 4.5	
F	-2.5	2.0 2.0	4.5 4.5	3.0 3.0	5.5 5.5	1.5 1.5	4.0 4.0	2.5 2.5	5.0 5.0	
G	-1.5	2.0	3.5	4.5	6.0	3.5 3.0	5.0	4.0 3.5	5.5 5.0	
Н	-1.0	2.0 5.0	3.5 6.0	4.0 6.0	5.5 7.0	4.5	4.5 5.5	5.0	6.0	
		4.5	5.3	5.5	6.5	5.0	6.0	5.0	6.0	

Table 1

Natural interior	exposures (a)	recorded	yellowness	<b>(b)</b>	increase	of	yellowness	over	original
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The four periods of natural interior exposure yielded broadly similar results. (It is interesting to note that generally there was no increase of yellowness after 18 months, though some fluctuations are recorded, possibly caused by seasonal variations in hours and intensity of daylight.)

Paint	Ori- ginal	3 hou	rs 2%	5 hou	rs 2%	2 dag	ys 5%	5 dag	ys 5%	8 dag	ys 5%	15 da	ys 5%
Faint	(a)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)
A	-1.5	4.0	5.5	6.0	7.5	14.5	16.0	19.0	20.5	21.5	23.0	23.5	25.0
в	-1.5	3.5 1.5	5.0 3.0	5.0 2.5	6.5 4.0	15.0 11.5	16.5 13.0	17.5 12.5	19.0 14.0	20.0 15.0	21.5	23.0	24.5
С	-3.0	0.0 1.0	1.5 4.0	1.5 3.5	3.0 6.5	11.5 12.5	13.0 15.5	12.5 16.0	14.0 19.0	13.5 18.5	15.0 21.5	15.0 21.0	16.5
D	-1.0	2.0	5.0 1.0	4.0	7.0	12.5	15.5 9.5	17.5	20.5	20.5	23.5	21.0	24.0
Е	0.5	0.5 4.0	1.5	2.0 6.5	3.0 6.0	8.5 21.5	9.5 21.0	10.0 24.5	11.0	11.5	12.5	16.5 29.0	17.5
F	-2.5	3.5	3.0 4.0	6.0	5.5	22.5	22.0	25.5	25.0	27.5	27.0	31.0	30.5
		1.0	3.5	1.5 1.5	4.0 4.0	11.5 10.5	14.0 13.0	11.5 11.0	14.0 13.5	14.0 13.0	16.5 15.5	13.0 15.0	15.5 17.5
G	-1.5	2.0 2.0	3.5 3.5	3.0 3.0	4.5 4.5	11.0	12.5	12.0 12.0	13.5 13.5	14.0 15.0	15.5 16.5	17.0	18.5
н	-1.0	2.0 3.0	3.0 4.0	4.5 5.5	5.5 6.5	16.5 16.5	17.5	19.0 18.0	20.0 19.0	20.5 20.5	21.5	22.5 21.5	23.5

The results obtained by exposure to ammonia did not generally correspond with those obtained by natural ageing. For instance, the ammonia tests predicted that paints A and C would have strong yellowing tendencies, but in fact they showed a relatively small degree of yellowing in the natural tests. The respective manufacturers of paints D and F claim their products to have superior whiteness retention. The ammonia tests support this, but the natural exposure tests do not.

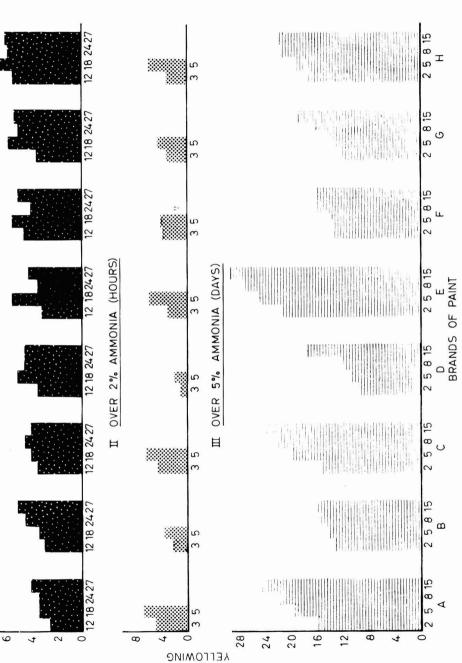
As far as the authors are aware, the ammonia test has never been suggested for predicting the yellowing of paints on exterior exposure. Chalking, erosion of the surface, intensity of incident uv light, etc., would be expected to affect the colour of a paint on exterior exposure. Nevertheless, as six of the above paints were submitted as being also suitable for exterior use, opportunity was taken to measure the yellowness of the paints shown on the exterior-exposure panels which form part of routine testing. At two years all showed a remarkably similar yellowness, viz.:

A = 4.0	B = 4.5	C=interior brand
D=4.5	E = 4.0	F=4.0
G = 4.0	H=interior	brand

Since the original degree of yellowness of these panels had not been measured the actual yellowing cannot be calculated. Nevertheless, it seems clear that the ammonia test is not suitable for predicting yellowing on exterior exposure.

It is concluded that simple ammonia tests of the type described are not reliable for the prediction of the yellowing properties of alkyd based hard gloss white paints in normal London atmospheres.

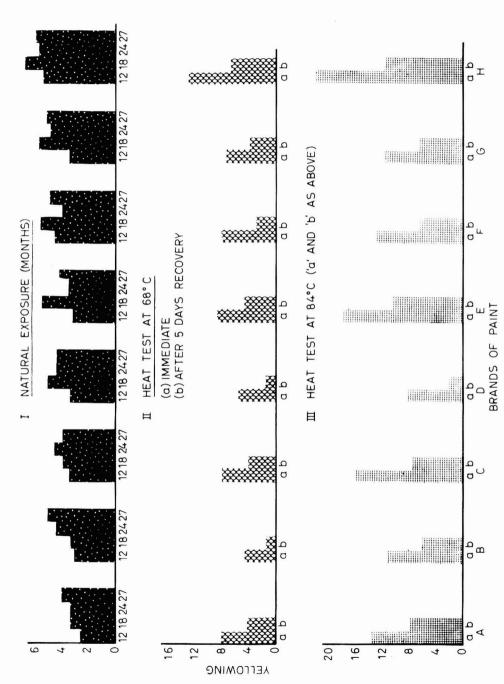
Another test method occasionally used for predicting the yellowing of white paints measures the degree of yellowing suffered by panels maintained at an elevated temperature. For most air-drying paints, panels are kept at a fixed temperature, usually between  $60^{\circ}$  and  $70^{\circ}$ C, for about four days. The Baltimore Club<sup>1</sup> commented favourably on this type of test but otherwise there seems to be little published information as to its reliability.





NATURAL EXPOSURE (MONTHS)





With most laboratory ovens, panels placed inside are maintained in total darkness and this can itself induce a temporary yellowing effect. Different degrees of yellowing can, therefore, be recorded according to whether the yellowness is measured soon after removal of the panels from the oven or later, after some days in the light, when partial recovery to whiteness may have occurred.

In the authors' preliminary tests, panels maintained at  $68^{\circ}$ C for four days in a dark oven and then left at room temperature in the light for five days, gave about the same degree of yellowing as panels given 18 months' natural interior exposure. This temperature was, therefore, accepted as suitable for the later tests, but tests at both higher and lower temperatures, viz., at  $50^{\circ}$ C and  $84^{\circ}$ C were also carried out. In each case, panels were left for four days (96 hours) in a dark oven adjusted to the selected temperature. Yellowness readings were made on the abstracted panels (i) almost immediately after cooling and (ii) at the end of a further 5-6 days normal exposure to daylight.

In the tests at 50°C only a small degree of yellowing occurred, and even this almost completely disappeared after leaving the panels five days in diffused daylight. Tests at 68°C and 84°C produced more definite yellowing: the figures recorded are tabulated in Table 3 and shown in histogram form in Fig. 2.

Paint	Original	4 day	∕s 68°C	4 days 68°C 5 days recovery		4 day	∕s 84°C	4 days 84°C 5 days recovery		
1	(a)	(a)	(b)	( a)	(b)	(a)	(b)	(a)	(b)	
А	-1.5	6.5	8.0	2.5	4.0	12.0	13.5	6.0	7.5	
B	-1.5	3.0	4.5	0.0	1.5	9.5	11.0	5.0	6.5	
С	-3.0	5.0	8.0	1.0	4.0	13.0	16.0	4.0	7.0	
D	-1.0	4.5	5.5	0.0	1.0	7.0	8.0	1.0	20	
E	0.5	9.0	8.5	5.0	4.5	18.5	18.0	11.5	11.0	
F	-2.5	5.5	8.0	0.5	3.0	10.5	13.0	4.0	6.5	
G	-1.5	6.0	7.5	2.5	4.0	10.0	11.5	5.0	6.5	
н	-1.0	12.0	13.0	5.5	6.5	21.0	22.0	10.0	11.5	

Table 3

Heat tests (a) recorded yellowness (b) increase of yellowness over original

As can be seen, correlation with natural interior exposure was poor in each series. It is concluded that this test is no more reliable than the ammonia test for the prediction of the degree of yellowing of alkyd-based hard gloss white paints in normal London atmospheres.

[Received 22 February 1966

#### References

1. Baltimore Club, Off. Dig., 1950, 22, 815.

2. Valentine, L., JOCCA, 1963, 46, 692.

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J. Oil Col. Chem. Assoc. 1966, 49, 756-769

# Coloured pigmentation for electrodeposition<sup>\*</sup>

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#### Summary

The effects of discoloration and hardness of films on baking caused by iron from anodes is discussed along with the effects on colour of films on various substrates. These are illustrated by spectrophotometric curves. Questions are raised as to the general application of results obtained by other workers using titanium dioxide pigmentation. The effects of oxidation at the anode and methods to eliminate defects arising therefrom are briefly discussed. The use of organic anti-corrosive agents suggested. The examination of electrodeposited film by Talysurf is illustrated.

#### La pigmentation des peintures de couleur pour l'électrodéposition

#### Résumé

On discute l'effet du fer en provenance des anodes sur l'altération de la couleur et la dureté des feuils après le sechage au four, ainsi que l'effet sur la couleur des feuils produite par des subjectiles diverses. Les effets sont illustrés par des courbes spectrophotométriques. On pose des questions quant à l'applicabilité en général des résultats obtenus par autres investigateurs qui ont utilisé le dioxyde de titane dans la pigmentation. On discute brièvement à la fois les effects de l'oxydation à l'anode et les méthodes pour éliminer les défauts qui en suivent. On propose l'utilisation des agents anti-corrosifs organiques. On démontre l'examen d'un feuil de peinture électrodéposé à l'aide de l'appareil Talysurf.

#### Farbige Pigmentierung für elektrophoretische Anwendung

#### Zusammenfassung

Es werden die durch von Anoden stammendes Eisen bei Ofentrocknung hervorgerufenen Wirkungen auf die Verfärbung und Härte von Filmen, ebenso wie diejenigen auf die Farbe von Filmen über verschiedenen Substraten besprochen. Sie werden mit Hilfe von Spektrophotometerkurven illustriert. Von anderen auf dem gleichen Gebiet arbeitenden Chemikern, die aber mit Titandioxyd pigmentierten, erzielte Resultate werden hinsichtlich allgemeiner Anwendbarkeit einer kritischen Betrachtung unterzogen. Die Auswirkungen von Oxidation an der Anode, und Methoden dadurch verursachte Fehlschläge auszuschalten werden kurz besprochen. Die Anwendung organischer, antikorrosiver Mittel wird angeregt. Die Prüfung mit Hilfe des Talysurf Instruments von durch Elektrophorese erzeugten Filmen wird illustriert.

#### Красочная пигментация для электро-осаждения

#### Резюме

Обсуждается влияние на обесцвечивание и твердость пленок при спекании, обусловленное анодным железом, а также влияние на цвет пленок на различных нижних слоях. Эти влияния иллюстрируются спектрофотометрическими кривыми.

Вызываются вопросы об общем применении результатов других сотрудников с использованием пигментации посредством двуокиси титана.

Влияния окисления около анода и методы для устранения таких дефектов кратко обсуждаются.

Предложено применение органических противокоррозийных средств. Иллюстрируется осмотр электро-осажденных пленок с помощыю Таллисурфического инструмента.

\*Read to Bristol Section, 7 January 1966—and containing work presented by W. B. Curtis to Newcastle Junior Section, 14 December 1965.

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#### Introduction

It is not proposed to devote time to the general principles of electrodeposition as these have been dealt with fully in the past by various authors<sup>1, 2, 3, 4, 5</sup>, but to consider some aspects of pigmentation and its effect on the electrodeposited film with particular reference to colour.

Many of the resins which have been used successfully for the electrodeposition of dark coloured primers and finishes are unsuitable for production of light shades because of the discoloration of the resin during stoving. Such resins are often of the phenolic and solubilised oil types. The colour of such films may also be affected when exposed in subdued light. These defects are overcome by the use of other resins, for example, acrylics. In part of the work described, acrylic water soluble resins, which gave pale coloured unpigmented films on stoving with good colour retention and which were not expected to show discoloration due to the reactions taking place at the anode, were examined.

#### The effect of type of resin on the colour of the deposited film

#### Discoloration of deposited films on iron anodes

It is well known that metal ions are often present in the deposited film because of their migration from the anode during electrodeposition. When iron anodes are employed the amount of iron present in the film has been found to be as high as 0.6 per cent for many resins and that in the case of thin films the presence of the iron has caused discoloration during stoving. Various theories have been put forward to explain the function of the iron in the film after stoving. Tawn and Berry<sup>3</sup> have suggested that it is present as a salt of the carboxyl in the resin. In our work the analysis of the iron content of a film of solubilised oil type resin has shown it to be in the range of 0.2-0.6 per cent dependent upon conditions of electrodeposition. This quantity is not sufficient to react with all the carboxyl groups in a resin of acid value 120 milligrams KOH/gm but has been found to produce darker coloured films on stoving than similar films applied to non ferrous metals by spraying.

Per cent solids content	Time of deposition (secs)	Resistance (ohms ft <sup>2</sup> )	Specific resistance	Per cent Fe in stoved film	Per cent carboxyl ions reacted
5	120	145	73	0.64	41.4
10	60	30	12	0.42	26.9
10	120	49.5	17	0.52	33.2
10	300	66.5	14	0.59	38.2
20	60	7.5	11	0.22	14.1

	Table 1
Iron content	of electrodeposited films

Fig. 1 shows graphically the variation in film resistance with percentage iron content, the broken curve represents film resistance in terms of resistivity, on deposition of an unpigmented resin.

Tawn showed that, in simple precipitation experiments, the amount of ferrous sulphate needed for complete coagulation of resin from solution was roughly

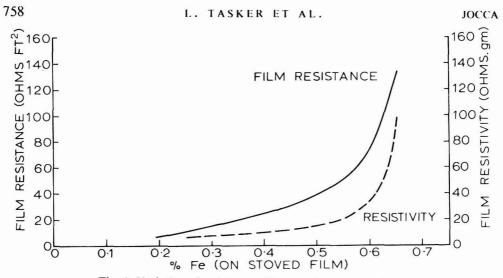


Fig. 1. Variation of resistance with percentage iron content

equal to that calculated from the apparent equivalent weight of the resin as defined above. Finally, the precipitates so obtained were unlike the electrodeposits on mild steel in being insoluble in organic solvents such as methanol and acetone.

It has been observed that the iron present in a film of maleinised oil has the effect of increasing the hardness of the film, acting as a catalyst during stoving. On zinc phosphated steel the amount of iron which may be present in the film and which can react with the resin is again dependent upon the electrical conditions employed, but is generally found to be lower than when untreated iron is used as the anode.

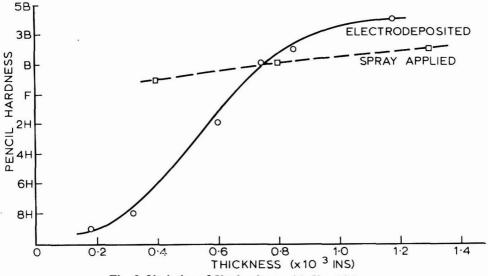


Fig. 2. Variation of film hardness with film thickness

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#### The effect of anodic iron on the hardness of electrodeposited film

During the examination of the variation in iron in the stoved film with time of deposition, and the determination of film resistance, it was observed that there was a significant difference in film hardness as the percentage of iron content changed. Fig. 2 shows the variation of film hardness with thickness for films of the same resin applied by electrodeposition and spraying. In the case of the electrodeposited film the hardness decreases with film thickness but the spray applied film does not show significant variation.

#### Effect of iron on pigment flocculation

During the investigation of the effect of anodic iron on film properties there has been no evidence to suggest that iron in solution has been responsible for the flocculation of multiple pigment systems and hence for colour changes when deposited films are compared with films of the same paint applied by conventional methods.

#### Non-ferrous substrates

A simple acrylic paint pigmented with Tioxide RCR at a pigment/binder ratio of 0.5:1 and diluted to 10 per cent solids was deposited under a potential difference of 60 volts for two minutes on the same area of anodes of various metals, viz, mild steel, tinplate, aluminium and Zintec.

Fig. 3 shows the Recording Spectrophotometer curves produced by these films.

From examination of these it is significant that the film applied to the mild steel anode has a poorer overall whiteness and a noticeable red tone. The characteristics of the reflectance curves on Zintec, aluminium and tinplate are similar, between wavelengths of 380 to 700 millimicrons. This difference in whiteness is possibly due to the difference in film thickness obtained under fixed conditions, the thickest film being obtained on tinplate and the thinnest on Zintec, under these conditions.

#### Continuity of colour on deposition under various conditions

An acrylic resin paint with a pigment/binder ratio of 0.5:1 using Tioxide RCR, tinted to a pale grey colour with Rajah Beads, dispersed in the same medium, was deposited under the following conditions :

3 amps per sq.ft 20, 40, 60 and 80 volts on mild steel panel.

Fig. 4 shows the reflectance curves obtained on the Recording Spectrophotometer. Although there are small differences in reflectance at the blue end of the spectrum, the colours are very similar at the red end.

We conclude that the concentration of iron ions produced under the various conditions of deposition does not have a significant effect on the colour of baked films.

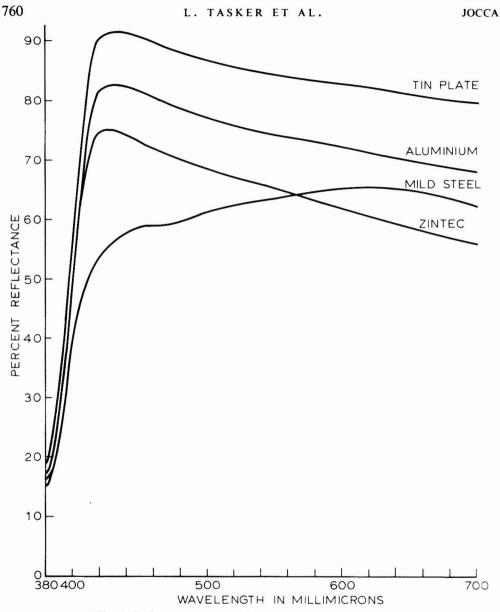


Fig. 3. Reflection spectrum of the paint on various substrates

The work of Rechmann<sup>4</sup> and Landon and Ashton<sup>5,</sup> on the electrodeposition of titania pigmented resins is of particular interest. Landon and Ashton have described the deposition of titania pigmented acrylic resins to zinc plated steel. This type of anode was selected in order to eliminate the effects of anodic iron in the films. They suggested that examination of the deposited films showed an increase in pigment/binder ratio with duration of deposition and that there was a deficiency of pigment in the film adjacent to the substrate. Rechman has suggested that the pigment concentration in a deposited film was either constant





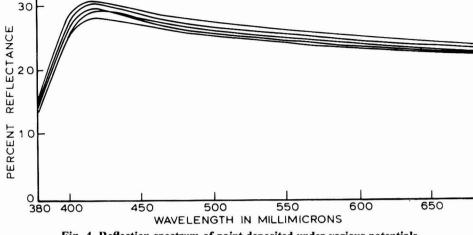


Fig. 4. Reflection spectrum of paint deposited under various potentials

or decreased on deposition in the case of rutile titania, but that some anatase titania showed an increase in pvc in comparison with the composition of the paint in the deposition cell. We do not agree that these suggestions are of general application to paints for electrodeposition. The general conclusion to be drawn from the work by the titania manufacturers is that consideration of pigment and resin must be undertaken as an entity since one grade of titania may be more suited to use with a particular resin than others.

#### Distribution of titanium pigments in electrodeposited films

Following Landon and Ashton's work, and in order further to examine distribution of pigment in electrodeposited films and its effect on the colour of the film, two pale blue paints were prepared.

The pigment combination was as follows :

Monastral Fast Blue LBXS	20 parts by weight
Tioxide RCR3	50 parts by weight

These were dispersed in two different water soluble acrylic resins reduced to pigment/binder ratio of 0.5:1 and deposited under a potential of 60 volts for two minutes at 10 per cent solids concentration. Films were deposited on mild steel anode at a thickness of about  $0.8 \times 10^{-3}$  in.

The stoved films were sectioned by rocking microtome, sections of about 0.1 micron thickness being obtained. After shadowing with palladium these were examined by electron microscope. Figs. 5, 6, 7 and 8 show that there is little difference in pigment dispersion between such films and between similar films applied by spraying. These results do not substantiate those obtained by other workers. The pigment is well distributed throughout the electrodeposited film and flocculation is not markedly different from that obtained in the sprayed film. There is not a clear resin film at the anode surface nor at the air interface in the cases of the deposited and sprayed paint films respectively.

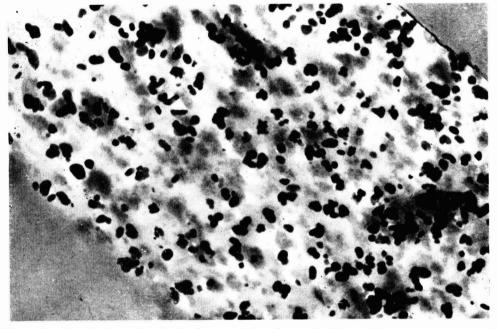


Fig. 5. Section of electrodeposited paint containing first resin. The paint/substrate boundary is visible at the top right

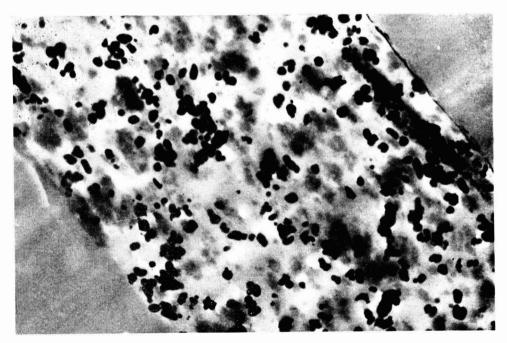
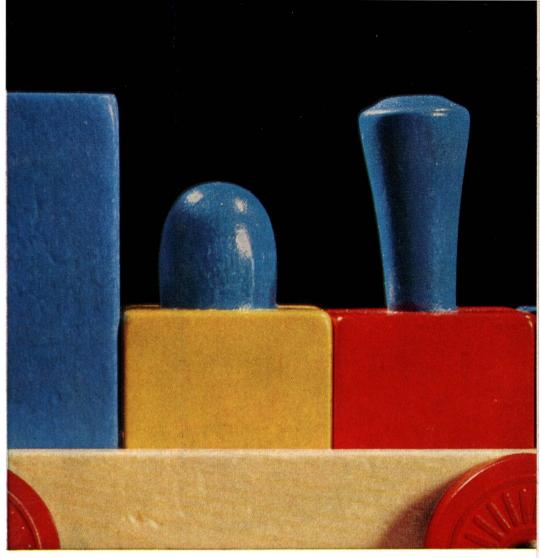


Fig. 6. Section of sprayed paint containing first resin. The paint/substrate boundary is at the top right



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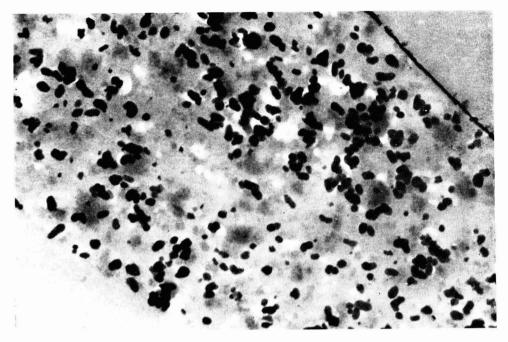


Fig. 7. Section of electrodeposited paint containing second resin. The paint substrate boundary is at top right

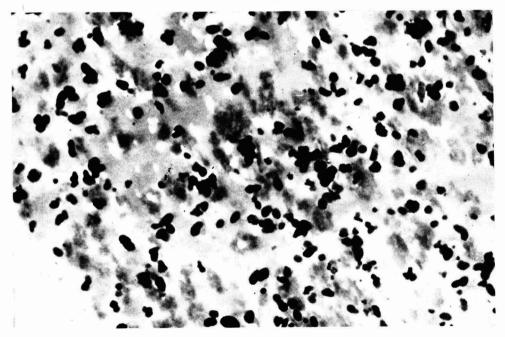


Fig. 8. Section of sprayed paint containing second resin. The air substrate boundary is visible bottom left, as in Figs. 5, 6 and 7

No surfactant was added to these paints since both resins were found to be good dispersants for the pigment systems.

Investigation of the use of surfactants in titania/acrylic resin paints for electrodeposition was made by Landon and Ashton. They found that the addition of Calgon S to such systems in quantities of the order of 0.1 per cent on the paint produced films of increased gloss, but had no effect on the coulomb yield. The use of polyacrylate dispersing agents also gave increased gloss and thicker films.

It has been the experience of the authors that white or coloured paints based on titanium dioxide yield thicker films of higher gloss when pigment/ binder ratio is reduced to a minimum. The balance between optimum film thickness with adequate gloss and satisfactory opacity is somewhat delicate to strike for any particular conditions of deposition.

#### Oxidation at the anode

Recent work has shown that oxidation of the polymer can take place due to the liberation of nascent oxygen at the anode. Sullivan<sup>7</sup> has published data on this effect. It is stated that the resin may be polymerised under these conditions, especially on prolonged periods of electrodeposition, the resulting paint on resin film becoming increasingly rough because of the increase in viscosity and reduction of flow of the polymerised resin. These have been confirmed in our recent work. The addition of pigments prepared from easily oxidised metal to paint formulations are suggested by Sullivan to prevent polymerisation of the resin and are described as scavenger metals.

We have investigated the use of anti-oxidants in the electrodeposition bath and found that the number of turnovers of the tank during use can be prolonged. For example, 0.2 per cent of hydroquinone, calculated on the weight of resin, has been found to increase significantly the stability of the paint when a simple maleinised vegetable oil was examined. It is not yet possible to make general recommendations as the type of anti-oxidant appears to be specific to a particular resin type and the following have been examined and found to be of interest : guaiacol,  $\beta$ -naphthol, phenol, methyl ethyl ketoxime and pyrocatechol.

## Pigmentation of paints containing more than one tinting pigment and maintenance of shade during long term

White and red oxide paints of the composition shown below were prepared and electrodeposited with daily work load greater than that expected to be used in practice and with pauses for weekends and seven-day intervals. Initially the tank contained only the red oxide paint but on replacing the contents of the tank to keep the solids at 12 per cent only white paint was added.

	Red oxide	White
Red iron oxide MR11	20	
Titanium dioxide Tioxide RCR		20
Epok WL.1762	36.4	36.4
Water	43.6	43.6
	100.0	100.0

The electrodeposition bath was maintained at 12 per cent solids and the two invidual paints had pigment/binder ratios of 1:1. The medium was an oil modified type resin.

By mixing the two paints in known proportions and preparing standard colour panels it was possible during the deposition to check if the proportions of pigment deposited were the same as expected from the known contents of the bath. Fig. 9 shows the varying amounts of red oxide and titania present in the mixture as the bath was made up. Over a period of three months the shades which were obtained by deposition were similar to those obtained by mixing known amounts from the freshly prepared paint.

The rate of consumption of the red oxide paint with surface area coated is shown graphically.

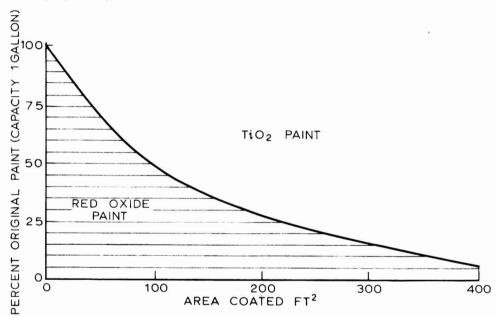


Fig. 9. Rate of consumption of red oxide with surface area coated

#### Effect of change in pH of the bath on the coloured film

Fig. 10 shows the effect of change of pH on the electrical resistance and film retained after washing of a deposited paint film. It is known that as the electrical resistance increases the uniformity and texture of the film often becomes unacceptable. It has not been evident that the colour of the film is altered, apart from the visual effect of roughness.

#### Effect of temperature in electrodeposition bath of the colour of the deposited film

Fig. 11 shows that change of temperature affects the electrical resistance and film retained after washing a paint film, as it is being built up on the surface of the anode. As the electrical resistance increases so the uniformity of the film surface decreases, but no change in colour has been noted.

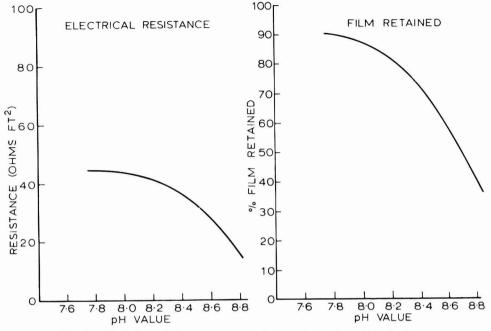


Fig. 10. Variation of film resistance and percentage film retained with pH

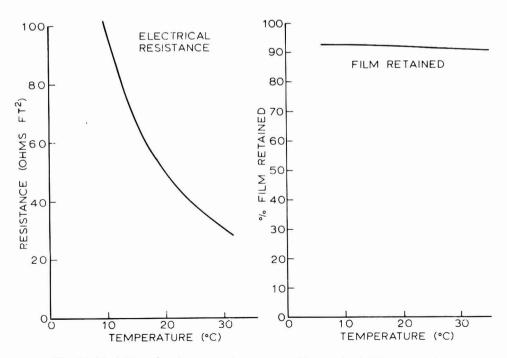


Fig. 11. Variation of resistance and percentage film retained with temperature

The results were obtained using a modified oil type resin pigmented as follows:

Red oxide primer

Formulation :				
Resin (55 per cent NV)	60.7 p	arts	by v	veight
Lead silico chromate (M.50)	0.8	,,	,,	,,
Natural red oxide (MR11)	7.9	,,	,,	,,
Micronised barytes	4.0	,,	,,	,,
Norwegian talc	4.0	,,	,,	,,
Water	22.6	,,	,,	,,
Ammonia to pH 7.8	trace			
	100.0			

Films were deposited on duplicate panels using a potential difference of 60 volts for two minutes, and solids content of 10 per cent. One panel was rinsed before baking. Bath temperatures between 4°C and 45°C were employed.

The experiment was repeated using the same pigmentation in a variety of water soluble resins. Whilst the order of resistance changed the graphs followed the same general pattern.

#### Agitation of the contents of the bath

Adequate agitation of the contents of the bath, by stirring or re-circulation, is essential to prevent settlement of the pigment. Apart from settlement in the bottom of the vessel it is necessary to prevent settlement of pigment on horizontal surface of articles which are being coated. Pigment settlement on such surfaces produces films which are unsatisfactory because of their rough surface texture. The colour of such films is not significantly different from that of the film which has been deposited satisfactorily. These films do, however, contain a higher proportion of pigment than that in the body of the liquid paint. Even if such deposition was acceptable from the point of view of film appearance the removal of excess pigment from the bath would necessitate very careful control and specially formulated paint for topping up. In practice prevention by adequate agitation is better than cure.

#### Pigmentation of anti-corrosive paints

The use of lead silico-chromate and strontium chromate has been widely recommended for the preparation of corrosion resistant paints for application by electrodeposition. As both have some tinctorial properties they are not suitable for use in the formulation of many very pale coloured paints. There is some objection to their use on the ground of toxicity. Chromates in general use are sometimes considered to be hazardous because of their possible irritant effect on the skin and lead is in some cases prohibited. There have been comments also that chromates show preferential settlement and suggestions that they may cause instability in some specific electrodeposition systems.

Alternative rust inhibiting additives have been briefly examined with promising results. Zinc phosphate confers appreciable rust inhibition and may

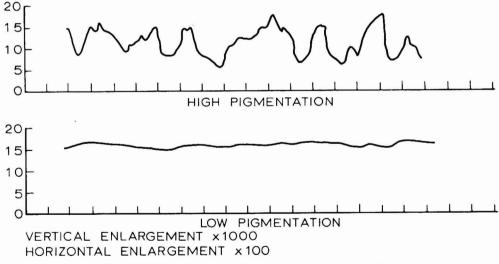
be considered to be a satisfactory substitute for the chromate, used in the same proportions.

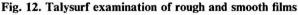
Exploratory work using organic phosphates has shown great promise and this work is continuing. Alternative organic compounds which have given promising results include metallic benzoates, for example, sodium lead and zinc salts. Quaternary amine compounds have also shown promising results. Apart from other advantages this type of rust inhibitor has no tinctorial effects.

It is necessary to avoid contamination of the deposition bath by soluble salts as these can give rise to instability in the same way as traces of pre-treatment carry over. Stability to soluble salts varies widely from resin to resin.

It has often been found by many workers that the corrosion resistance of an electrodeposited film may be inferior to that of a similar film applied by conventional methods or solvent based analogues. Careful examination of films has shown that the reduction in corrosion resistance can be caused by disruption of the film due to either gassing at the anode if the initial deposit has very low electrical resistance or very uneven deposition if the resistance is too high. In his lecture to the OCCA Conference in 1965, Rechmann illustrated such film defects with some very well prepared photographs.

Fig. 12 shows traces of Talysurf examination of rough and smooth films applied by electrodeposition. In this case the variation in surface roughness was produced by the change in pigment/binder ratio of an iorn oxide based primer from  $1\frac{1}{2}/1$  to  $\frac{1}{2}/1$ .





#### Conclusions

So long as resins under consideration do not discolour on baking or on subsequent storage in subdued light the pigmentation of coloured paints for application by electrodeposition is possible. Batch to batch variation will not be a serious problem providing that the effects of variation of pH, temperature, solids content and dispersion are taken into account.

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Properties apart from colour, such as stability of the bath in long term use, and pigment suspension, may vary between trials in a small cell and in a production unit. It does not yet appear that scaling up follows small scale results. The larger the scale on which trials can be carried out the better.

#### Acknowledgments

The authors are indebted to Mr. B. Curtis, for his assistance with the experimental work, to Mr. J. J. Wilson of Distillers Plastics Services Company Limited for the photography and preparation of illustrations, and to the Directors of British Resin Products Limited for permission to publish this work.

#### [Received 30 March 1966

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# Prefabrication primers for structural steelwork<sup>\*</sup>

#### By D. A. Bayliss and D. C. Wall<sup>†</sup>

Research & Development Department, S.E. Region, Central Electricity Generating Board, Cockfosters, Herts.

#### Summary

An examination of prefabrication primers carried out for the Central Electricity Generating Board has shown, after 18 months' exposure, that a phenolic resin modified single pack etch primer has advantages over certain zinc or aluminium containing primers in holding blast cleaned structural steelwork until it can be painted on site.

The etch primer shows advantages in respect of cost, and ease and consistency of application and, at the same time, gives reasonable corrosion protection on various types of blast cleaned surfaces, even in thin films. If rusting of the exposed primer does occur, it is light and more uniform than in the case of the zinc containing primers and is easily prepared for subsequent painting. Alternatively, if the primer film is still largely intact, it will present less difficulty when overcoated by conventional paint systems.

## Peintures de fond pour protéger les charpentes en acier après leur préfabrication

#### Résumé

Une investigation des peintures de fond pour cet emploi effectuée au nom du Central Electricity Generating Board (la commission pour la génération d'électricité), a démontré après une exposition de 18 mois aux intempéries les avantages que possède un "wash-primer" à seul emballage modifié à une résine phénolique, auprès de certaines peintures de fond contenant zinc ou aluminium, en ce qui concerne la protection, jusqu'à la peinture à pied d'œuvre, des charpentes en acier décapées par grenaillage.

Le "wash-primer" démontre des avantages à l'égard de prix, et à la fois de la facilité et de la consistance de l'application, et en même temps donne une protection anti-corrosive raisonnable aux types divers de surfaces décapées par grenaillage, même avec des couches minces. Si la formation de rouille se produira pendant l'exposition du "wash-primer," en comparaison des peintures de fond au zinc ou à l'aluminium, elle est légère et plus uniforme, et la surface est facilement préparée pour la peinture suivante. De l'autre part, si le feuil du "wash-primer" reste encore presqu'intact, il présentera moins de difficulté lorsqu'on appliquera au-dessus des systèmes de peinture conventionnels.

#### Fabrikmässige Voranstrichmittel für Eisenkonstruktionen

#### Zusammenfassung

In einer für den Central Electricity Generating Board ausgeführten Prüfungsserie wurde nach achtzehnmonatlicher Bewitterung aufgezeigt, dass ein mit Phenolharz modifizierter Eintopf-Washprimer gewissen Zink oder Aluminium enthaltenden Primern insofern überlegen ist, als er durch Strahlung gereinigte Eisenkonstruktionen besser schützt ehe sie auf der Baustelle angestrichen werden können.

<sup>\*</sup>Read before the Newcastle Section on 4 November 1965.

<sup>†</sup>Smith and Walton Ltd., Haltwhistle, Northumberland.



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Die Vorteile des Washprimers bestehen in niedrigeren Gestehungskosten, leichter und gleichmässiger Anwendung und, weil er ausserdem verschiedenen Arten durch Strahlen gereinigten Oberflächen selbst in dünnen Filmen angemessenen Korrosionsschutz verleiht. Selbst wenn bei Bewitterung Verrostung so vorgestrichener Flächen eintritt, ist diese leicht und gleichmässiger als bei Anwendung zinkhaltiger Primer, und sie lassen sich leicht für den endgültigen Anstrich vorbereiten. Wenn andererseits der Primerfilm im grossen und ganzen noch unbeschädigt ist, wird das Überstreichen mit den üblichen Anstrichsystemen mit weniger Schwierigkeiten verbunden sein.

#### Заранее приготовленные грунтовки для структурных стальных сооружений

#### Резюме

Исследование заранее приготовленных грунтовок, выполненное ддя Центральной Электрической Генераторной Комиссии показало что, после восемнадцати месяцев выветривания однослойная травленная грунтовка измененная фенолом имеет преимушества по сравнению с некоторыми грунтовками содержащими цинк или алюминий для хранения в чистом состоянии структурных стальных сооружений, очищенных дутьем, до их окрашивания на месте. Травленная грунтовка обнаруживает преимущества в отношении стоимости, удобства и постоянства применения и, в тоже время, дает умеренную защиту против коррозии на различных типах поверхностей очищенных дутьем, даже для тонких пленок. Если подучается ржавление на обнаженной грунтовке, то оно будет носить легкий характер и будет однороднее чем в случае грунтовок содержащих цинк, и грунтовку можно легко приготовить для последующей окраски. С другой стороны, если пленка грунтовки еще в значительной степени цела, то она представит меньше затруднений при перекрытии обыкновенными системами окрашивания.

#### Introduction

In the next five years the building programme of the Central Electricity Generating Board calls for approximately 80,000 tons of structural steel each year. There is a special interest therefore in the provision of long-term maintenance-free protective coatings for such steelwork and in the initial cleaning and the protection of the steel surfaces during the building period, since the general painting must be carried out after fabrication.

Much of the published work on blast cleaning and prefabrication or holding primers has been concerned with submerged marine conditions and it was considered by the authors that more information was required in respect of normal atmospheric exposure. In particular :

- 1. Which holding primer was most suitable for such application?
- 2. What degree of blast cleaning was required?
- 3. How much surface preparation of the weathered holding primer was necessary before the follow-on painting?
- 4. Assuming that the follow-on paints would be conventional undercoat and gloss systems, which was the most satisfactory conventional primer to use?

It seemed necessary to carry out some experimental work on as practical a level as possible and early in 1964 the CEGB and Smith & Walton Limited decided to collaborate on an investigational programme. It was agreed that the results obtained should be made freely available to all, and this paper represents the completion of the first stage of the programme.

#### Millscale

Whilst it is generally accepted that the removal of millscale from structural steelwork is a fundamental requirement to obtain maximum performance

from subsequently applied paint coatings, it was considered important to establish that the process of cleaning and the preservation of the clean surface with a prefabrication primer offers considerable advantage over the current practice of weathering and wire brushing.

In the exposure programme, samples of weathered and wire brushed steelwork were exposed alongside the blast cleaned and treated steelwork. The results re-emphasise the well-known detrimental effect of painting on to millscale.

A paint system of red lead primer to BS 2523, Type B, an alkyd gloss and undercoat, was applied to intact millscale and weathered at Shoreham for 18 months. Isolated breakdown and rusting occurred.

After 16 months' exposure at Shoreham of the same paint system applied to steel which was weathered for two months before wire brushing and painting, the breakdown of the paint system was serious; and before repainting could be carried out it would be necessary to clean down to bare metal.

There was extensive breakdown of the same system after only 12 months' exposure at Shoreham when applied to a surface wire-brushed and painted after six months' weathering.

Rust spotting occurred after six months' exposure on a surface wire-brushed and painted after 12 months' weathering.

It should be noted that even after 12 months' weathering at Shoreham there were still patches of millscale on the steel which were not removed by good hand and mechanical wire-brushing, and that it is to be expected that this standard of preparation was higher than that normally achieved on a construction site.

An identical series of samples were exposed on a roof at Battersea and gave similar results, but at a slower rate of breakdown.

In practice the length of time that structural steelwork for the CEGB is stored on site varies considerably according to the contingencies of the supply position and plant installation programme. The steel can be partly or wholly exposed and the exposure environment can vary as from Brighton to Battersea. It is very unlikely that a uniformly rusted millscale-free surface will be obtained unless the weathering period is very long, and even if this occurs, the surface will then be rough and contaminated with corrosion products.

#### **Experimental methods**

#### Preparation of samples

The basic specimen for the exterior weathering trials was an RSJ steel section 4 ft long and 6 in wide with 3 in deep flanges and 127 such lengths were used in the experiments. Some of these were shot blasted with S230 chilled iron shot on an efficient automatic blast cleaning plant of the airless (impeller) type at the Stockton on Tees works of the South Durham Steel & Iron Co. Ltd.

Half of the sections were passed through the plant at the lowest speed possible (2 ft per minute) to obtain a "white" metal finish and the other half at the fastest available speed (8 ft per minute) to obtain a commercial blast finish.

The level of cleanliness, described as a commercial blast finish, approximated to the Swedish Standard SIS 05.59.00, Type C, Sa 2.

The other RSJ sections were grit blasted manually with abrasive grit Silikasafe, Type B (passes 16 screen, retained on 36 screen), again to two standards of cleanliness similar to those described above.

In all cases the cleaned sections were coated with prefabrication primers, immediately after blasting, by a trained operator using airless spray equipment.

A number of steel sections from the same batch were used to carry out the comparison trials on surfaces which had received no blast cleaning treatment.

#### Formulation

Initially, ten prefabrication primers were included in the experiments and each of these was applied to the two types of blast cleaned surface at two levels of surface cleanliness at two exposure sites. After six months' exposure experience more prefabrication primers were added to the programme, bringing the total to 15.

Before deciding on the composition of these primers, several consultations were held and advice was sought from various experts within the paint industry, both manufacturers and raw material suppliers. Eventually the choice was as given in Tables 1 and 2.

It appeared that the general types to be included should be cold cured epoxy resin based zinc containing primers, both zinc rich and reduced zinc types, epoxy resin based aluminium primers and finally etch primers.

After some consideration it was decided to include in the experiments a number of proprietary prefabrication primers and four of the Board's regular suppliers agreed to co-operate in the programme and submitted their standard products. The composition of these paints is unknown to the authors, but they are all obviously zinc-containing two-pack types.

#### Zinc-containing primers

The variables which had to be considered were the binder and the pigment type and quantity.

Published work indicated that polyamide resin cured epoxy resin provided an ideal binder for zinc rich primers and this combination was accepted as a general standard, but it was decided that an alternative isocyanate curing agent should be used in one instance in order to establish whether or not curing mechanism had any effect on performance. In addition the isocyanate cured epoxy resin combination could have the advantage of curing at lower temperatures than the polyamide cured type.

Table 1     Percentage compositions of zinc and aluminium containing primers—dry film portion
---

					Prim	er refer	Primer reference C.E.G.B. No.	E.G.B.	No.			3	
Component	17	18	1	2	3	4	5	9	10	11	12	15	16
Zinc dust			87.0			92.5	91.7	74.1	66.5			60.2	46.5
Aluminium (flake)	38.4	38.6	1.2				1.0	17.8 0.8				26.8 1.3 0.6	37.2 1.5 0.5
Bentonite derivative A (Bentone 38) Bentonite derivative B (Bentone 27) Unmodified butylated UF resin (base)	2.5		1.5	Pr	Pr	0.7		1.0		Pr	Pr	1.5	2.0
Epoxide resin—approx. epoxide equivalent 500	39.0		6.5	oprieta	oprieta			3.9	22.4	oprieta	oprieta	6.1	8.3
Cocondensate of bisphenol A and epichlorohydrin—high mol. wt. Epoxide resin/DCO ester (55% fatty acids)		61.3		ary primer A	ary primer B	4.0	6.7			ry primer C	ry primer D		
Cobalt naphthenate (6% cobalt) Curing agents Amine adduct of epoxide resin Reactive polyamide—approx. amine	20.1	0.1					•						
value 90			3.8			2.5		2.4	1.11			3.5	4.0

#### Table 2

Component		CEGB7	CEGB8
Heat hardening phenol M.P.48-56 Zinc chromate Asbestine Methylated spirit 64.O.P. Methyl ethyl ketone Toluene Phosphoric acid	c resin 	5.2 9.7 1.4 26.3 19.4 2.4 5.4 25.0 100.0	Composition to Specification DEF.1408 Spraying quality

Composition of etch primers (percentage by weight)

There seems little doubt that a very small particle size zinc dust (2.5-3.0 microns) gives best results in primer coatings and so this grade was used throughout, except in one instance where a leafing zinc flake pigment was used in place of the zinc dust. The zinc flake primer was included in view of the recent general interest in this type of pigment and because of the claims made that the high specific surface area of the flake permits the formulation of zinc rich primers containing 25-30 per cent solid binder which are still capable of providing cathodic protection to bare metal areas. The higher binder content is said to improve the mechanical properties of the primer and to provide a satisfactory surface for overcoating with drying oilbased paints.

It was also well known that "reduced" zinc primers were being used in the marine field, presumably on economic grounds, and it seemed desirable to include a range of paints which contained less zinc dust than normal whilst still being pigmented overall at a high level (92 per cent) on dry film weight. Zinc oxide and a siliceous type extender were used to reduce the zinc dust content.

In the case of CEGB 1, which was intended to be a "true" zinc rich type, the zinc content on dry film weight was 87 per cent instead of the recognised 92 per cent. This happened more by accident than by design, but this mistake has provided interesting information on the effect of zinc content on performance as will be seen later in this paper.

Reference should also be made to CEGB 5. This is a single-pack paint based on a high molecular weight cocondensate of bisphenol A and epichlorohydrin. In the authors' opinion and experience, zinc rich primers based on polystyrene and chlorinated rubber resin (normal single-pack types) do not perform as well in many respects as do the cold cured epoxy types. However, there are

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obvious advantages in using a single-pack material and if such a paint could be produced it would be attractive to the user industry. The particular epoxy resin used in CEGB 5 produces, by solvent evaporation alone, films which, it is claimed, have excellent adhesion, hardness, flexibility and chemical resistance.

#### Aluminium primers

Aluminium primers were not included in the original series, but subsequent discussion with interested parties in the industry indicated that such paints were being used as prefabrication primers and had certain advantages in respect of overcoating. Again it was decided to confine attention to epoxy resin binders and the choice of an epoxy resin ester and an amine adduct cured epoxy resin appeared to represent fairly the possible variations and combinations.

#### Etch primers

Finally, etch primer formulations were considered and, to avoid using proprietary formulations, the main choice was the widely used two-pack type described in specification DEF 1408. However, the authors had experience of a single-pack etch primer which contains zinc chromate and is reinforced with a phenolic resin. Because of the user advantages to be obtained with a single-pack paint, it was decided to include this paint (CEGB 7) in the trials. Every attempt was made to produce paints which would :

- (a) be suitable for spray application,
- (b) dry within a few minutes of application to facilitate speedy and easy handling of the coated section, and
- (c) store satisfactorily in the container for a reasonable period of time.

Attention was given to the choice of solvents, the inclusion of anti-settling agents and anti-gassing agents in the case of certain of the zinc rich primers. In all instances pigment dispersion was carried out carefully and thoroughly.

Table 3 shows the physical and mechanical properties of the primers included in the tests.

It could be said that some of the zinc primers were comparatively slow drying, but this defect could be rectified by the use of slightly more volatile solvents. Some gassing was experienced with the zinc primers, including two of the four proprietary paints, but subsequent evidence has shown that the more precise use of a micronised silica gel anti-gassing agent controls this undesirable feature, particularly if additional care is taken to ensure that water-free solvents are used. Settlement did not generally present a problem in the zinc rich paints with the use of "Bentone" gellants and small quantities of finely divided silicas, although, for some unknown reason, CEGB 4 provided the exception.

Prefabrication primer	icatio	n prin	ner	Settlement	Gassing	Touch dry time	Impact resistance (concave surface)
EGBI	:	:	:	soft	very slight	5 minutes	good
CEGB4		:	;	hard	fairly severe	15 minutes	good
EGB5	:	:		soft	severe	10 minutes	good
EGB10	:			soft	very slight	15 minutes	very good
EGB6		•	•	soft	fairly severe	4 minutes	fair
EGB15				soft	Severe	4 minutes	poor
EGB16	:	:		soft	slight	4 minutes	poor
EGB17	:	:	:	soft	none	15 minutes	very good
EGB18	:	:	:	none	none	15 minutes	very good
EGB7	:	:	:	soft	none	3 minutes	very good
EGB8	:	•	•	soft	none	3 minutes	very good
EGB2	:		•	soft	moderate	3 minutes	fair
CEGB3			•	hard	none	3 minutes	boog
CEGBII	:	:	:	soft	slight	3 minutes	boog
CEGB12	:	:	;	hard	very severe	3 minutes	fair

Table 3 Properties of paints (five months' storage)

Method No. 17(a), Resistance to Impact (falling weight). I CST 5001 DEF E described method Impact resistance carried out according to the

1966

#### STRUCTURAL STEELWORK

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#### Exposure programme

After the completion of the blast cleaning and prefabrication primer application, the girder sections were transported to the exposure sites without any particular care being taken and, in fact, were handled fairly roughly.

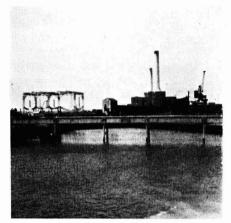


Fig. 1. The exposure site at Brighton Power Station

The main exposure site is at Brighton "A" Power Station, Shoreham, which is considered to be one of the most aggressive sites in the country. The girder sections were laid horizontally on the centre parapet of a cable bridge. The water under the bridge is part of the station cooling system and, although it is sea water, it is not subjected to tidal movement. In Fig. 1, Shoreham Harbour is to the right of the bridge and the open sea can be seen to the left. Brighton "B" Power Station is in the distance with Brighton "A" and the gasworks behind.

Exposure tests of the same type have been carried out at a roof site at Battersea, and flat plates, blast cleaned and treated with prefabrication primer,

have been exposed at Hayling Island. The results of the exposure trials at Hayling Island are not included in this paper since they were merely confirmatory of the results obtained at Battersea and Brighton, the rate of breakdown being intermediate between those obtained at these two main sites.

#### Overpainting

It is important to know which primer should be used in the final paint system to ensure adequate adhesion and provide maximum performance from conventional "decorative" undercoat and gloss which it is desirable to use on power station structures. It has been said by a number of workers in this field that if zinc based prefabrication primers are used to protect blast cleaned steel, they should be overpainted with a chemical resistant coating if blistering and failure by intercoat adhesion are to be avoided. However, these statements generally refer to experience under marine conditions and it was felt that under the less stringent conditions of atmospheric exposure it was possible that the systems in current use in power station painting would behave satisfactorily. Perhaps of more importance is the desirability, on economical grounds, of finding a satisfactory prefabrication primer which will permit the continued use of the existing paint systems.

To complete the exposure programme, each pretreated girder section was marked off into 1 ft lengths and treated as follows :

First 1 ft length — overpainted with appropriate primer two days after surface treatment. A "standard" white undercoat and gloss paint (both alkyd based) were then applied after a further seven days. Second 1 ft length --- painted as above at exposure site two months after surface treatment.

- Third 1 ft length painted as above at exposure site six months after surface treatment.
- Fourth 1 ft length painted as above at exposure site 12 months after surface treatment.

In all instances, except the first, the prefabrication primer treated steel was exposed to the site conditions during the period between surface treatment and the subsequent application of the final paint system. At each painting half of the area of prefabrication primer to be overcoated was washed down with water, otherwise no other surface preparation was carried out, with the exception of the non-treated RSJs, which were, of course, appropriately wire brushed.

It was intended originally to examine the three primers, Red Lead Primer BS 2523, Type B, Zinc Chrome Primer to Specification DEF 1039A, and Calcium Plumbate Primer to BS 3698, Type A, over all of the prefabrication primers, but this was not practical because of the vast number of girder sections involved. As a compromise, the following "standard" primers were applied :

On zinc rich and reduced zinc prefabrication	
primers	— Calcium Plumbate Primer
On aluminium prefabrication primers	— Zinc Chrome Primer
On etch prefabrication primers	— Red Lead Primer

However, on a limited number of sections it was possible to apply further variations so that each primer has been applied over each prefabrication primer on one specimen at least.

It is not our intention to discuss in detail the performance of the various primers over the prefabrication primers in this paper since the maximum exposure of 18 months is not sufficient to show any significant differences.

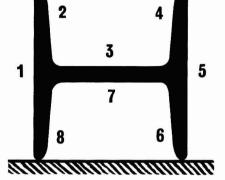
# Assessment of performance of prefabrication primers

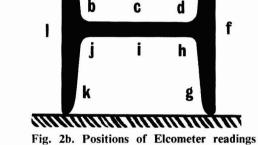
The girder sections exposed at Brighton and Battersea were examined at 6-, 12- and 15-monthly intervals. The performance of the single coat films of the primers was assessed visually and the amount of rust formation recorded as a percentage of the surface area of each "side" of the girder section, the "sides" being numbered one to eight as shown in Fig. 2a. At the same time a colour photograph was taken of each girder at every inspection.

# Results

# Film thickness

It is important to examine the film thickness of the primer coatings since this could have a bearing on the interpretation of the results obtained. The spray application of the prefabrication primers was carried out by an experienced operative, but, even so, a wide variation in film thickness was obtained, presumably due to the shape of the sections. Magnetic thickness gauge (Elcometer) measurements were taken on each section after the prefabrication primers had been allowed to harden thoroughly, but before exposure commenced. Twelve measurements were taken on each coated section and





a

Fig. 2a. Numbering of sides for rust assessment

the points indicated on Fig. 2b and these show that the central area of the horizontal surfaces received a comparatively thick coating, whilst the internal vertical surfaces were coated with films of a thickness normally prescribed for these types of primers. Table 4 gives the results obtained on 21 specimens together with the mean average film thickness measurements.

Sample No.	Α	В	С	D	E	F	G	н	ſ	J	ĸ	L	Mean (12)
IA	0.3	0.8	1.8	1.6	0.4	1.0	0.4	1.3	1.8	1.2	0.4	1.4	1.0
1C	0.7	1.4	3.0	1.8	1.0	2.4	1.0	0.7	3.2	3.0	1.3	1.7	1.8
2A	0.5	0.8	1.4	1.0	0.8	1.3	0.8	2.0	2.2	2.4	0.4	1.4	1.25
2A 2C	0.7	1.6	2.6	1.6	0.8	2.4	1.0	1.3	2.0	1.8	1.7	2.0	1.6
3A	0.7	1.4	1.7	1.0	0.4	1.0	0.5	1.4	1.4	1.4	0.2	1.5	1.05
3C	1.5	1.4	2.0	1.4	1.2	3.1	1.5	2.8	2.5	0.8	2.1	1.8	1.8
4A	0.3	.1.4	2.8	2.0	1.4	1.8	0.6	1.6	2.0	2.2	1.1	2.1	1.6
4C	0.7	0.5	3.6	3.0	1.1	3.8	0.1	2.5	3.9	1.4	1.7	3.5	2.15
6A	0.2	0.2	1.1	0.9	0.1	1.3	1.0	0.2	1.5	1.3	0.2	1.0	0.75
6C	0.8	1.0	1.7	1.8	2.0	1.5	1.6	0.1	3.4	1.5	0.8	1.5	1.5
7A	0.2	1.5	1.9	1.0	0.1	1.9	0.8	0.1	1.6	0.8	0.3	1.3	1.0
7C	3.7	2.1	2.1	0.9	0.3	1.3	0.1	0.2	1.5	0.9	0.4	1.2	1.0
8A	0.1	0.1	0.7	0.1	0.4	1.3	0.1	0.4	1.9	0.9	0.3	1.0	0.6
8C	0.8	0.3	1.0	0.3	0.1	0.9	1.1	0.3	1.5	1.4	1.1	1.5	0.9
9A	0.4	0.2	1.3	1.2	1.5	1.5	0.6	0.1	1.4	0.1	0.9	1.1	0.9
10A	0.2	0.1	2.0	0.2	0.7	1.5	0.7	0.2	2.0	1.2	1.5	1.7	1.0
10C	3.5	1.4	3.5	3.5	0.3	1.7	1.1	0.3	0.9	1.3	1.0	1.9	1.5
11A	0.8	0.5	2.7	2.1	1.5	1.4	1.4	0.4	2.5	0.8	1.5	1.6	1.4
11C	1.7	0.6	3.8	1.7	1.6	2.2	0.8	0.1	2.3	0.7	0.8	2.5	1.6
12A	0.1	0.5	2.5	0.7	1.1	1.6	1.4	0.3	1.9	0.7	0.8	1.6	1.1
12C	0.5	0.4	2.0	2.1	0.9	0.9	0.9	0.3	1.9	0.8	1.1	1.6	1.1
Aean (21)	0.9	0.9	2.2	1.4	0.8	1.7	0.8	0.8	2.1	1.3	0.9	1.7	1.3

Table 4Elecometer readings in inches  $\times 10^{-3}$ 

Film thickness measurements by the Elcometer, particularly on blast cleaned surfaces, can be inaccurate and so the results obtained were compared with measurements taken by projection microscope (magnification  $300 \times$ ) on prepared cross-section specimens taken from the girders, as in the following example :

	Sample	No. of determinations	$Mean \\ (in \times 10^{-3})$	Standard deviation (in×10 <sup>-3</sup> )
Magnetic gauge (Elcometer)	6A	50	1.3	0.5
Projection microscope	$ \begin{array}{c} 6A & top \\ 6A \\ bottom \end{array} \right\} $	50	1.0	0.6

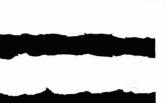
Whilst the results are not exactly similar, they confirm that the magnetic gauge readings were of the right order and, in any case, indicate thick and thin areas. The comparison of sets of figures from a number of specimens show the Elcometer measurements to be generally slightly higher than the projection microscope results, but the latter were taken from a much smaller area.

The average film thickness of the prefabrication primers on the internal vertical surfaces was  $0.8 \times 10^{-3}$  in and is relative to the rusting assessment of sides 2, 4, 6 and 8, but particularly to sides 2 and 4, which have been fully exposed to the weather. The average film thickness of the horizontal (centre) surfaces was  $2.15 \times 10^{-3}$  in and relates to the rusting assessment of sides 3 and 7, but particularly side 3, which has been fully exposed to the weather. It is interesting to compare the performance of a selection of zinc primers in relation to film thickness (see Table 5). As might be expected, in general the level of rust formation is inversely proportional to film thickness, but increased film build does not affect the order of merit of performance, i.e. the poorer primers still behave badly when applied in thick films.

#### Blast cleaning

It has been stated previously that different methods of blast cleaning were used to obtain different levels of surface cleanliness and it is desirable to determine the effect of surface preparation on the performance of the prefabrication primers, both individually and collectively. When cross-sections taken from girders are viewed on the projection microscope, a 6 in wide image is obtained on a screen. By moving the specimen along and tracing the surface outline on to paper of 3 ft lengths an actual surface profile is recorded. This recorded profile represents 10 in of surface only. Whilst this appears to be very small, it is 100 times greater than the paint film thickness. The shaded area of the projections is the metal. Obviously this has been condensed otherwise the actual  $\frac{1}{2}$  in of metal would have to be shown at a thickness of  $12\frac{1}{2}$  ft. However, the surface profiles and the paint films are to scale and represent the top and bottom faces of the specimen. Even though the sample area is extremely small, the isolated surfaces of each specimen have similar profiles, therefore it is reasonable to assume that they are representative of the whole area. A general examination of the profile shows that shot blasting gives a smoother finish than grit blasting, which is not unexpected.

The commercial shot blasting gives the smoothest finish (Fig. 3) although not greatly different from the white metal finish achieved by this method of surface cleaning (Fig. 4). Grit blasting to white metal gives the roughest finish (Fig. 5) with the commercial finish by this method being slightly smoother (Fig. 6).



PRIMER 2-SHOT BLAST-COMMERCIAL

PRIMER 1-SHOT BLAST - COMMERCIAL

PRIMER 3-SHOT BLAST-COMMERCIAL

PRIMER 11- SHOT BLAST-COMMERCIAL

PRIMER 6-SHOT BLAST-COMMERCIAL



PRIMER 7- SHOT BLAST-COMMERCIAL

FIG. 3

PRIMER 1-SHOT BLAST-WHITE METAL

PRIMER 2-SHOT BLAST-WHITE METAL

PRIMER 3-SHOT BLAST-WHITE METAL

PRIMER 11-SHOT BLAST-WHITE METAL

PRIMER 6-SHOT BLAST-WHITE METAL

PRIMER 7-SHOT BLAST-WHITE METAL

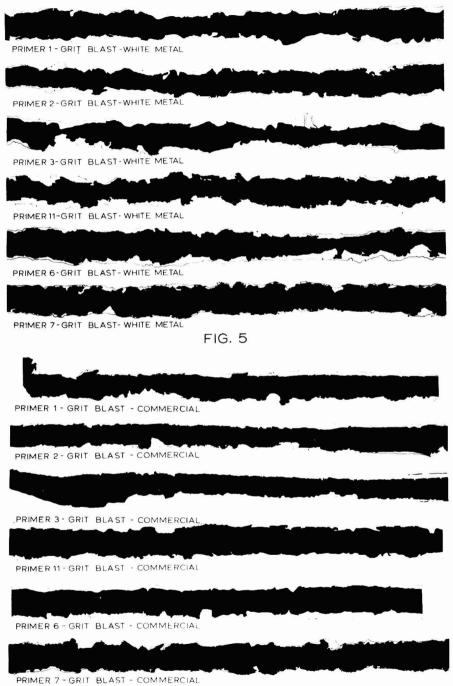


FIG. 6

Table 5	<b>3RIGHTON SITE 12 MONTHS' EXPOSURE</b>	Performance of primers in relation to film thickness	
	BRIGHTON	Performance of	

Prefabrication primer	Section		Rus	ting as per	centage c	Rusting as percentage of surface area	
		Thin 6	Thin coating $(0.8 \times 10^{-3} \text{ in})$	× 10 <sup>-3</sup> in		Thick coating	Thick coating (2.15 $\times$ 10 <sup>-3</sup> in)
	1	Side 2	Side 4	Average	age	Side 3	Average
CEGBI	A	66	100	Side 2		70	
	шUД	90 00 90 00	80 <sup>000</sup>	82	95	30 20	52
CEGB2	4muQ	5 10 10	10 140 100 100	9	47	none none 5	none
CEGB4	AGOD	none 25 100 95	40 100e 5	55	36	none 10 5	Q
CEGB6	4muD	<u>8</u> 282	5 100 90 90	80	50	30 100 2000 2000	62

# D. A. BAYLISS ET AL.

	t on primer performance
9	uo l
Table	reatmen
	surface 1
	of.
	Effect

Rusting as percentage of surface area	Shot blasting	ish ". White " metal Commercial finish	Battersea Brighton Battersea Brighton Battersea	46 42 45 42 21	5 3 16 2	40 15 14 14	51	47 70	49	85 21	73 38 81	52 60 46 59 51	55 35 50			
	Grit blasting	Commercial finish	Brighton	16	6	4	74	70	70	16	70	51	59			
	Gri	Gr	Gr	" White " metal	Battersea	61	10	7	Ξ	-	24	99	50	43	30	>
		" Whit	Brighton	09	7	10	Π	70	60	96	72	54	48	2		
L				:	:	:	:		:	:		:		:		
prime				:	:	:	:	:	:	:	:	:				
cation				:	:	:	:		:	:	:	:		:		
Prefabrication primer				CEGB1	CEGB2	CEGB4	CEGB6	CEGB7	CEGB8	CEGB10	CEGBII	CEGB12	Mean			

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Table 6 gives a comparison of the effect of surface treatment on primer performance which has been made by determining the rust formation on each girder section as a percentage of the surface area after 12 months' exposure at both Brighton and Battersea. There are a few discrepancies between individual primers, but, taking the mean at Brighton as a guide, it can be seen that there is little significant difference between the four surface treatments :

White metal grit blasting	•••		48 per cent
Commercial finish grit blasting			59 per cent
White metal shot blasting	••	• •	55 per cent
Commercial finish shot blasting	• •		50 per cent

At the less aggressive Battersea site, the commercial finish of shot blasting has given the most favourable results except in the case of etch primers, which have performed at least as well on the grit blasted surface as on the shot blasted surfaces.

## Rusting assessment after various periods of exposure

The rusting assessments made after exposure periods of six months, 12 months and 15 months are presented in tabular form, together with graphical summaries to obtain a general picture of the anticorrosive properties of the prefabrication primers examined. Rust formation is expressed as an average percentage of the surface area of all the exposed RSJ sections for each primer, the mean figure for each site being obtained from at least 32 sides.

Fig. 7 is a graphical representation of the average rusting figures after six months' exposure. The left hand column for each primer represents exposure at Battersea and the right hand column that at Brighton. The zinc rich, reduced zinc above 74 per cent zinc, the proprietary primers and the etch primers were all in very good condition after this period of weathering. However, the aluminium primers and the reduced zinc primers below 74 per cent zinc content were already showing a 50 per cent rusting, even at Battersea. The rust formation for all "sides" of the girder sections for the original series of primers have been assembled in table form and the mean averages determined for the six months' and 12 months' periods (Tables 7-16, pp. 792-801).

After 12 months' exposure only the primers containing a minimum of 91.5 per cent zinc on the dry film weight are in reasonably good condition, that is, showing less than 10 per cent rust formation. Even when the zinc content on the dry film is allowed to fall only slightly below the theoretically required figure of 92 per cent, there is a marked deterioration in performance. (See Table 7 for Primer 1.)

It is interesting to observe the performance of the proprietary primers in the series. The formulations were not disclosed, but by analysis and by results they could be placed in the following order :

Primer 2 (Table 8) appeared to have a minimum of 92 per cent zinc on the dried film and was similar in result to CEGB 4 (Table 10).

Primer 3 (Table 9) had less than 92 per cent, but not below 85 per cent zinc on the dry film weight and appeared similar to CEGB 1 (Table 7).

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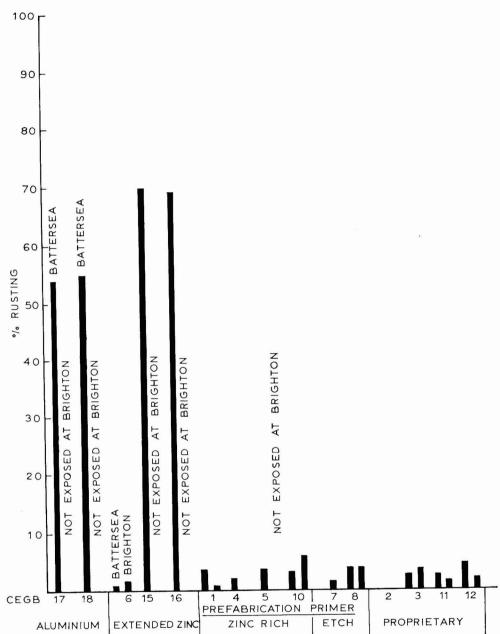
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Primers 11 and 12 (Tables 8 and 9) had approximately 65 per cent zinc on the dry film and behaved similarly to CEGB 6 (Table 11).

The zinc flake primer was included in the trials in view of the considerable interest in this type of pigment at the time at which the exposure programme was designed. After 12 months at Brighton the sections were rusting badly (Table 14). However, it was subsequently found that due to an error in the originally suggested formulation, the zinc content was low and this probably reduced the effectiveness of this particular test.

The two-pack etch primer, after 12 months' weathering period, showed an average rust formation 40 per cent of the surface area at Battersea and 70 per cent at Brighton, but the one-pack type performed extremely well at Battersea after 12 months, showing less than 20 per cent rusting. At Brighton it was estimated, after 12 months, to have an overall rusting of 70 per cent of the surface area, which is approximately equivalent to a number of the zinc rich primers, but, due to the colour of this particular primer, the assessment was difficult to make. Certainly the overall rusting was light in nature.

A summary of the 12 months' exposure results is shown in graphical form in Fig. 8.

After 15 months' exposure at Brighton all the primers were failing, and even the full zinc rich primers 2 and 4, which gave the best all round performance, had an average of over 40 per cent rusting. Perhaps what was more significant was that in places the rusting tended to be of a pitting nature (Fig. 9) and more severe than the overall light rusting present on the single-pack etch primer CEGB 7.

At Battersea, where the exposure conditions were not so severe, the single-pack etch primer was still affording good protection to the steel after 15 months. The condition of the zinc rich primers was similarly better, but there was still a reasonable percentage of heavily rusted areas. Fig. 10 shows the side face of a girder primed with a single-pack etch primer compared with that surface primed with zinc rich primer CEGB 4.

On the underneath surfaces which were not exposed directly to the weather the performance of the holding primer was good in all cases, but on the zinc rich primers there were deposits of zinc corrosion salts which were even heavier on the extended zinc primers (Fig. 11).

# Accelerated weathering

Attempts were made to reproduce by accelerated means the results obtained from the natural exposures. The tests included accelerated weathering and exposure to salt spray, humidity, sulphur dioxide and ammonium sulphate. In practically each case it was possible to distinguish an order of merit for the zinc containing primers, but not possible to relate the performance of any of these primers with that of the etch or aluminium primers.

#### **Discussion of results**

From the results of this work it is obvious that there are a number of primers which will "hold" blast cleaned steel in a rust free condition on even the most aggressive site for a period of six months. However, longer periods of exposure result in some breakdown with all the primers examined, particularly at film thicknesses below  $10^{-3}$  in. It is assumed that it is important not to exceed a film thickness of  $10^{-3}$  in with any prefabrication primer to minimise adverse effects on welding, friction grip surfaces and to maintain cost at the lowest possible level. In the opinion of the authors it is impractical to apply by spray a uniform coating of zinc containing primers on to large complicated

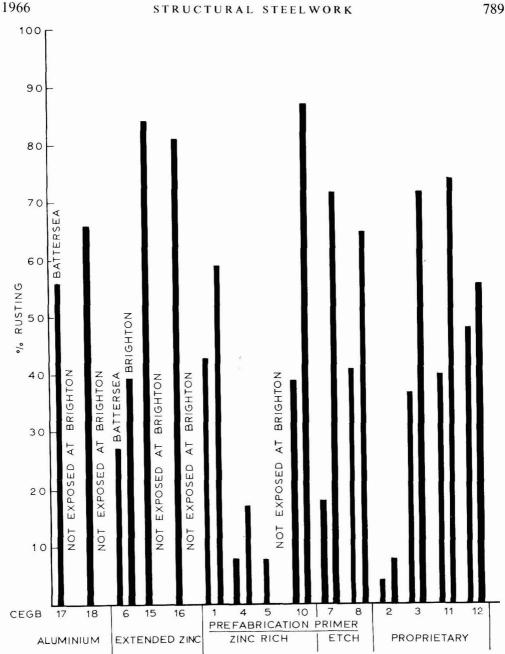


Fig. 8. 12 months weathering. Anti-corrosive properties

areas as met with in practice. On small flat surfaces it is very easy to exceed a thickness of 10<sup>-3</sup> in and it is suggested that this is the reason why some test work on flat plates has shown that even reduced zinc primers give satisfactory protection to the steel for long periods of time.

Fig. 9. Exposure at Brighton after 15 months; zinc rich primer showing rusting of a pitting nature

Fig. 10. Single-pack etch primer (right) compared to zinc rich primer CEGB 4. Exposure at Battersea after 15 months

Fig. 11. Underneath surface with zinc rich primer showing zinc corrosion salts The durability of zinc based primers depends largely on the zinc content, but even the primers examined in this programme with a minimum of 91.5 per cent zinc on the dry film weight had approximately 40 per cent rusting after 15 months' exposure. In addition the isolated areas of rusting of steel coated with even the best zinc rich primer were heavier and more aggressive to the base metal than the overall rusting obtained with etch primer for example. If these rusted areas are not to be the weak link in the protective system they would need to be cleaned and prepared for overpainting, but, at the same time, the surrounding zinc primer coated areas may also need cleaning and preparing. The methods employed for cleaning these two different surface conditions and the types of following-on primers required to give the best performance on each surface are not necessarily compatible.

# Conclusions

There is doubt of the desirability of using zinc containing primers on power station structures where steel coated with prefabrication holding primer is likely to stand on site for periods well in excess of 12 months before being overpainted. Since there is no primer type which will provide overall protection for the periods of time involved, one could question the desirability of blast cleaning at all. However, the non-uniformity of the weathering effect on millscaled steel inevitably presents an inadequate surface for subsequent painting. There is undoubted merit in the use of blast cleaning, even to a commercial grade, to remove millscale, and, in the authors' opinion, the single-pack etch primer of the type used in this work will give many advantages when applied to this surface.

Advantages of one-pack etch primers over zinc rich types

- 1. They are cheaper. Assuming similar coverage rates then the ratio of cost, considering raw materials only, would be 3 : 1, full zinc rich primer to one-pack etch primer.
- 2. They are one-pack materials with very obvious practical advantages to the applicator.
- 3. They will provide good protection for six months at any site and for 12 months at a protected site.
- 4. They give similar corrosion protection on both a comparatively rough grit blasted surface and a smooth shot blasted surface.
- 5. The eventual rusting of the coated steel is uniform and light and can be easily prepared by the traditional wire brushing. Practical tests demonstrate that a well weathered etch primed surface can be properly prepared for painting three times quicker than a steel surface which has not been blast cleaned but which has weathered naturally for the same period of time.
- 6. It is difficult to apply etch primers in thick films and this would reduce considerably the practical possibilities of the paint film having an adverse effect on welding.
- 7. To date the one-pack etch primer has presented no problem with regard to overcoating. Although it is at an early stage yet there are already some indications from this particular exposure programme that the weathered zinc rich primers are not ideal surfaces to receive subsequent paint systems of the types normally used in power station work.

	total 12 months		480	490	730	3/0	335	005	335
Rusting as percentage of surface area	total 6 months	15	80	10	30	none	20	none	S
of sur	8		80	80	100	09	20		
itage	2		10	30	10	30	5		01
ercen	9		25	90	100	20	20		v
g as p	5		90	20	100		10	DC DC	75
usting	4		100	90		100		8	80
R	3	10	70	80	90 10	08	30	00	20 10
	7	S	90 5	90 2	100	09	50	3	90 40
	Side 1		50	10 5	100	20	100	00	70 5
Surface Preparation		Grit blast " white " metal	: :	Grit blast	commercial	Shot blast	white metal	Shot blast	commercial
Exposure		6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	6 months	12 months 6 months 12 months
Site		Brighton	Battersea	Brighton	Battersea	Brighton	Battersea	Brighton	Battersea
		:	:	÷	:	:	:	:	:
Section		:	•	:		:	:	:	
S		IA .	IA .	B	1B.	U.	J.	1D	D

Table 7

Rusting assessment Primer CEGBI (Epoxy/polyamide 87 per cent Zn content on dry film weight)

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		total 12 months		15	80	70	01	2	40	25	125	20
	Rusting as percentage of surface area	total 6 months	none	none	none		none	5	eucu	none	v	)
	of sur	8			10					S		
	Itage	7			10		Water			S		5
	ercen	9	-		10							10
	g as p	S				10					10	
	ustin	4		10	20	40		S	40	10	100	S
	R	б			10		Ŷ	)		5	Ś	2
•		5		Ś	10	10	Ŷ	3			10	
		Side 1			10	10						
	Surface Preparation		Grit blast " white " metal	: :	Grit blast	commercial "	::	Shot blast	;	 Shot blast	commercial	5 5
	Exposure		6 months	12 months 6 months	12 months 6 months	12 months	6 months	6 months	12 months	12 months 6 months	12 months 6 months	12 months
	Site		Brighton	Battersea	Brighton		Battersea	Brighton	Battersea	Brighton	Battersea	
	c	(ma m) -	:	:	:		÷	:		: :		
	Section		:	:	:		:	:		: :		
	S		2A	2A	2B		2B	2C	30	2D	2D	Ì

 Table 8

 Rusting assessment Primer CEGB2 (Proprietary primer A)

	total 12 months		680	660	565	440	490	00	not recorded 45
Rusting as percentage of surface area	total 6 months	30	15	45	45	40	10	25	15
of sur	8	10	100	100	95	10		10	
tage	2	10	20	5 5	10	202	5	10	10
ercen	9	10	95	100	Ś	50	100	10	ŝ
g as p	S		100	100		90	50		ded
usting	4		100	100	5	80	100		not recorded 10
R	m		5 80	30	70 5	50		<u>c</u>	not 10
	10		95	30	100	50	100	S	20
	Side 1		95	100	80	90	100		
Surface Preparation		Grit blast " white " metal	: :	Grit blast	: :	Shot blast	WIIIC 111CL01	Shot blast	
Exposure		6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	6 months	<ul><li>12 months</li><li>6 months</li><li>12 months</li></ul>
Site		Brighton	Battersea	Brighton	Battersea	Brighton	Battersea	Brighton	Battersea
E		:	:	:	:	:	:	:	:
Section		3A	3A	3B	3B	3C	3C	3D	3D

 Table 9
 Rusting assessment Primer CEGB3 (Proprietary primer B)

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Table 10	Rusting assessment Primer CEGB4 (Epoxy/isocyanate 92.5 per cent Zn content on dry film weight)	Rusting as narcentage of surface area
	CEGB4 (Epoxy/isc	Cumford
	essment Primer	E
	Rusting ass	0.55

Section	Site	Exposure	Surface Preparation			R	usting	as pe	srcent	age o	Rusting as percentage of surface area	
				Side 1	7	e	4	s	9	1	8 total 6 months	total s 12 months
÷	Brighton	6 months	Grit blast " white " metal						S		5	
		12 months	:				80					80
:	Battersea	6 months	: :		10	S					попе	15
:	Brighton	6 months	Grit blast								none	
		12 months			25	10						35
:	Battersea	6 months 12 months	: :		S	Ś					none	01
:	Brighton	6 months	Shot blast " white " metal		S		S			-	10	
:	Battersea	12 months 6 months	: :	S	2°2	502	202	8			5 75	320
:	Brighton	12 months 6 months	Shot blast		10	8	2v				5 5	17
		12 months			95	5	S	10				115
:	Battersea	6 months	: :	S	6	10		\$				110

	metal)
	cent
	per
	74
	d zinc 74 per cent n
	reduced
Table 11	(Epoxy/polyamide
	CEGB6
	primer
	assessment
	00

Rusting as percentage of surface area	3         4         5         6         7         8         total         total           6         6         7         8         6         12         10	none	30 5 20 10 90	30         5         20         10         10         90           20         10         10         50         90	100 100 100 80 10 10 590	40 40 10 10 250 none 250	20 10	90 100 20 50 40 none 410	20 90 20 230
	8		20	1020	100	100		10	100
	Side 1		S	S	90	50	100	100	
Surface Preparation		Grit blast " white " metal	:	". Grit blast	commercial	". Shot blast	" white " metal "	Shot blast	commercial
Exposure		6 months	12 months	6 months	12 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months
Site		Brighton	Rattarcan	Brighton	Dottorion	Brighton	Battersea	Brighton	Battersea
Section		6A	44	6B	¢D	6C : :	6C ::	6D	()

Exposure Surface Rusting as percentage of surface area Preparation	Side 12345678totaltotal6 months12 months	6 months Grit blast general comment no rusting "white " metal	12 months general comment 70 per cent rusting none	12 months	12 months 6 months	12 months 5 5 5 5 30 5 5 5 60 60 months Shot blast 5 5 5 30 5 10 20 30 60	12 months 6 months	6 months Shot blast general comment no rusting	12 months commercial general comment 70 per cent rusting none
Exposure				6 months	12 months 6 months			6 months	12 months 6 months
Site		Brighton	Battersea	Brighton	Battersea	Brighton	Battersea	Brighton	Battersea
Section		7A	7A	7B	7B	7C	7C	7D	()

 Table 12
 Rusting assessment primer CEGB7 (one pack etch primer)

1966

		total 12 months			190		250		060	490
	Rusting as percentage of surface area	total 6 months	40	5	20	25	20	none	30	80
	of su	~	10	50	30	ŝ		50	2	80 IO
	ıtage	7	10	rustin	40	usting 5	10	usting	3	usting 10 40
mer)	percei	9	10	general comment 60 per cent rusting	30	general comment 70 per cent rusting	0 50 70 30 20 10 10 20	ent r	)ç	to the sent r 10 40
ch pri	g as I	s		0 per	20 20	ber o	30	per o	2	per 10 60
ck eta	tustin	4	S	ent 6(	50	int 70	10	int 70	15	nt 60 10 70
<i>чо ра</i>	R	m		omm 5	20 20 20	5	50	omme	<del>1</del> 2	10 70
B8 (t1	-	7	S	eral c	20	eral co	10	eral co	R	ral co 10 80
ner CEG		Side 1		gen	10	gene	10	gene	00	general comment 60 per cent rusting           10         10         10         10         10         10           50         80         70         70         60         40         40         80
Rusting assessment primer CEGB8 (two pack etch primer)	Surface Preparation		Grit blast " white " metal	: :	Grit blast	, ,	Shot blast	*	Shot blast	
Rus	Exposure		6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	6 months	12 months 6 months 12 months
	Site		Brighton	Battersea	Brighton	Battersea	Brighton	Battersea	Brighton	Battersea
	Section		:	:	:	:	:	:	:	:
	Š		8A .	. <b>A</b> 8	8B .	8B .	8C .	8C .	8D .	8D

Table 13

Table 14	Rusting assessment CEGB10 (epoxy/polyamide zinc flake primer)
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Section	Site	Exposure	Surface Preparation			К	ustin	g as p	ercen	tage	of sur	Rusting as percentage of surface area	
				Side 1	7	m	4	w	9	2	~	total 6 months	total 12 months
10A	. Brighton	6 months	Grit blast	20	10	10	10	20	20	5	10	105	
10.4	Rattersea	12 months	wille 1110-041	100	100	100	100	100	100	70	100	02	770
10B	. Brighton	12 months 6 months	". Grit blast	90	30 80	80	88 v	30	1020	30	80	55	530
10B	. Battersea	12 months 6 months	commercial	100	100	100	100	100	100	30	100	30	730
10C	. Brighton	12 months 6 months	Shot blast	70	10 80	5	90	60	30	60	80	25	540
10C	Battersea	12 months 6 months	willC IIICIAL	100					100	20	60	none	680
10D	. Brighton	6 months	Shot blast	70	9	05	<u></u>	2				none	1/0
10D	Battersea	12 months 6 months 12 months	, , , , , , , , , , , , , , , , , , ,	50	100	75	75	90	100	SUN	80 5	5	610 10
					•						a na		

STRUCTURAL STEELWORK

		total 12 months		575	400		555	420		585	305		650	155
	Rusting as percentage of surface area	total 6 months	20	10		70	20	,	10	45		none	none	
	of sur	×		80	10		10 20	100		100	30		95	10
	tage (	7		S	10		10			20				S
	ercen	9		90	90		6	80	10	50	5		95	20
	g as p	S		90	40		100	10		100	10		100	10
er C)	usting	4	20	90	09		95	100		100	80		100	70
prim	R	3		20	6		50	50		<b>4</b> 0	70		60	30
etary		2		100	86	<b>N</b> 7	100	60		30	100		100	10
II (propri		Side 1		100	60		60	20		75	10		100	
Rusting assessment CEGB11 (proprietary primer C)	Surface Preparation		Grit blast " white " metal	:	:: :: ::: :::	commercial	:		Shot blast " white " metal	: 1	: : :	Shot blast commercial	:	: :
Rusting	Exposure		6 months	12 months 6 months	12 months		12 months 6 months	12 months	6 months	12 months 6 months	12 months	6 months	12 months 6 months	12 months
	Site		Brighton	Rattersea	Duichton	prignion	Battersea		Brighton	Battersea		Brighton	Rattersea	
	Section		11A	114			11B		11C	11C				:

Table 15 EGB11 (nronrietary primer C) t

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	total 12 months		435	345	410	425	480	370	475	405
Rusting as percentage of surface area	total 6 months	10	enon	15	35	35	120	none	v	C
of sur	œ		S	50	10	80		20	2	30
ıtage	7							S	v	ראי ר
bercer	6		S	Ś	S	S	10	Ś	10	. 30
g as p	s		75	80	60	10	100	20	95	10
ustin	4		100	100	100	20	<u>100</u>	100	100	100
R	m		75	20 5	40	70 5	10	60	70	70
	7	10	95	60	95 20	100	100	80	100	100
	Side 1		80	30	100	60	100	80	95	60
Surface Preparation		Grit blast " white " metal		". Grit blast commercial	, ,	Shot blast	WIIIC 1110141	Shot blast	commercial	: :
Exposure		6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months 6 months	12 months	12 months
Site		Brighton	Battersea	Brighton	Battersea	Brighton	Battersea	Brighton	Rattersea	
-		:	;	: :	:	:	:	÷	1	;
Section		12A	12A	12B	12B	12C	12C	12D	12D	

Table 16Rusting assessment CEGB12 (proprietary primer D)

1966

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Acknowledgments

The authors wish to thank their colleagues in the CEGB and Smith & Walton Limited for their help, in particular Mr. M. J. Baillieu and Mr. K. J. Harden. They also thank the Director of the SE Region CEGB and the Directors of the Wall Paper Manufacturers Limited respectively for their permission to publish the paper. Thanks are also due to the South Durham Steel & Iron Company Limited for the use of their automatic blast cleaning plant at Stockton-on-Tees.

[Received 5 April 1966

#### Discussion

The first question was on the method of assessment of rusted areas, to which the reply was that it was visual.

MR. D. S. NEWTON said that he would have expected increased corrosion in cool moist conditions and therefore that the underside (facet No. 7) of the girder would rust more than the upper side (facet No. 3). Dr. J. B. Harrison had found this in his girder trials.

THE AUTHORS replied that in fact facet No. 3 rusted more rapidly than facet No. 7 and with more uniform corrosion than the latter, which became spotty.

MR. NEWTON asked whether this were perhaps due to acidic fall-out, rather than purely vapour-phase corrosive agents.

THE AUTHORS agreed and said that Dr. Harrison's girders were propped up against a wall and therefore did not present a horizontal surface.

MR. E. L. FARROW asked about the effect of washing down the prefabrication primers, especially those exposed at Brighton.

THE AUTHORS said that, although the exposures had only been for 18 months, the conventional paints were already showing an alarming lack of adhesion to zinc primers and washing had had little effect.

DR. T. A. BANFIELD remarked on the reported high percentages of rusting and thought that much of it might be rust staining of the paint surface rather than rusting of the metal; he asked whether the film detached from rusty metal. Further, he pointed out—on the subject of cost—that etch primers were cheaper than zinc primers per gallon, but not when calculated per square yard to the same dry film thickness.

THE AUTHORS replied that the type of rusting was hard to assess with the etch primers on account of the small colour differences. With these primers rust was formed "gently" and if brushed off it revealed primer underneath.

On the matter of cost, MR. BAYLISS said that the primers had been applied at similar spreadage rates and therefore the cots per unit area would be relative to the cost per gallon. He appealed to MR. R. P. HANSON, who said he had recently conducted a questionnaire to paint manufacturers on the subject of shop primers on behalf of his Company (Swan, Hunter & Wigham Richardson Ltd.). The solids content by volume of a primer affected its true cost greatly and other primers of higher solid-content had been found cheaper than the wash-primer types.

MR. HANSON remarked that the weathering period for etch primers had been found to be less on shot- than on grit-blasted surfaces and asked why. THE AUTHORS could not explain this.

MR. HANSON said that this was unexpected since the angular abrasives might produce a profile of  $8 \times 10^{-3}$  in amplitude, compared with  $2 \times 10^{-3}$  in for round steel shot.

#### JOCCA

THE AUTHORS replied that the performances of the schemes had not varied significantly on any of the surfaces used. The effect of a surface profile might be expected to relate to the number of "peaks" penetrating the paint film; the etch primers had more such peaks than the rest, but rusted less.

MR. J. C. LION said that zinc-epoxy primers had the ability to protect steel in the vicinity of scratches in the paint film, and asked whether the specimens had had rough enough treatment to bring out this important practical advantage.

THE AUTHORS said that the treatment was pretty rough. They were thrown on to a lorry at Stockton, thrown off again at their destinations, and all the samples at Brighton were on one occasion thrown off a roof on to the earth. But in any case serious mechanical damage was most likely to result in a comparatively large bare areas across the paint surface rather than a scratch. Cathodic protection could only be effective within fractions of an inch from the edges of damaged areas.

MR. D. P. DODGSON commented on the surprising difference between paints with high zinc contents and those slightly lower.

THE AUTHORS agreed, but pointed out that an "extended-zinc" paint with a zinc content of 74.5 per cent and a total pigment content of 92 per cent behaved as well as a pure zinc paint containing 88 per cent of zinc, so the total pigment loading also had an effect.

MR. K. F. BAXTER asked whether the one-pack etch primer was used fresh or after ageing.

THE AUTHORS said that it was aged a little while before use. No tests on the effect of ageing were done in this series, but previous tests had shown three or six months' ageing to have no effect.

MR. E. A. DULIGAL asked whether Elcometer and microscopic readings of film thickness agreed on all parts of the girders; he would have expected magnetic shielding on the enclosed sections.

In general he commented that the authors' condemnation of zinc primers ran contrary to the experience of constructional engineers and shipyards.

THE AUTHORS replied that to avoid the magnetic effects the Elcometer readings were done on 1 in long sections cut from the ends of the girders.

Regarding experience, they said that conditions in power station constructions were different from those in shipyards.

In reply to a question about the uniformity of paint film thickness, THE AUTHORS said that the rusting was uniform over each side of the girders, which suggested that the films were uniform too.

MR. D. M. JAMES asked about differences in the abilities of different paints to follow the contours of grit-blasted steel. His laboratory had found that etch primers were better than zinc-epoxies in this respect and this would help to maintain their performance at low overall film thicknesses. Had the authors found differences in the order of ranking primers at different film thicknesses?

THE AUTHORS agreed with the observation on etch primers and commented that differences were observed at different film thicknesses, but ultimately film thickness had no effect on the order of ranking the primers.

MR. D. S. NEWTON referred to experiments he had done on extending zinc dust with colloidal zinc oxide and asbestine : he had found the best results to be near the critical PVC.

THE AUTHORS thanked him for this suggestion, which they would consider in future work.

MR. K. F. BAXTER asked if there were significant differences in the performance of zinc primers based on different media.

THE AUTHORS replied that the effect of zinc-content was overwhelming. However, accelerated tests did differentiate between media and showed isocyanate-cured epoxies to be better than polyamide-cured.

MR. E. L. FARROW asked whether these were alkanolamine adducts of epoxy resins cured with isocyanates; THE AUTHORS replied that they were not, a simple solid epoxy resin was used (Epikote 1007).

# Next month's issue

The Honorary Editor has accepted the following papers for publication and these are expected to appear in the October issue of the *Journal*:

- "Texture of pigments," by W. Carr.
- " Influence of particle size and particle form of inorganic pigments on change of shade in coloured paints and lacquers," by P. Kresse.
- "Recent advances in the chemistry of fatty acids," by M. E. Ansell.

"The interaction between paint and substrate," by J. van Loon.

# Correspondence

# The varnish holding properties of timber

SIR,—The above paper by the late Mr. K. L. Jones, published in the April 1966 issue of *JOCCA* (pp. 314-339), raised a very pertinent issue which perhaps can be best expressed in a query :

"Can a statistical analysis of the behaviour of grouped types of a heterogeneous material such as timber give results of value in their reactions to protective coatings such as the varnishes described in the paper?"

That is the widest approach possible to the author's problem, which is, however, further complicated by his random selection of groups of 12 species of wood examined (not 16 as stated in the summary), and of these two unidentified—this, however, is a minor fault for which the taxonomists are perhaps most to blame.

The acid test of any study of this nature is whether the results obtained are repeatable and reproducible. In the opinion of the writer, as a forester who spent a considerable part of his service in India on timber and allied research (and also, strangely enough, for a while in Australia\*), the problem should be re-investigated in close co-operation with the forestry research organisations in Canberra and elsewhere in order to bring about a better understanding of the natural characteristics of timber in their relation to protective coatings. To mention a few : habitat, width of annual rings (rate of growth), nature and duration of seasoning, ratio of sapwood to hardwood in test samples, occurrence of resin, and so on. An improved classification and evaluation of timber defects may result and further light be shed on that unusual and elusive expression of the author's "the integrity value" of a timber.

The writer was influenced in his decision to raise the matter in the correspondence columns of *JOCCA* by the fact that the statistical analysis resulted in *pinus radiata* being placed top. The Radiata Pine (the only true pine tested in the analysis) is not generally recognised as a good timber tree, mainly because of its branchy character, while all paint technicians know that pine knots contain resin, which normally require special treatment before application of the final protective coatings. Perhaps the varnishes applied served the double purpose of sealer and of coating?

Yours faithfully,

6 Chesil Court, London, S.W.3. A. J. Gibson.

2 June 1966.

SIR,—We were interested to receive Mr. Gibson's letter and offer the following comments in reply.

In the assessment of the varnish holding properties of the 12 timbers exposed on our test fence, it was apparent both to the late Mr. K. L. Jones and to his colleagues that *pinus radiata* was superior to the other timbers. Each board was then rated in the manner described in the paper, both with respect to

<sup>\*</sup> See Commonwealth of Australia CSIR Pamphlet No. 9, 1928

## CORRESPONDENCE

whole board and sound areas; detailed statistical examination of the results confirmed our general impression that Radiata Pine was outstandingly the best timber for overall performance when finished with varnish.

The timbers used in the test fence were supplied by two organisations already acknowledged and were selected by the Timber Development Association of Australia as representing the timbers most widely used as cladding materials in the Australian building trade. Full details of these timbers of interest to the forester were not available, and I regret that we are unable to continue this work and to investigate more fully the timber variables as suggested by Mr. Gibson ; however, I have sent a copy of his letter and a copy of my reply to the Forestry and Timber Bureau, Canberra.

Although Mr. Gibson's criticism of Radiata Pine is valid, this timber is and will remain important in Australia and cannot be ignored. Climatic conditions in this country are such that *pinus radiata* has been recommended by the Australian Forestry Council as the principal species for extensive planting to provide for softwood requirements as far ahead as the year 2000. Further, it is considered economic in some areas to prune off side branches in order to eliminate knots in the mature timber. *Pinus radiata* is subject to a high rate of resin bleed from knots during kiln drying, but the subsequent rate of resin bleed is then much lower than after air drying. Kiln drying is therefore recommended for Radiata Pine when used for external cladding where painting or clear finishing is required. In the test fence set up at these laboratories, holes were stopped with linseed oil putty, but knots were not given any special treatment.

Yours faithfully,

Defence Standards Laboratories, South Australian Branch, c/o Post Office, Woodville, South Australia. H. C. Baghurst.

18 July 1966.

# Editorial

## Le divertissement nouveau

We were recently privileged to attend press shows of two films produced by firms well known to all our readers.

"Careers in Chemicals" has just been completed by Distillers Chemicals and Plastics Group in co-operation with the Appointments Boards of eight universities. It seeks to answer some of the questions which undergraduates are wont to ask about industry. On the whole it does a good job of presenting a balanced picture of what they can expect, but it is unfortunate, if perhaps inevitable, that it raises more questions than it answers.

"Tioxide" is the sequel to "The Titanium Pigment Story," made by British Titan Products Ltd. To one like ourself, knowing no more about pigment manufacture than can be found in the text-books, its portrayal of the processes involved appears realistic and, if the "hidden persuasion" is not too well hidden, what matter? A criticism which may be levelled at this film, among many others of its kind, is that one is not at all sure at what audience it is aimed. This information should be made available whenever such a film is launched and we would exhort the Royal Institute of Chemistry to insist on its inclusion in the notes prepared for the otherwise excellent Index of Chemistry Films.

Did you know that the RIC Index lists some hundreds of films and film strips covering all aspects of chemical science and technology? Did you know that, alongside those produced for sale by publishing houses like McGraw-Hill and Encyclopedia Britannica, there is a majority available on free loan from firms which together constitute a representative cross-section of the industry? Those responsible for forward thinking, not to mention training, could do a lot worse than devote a couple of hours of their employer's time, at intervals which should not be too short, to taking advantage of this free service.

Scientific principles and laboratory techniques are well presented in films by ICI, Perkin-Elmer, Crookes Laboratories and Oertling, to name but a few. Plant, instrumentation and materials handling are featured in profusion by a score or more manufacturers, and the more disinterested works of such bodies as the British Electrical Development Association will commend themselves particularly to those resentful of the flavour of publicity, however subtle, which is inseparable from the majority of these productions. Petroleum and petrochemical interests are well catered for by all the major oil companies and few would deny the Shell Film Unit honourable mention.

Safety is a subject which lends itself to disinterested and particularly effective presentation on "celluloid." Pyrene and Walter Kidde offer convincing demonstrations of fire-fighting equipment, but we would divide the honours between "It Couldn't Happen To Me" (J. Lyons), "Luck of the Devil" (Distillers) and "Black Monday" (ICI); the last is not for the squeamish.

If you seek a well balanced programme, you will no doubt want to include something about paint, pigments or resins. There is again no lack of choice, but you may have to sublimate your aversion to the voice of the tempter with the thought that you are discovering how the other fellow handles his publicity, if not his raw materials. To sweeten the pill, you may round off your programme with Cadbury's "Chocolate Odyssey," or, if you prefer other flavours, Harvey's "Your Glass of Sherry," or the Scotch Whisky Association's "Keepers of the Mystery." So why bemoan the fate of our picture palaces, converted by the hatchet men into bowling alleys and masonry-strewn car parks? If our proposals do not satiate your craving for bygone diversions, take heart—you still have the telly. And if you did not avail yourself of the privilege of viewing that inspired exercise in international relations, the World Cup (available on two channels, mark you), there is no doubt that you will be welcomed back into decent society, provided you keep such trivia as economic crises firmly in their place and make up for your lack of taste—by watching a reasonable number of the post-mortem presentations and buying the special bumper commemorative issue of the Evening Blurb, on which you will surely be able to dine out for the next four years.

# Reviews

OILS, FATS AND FATTY FOODS, THEIR PRACTICAL EXAMINATION Fourth Edition. By K. A. WILLIAMS. J. & A. Churchill Ltd., London, 1966. Pp. viii+488. Price 100s.

This book is remarkable in several different ways, not the least of which is the fact that it has managed to retain the same convenient size over the years, whilst most of its contemporaries have grown beyond all recognition. First published by Bolton and Revis in 1913, the present author received honourable mention for his assistance with the second edition of 1928 and he has had sole responsibility for the two further revisions, in 1950 and 1966. Keeping within bounds in this fourth edition has not been achieved without the use of certain stratagems, such as increasing the average number of words per page, but the high quality of the publication has not been jeopardised thereby.

The second point to notice is that the manuscript is founded upon the personal experience of analytical consultants specialising in oils and fats, and readers accustomed to literature surveys should not be dismayed to find that in some of the sections the references appear to die out in about 1930, and in others in about 1950. The first four chapters, dealing with analysis and the interpretation of results, have been very carefully revised and can be thoroughly recommended to anyone interested in the particular subject. But this is not just an analytical textbook, but one which provides sufficient background information to excite the analyst's interest in the material he is examining. Chapters on such topics as "Industrial Production of Vegetable Oils and Fats," "Hydrogenated or Hardened Oils," "Butter and Margarine," etc., should be regarded in this light rather than as up-to-date summaries of the technologies concerned. Nevertheless, at least one reader would have been happier if more attention had been given to the revision of some of them.

We now come to what many will consider to be one of the most interesting chapters in the book, entitled "Vegetable Oils and Fats," which gives details of the source and properties of about 150 varieties, including all of the important materials of commerce and many more besides. Here we must draw the line and say, quite firmly, that the material presented under this heading needs

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extensive revision to bring it up to date. The statement that cay-cay fat is produced in Cochin China and used by the natives for making candles may have meant something to readers of the second edition, but since then a whole generation has grown up that has not even heard of Cochin China as a place. nor even as a chicken. Of course, you may not require to look for cay-cay fat, but you will certainly want to read about soya bean oil. Perhaps it will surprise you to learn that the linolenic acid content is around 3 per centan idea that was out of date even when the statement first appeared in the third edition. Indeed, if you compare the chapter with the corresponding one in previous editions you will find much of the material going back for 40 years or more. Not that this necessarily invalidates the information, but interest in or availability of the lesser known oils changes from time to time and the list of those deemed worthy of mention should be adjusted accordingly. Oils such as kamala, ongokea, crambe, kenaf and many others spring to mind as having been much in the news during recent years, but no mention is made of them here. Vernonia anthelmintica oil is said to have evoked much interest on account of its unusual fatty acid composition-but what that is we are not told. The inclusion of an appendix giving a list of oil sources investigated during the period 1952-62 does not help in any way to redress the balance.

In spite of what has been said about the chapter on vegetable oils, experience has proved it to be useful in the past and no doubt it will continue to be so in the future. But no book is so good that it cannot be made even better and the revision of an old one is often more difficult than the production of an entirely new text. There is no doubt that Dr. Williams' new volume will occupy a prominent place in the libraries of all analysts dealing with oils and fats.

M. R. MILLS.

#### CHEMISTRY OF VEGETABLE TANNINS

By E. HASLAM. Academic Press Inc. (London) Ltd., 1966. Pp. 179. Price 47s. 6d.

The relatively recent development and application of new techniques has resulted in considerable progress in the analysis and elucidation of the structure of vegetable tannins—complex polyphenols with molecular weights in the range 500 to 3,000. This book meets the need which has thereby arisen for an up-to-date and critical survey of the present state of knowledge.

The subject is treated solely in relation to the use of polyphenol compounds as tanning agents for leather, and the first chapter, in fact, includes brief descriptions of the structure of collagen fibres in animal skins and of the mechanism of vegetable tannage.

The major part of the book is devoted to hydrolysable and condensed tannins, the two main structural types, and the related simpler polyphenols, such as flavanoid compounds, which are present in tannin extracts. The application of various techniques, such as chromatography, counter-current distribution, nuclear magnetic resonance and spectroscopy to the problems of analysis and characterisation is critically assessed.

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Finally, there is a detailed account of investigations concerned with the identification of precursors in the biosynthesis of plant polyphenols and the elucidation of probable routes for their formation in nature.

A large amount of factual information is condensed into this book and the text is well illustrated by numerous formulae and tables. It provides a very readable account for those who wish to inform themselves on the latest developments in tannin chemistry, but it is primarily intended for the specialist in this or related fields of natural products chemistry.

R. N. FAULKNER.

# Information Received

(In case of difficulty regarding addresses, members and subscribers to the JOURNAL should apply for details to the General Secretary of the Association at the address shown on the front cover.)

The SERICOL GROUP LTD. showed a number of interesting new developments at the "FESPA" Screen Printing Exhibition in Zurich. Their main exhibit comprised an automatic line suitable for the production of 50 in  $\times$  36 in screen prints on a variety of materials ranging from corrugated board to plastic sheeting for road traffic signs. The line consisted of an Aeropress Model III screen printing machine, the Autotake delivery unit and the Aerojet Type HHC jet air dryer. The Aeropress was shown for the first time with the new SPM automatic ink feed, which obviates the frequent stoppages hitherto necessary for the replenishment of the screen.

The Board of AUSTRALIAN CHEMICAL HOLDINGS LTD. has announced the completion of its plans to combine its wholly-owned subsidiaries in the chemical field into one chemical division. The new company, to be known as A.C. Hatrick Chemicals Pty. Ltd. from 1 July 1966, will encompass the entire operations hitherto carried out by Reichhold Chemicals Inc. (Aust.) Pty. Ltd., A.C. Hatrick Pty. Ltd. and Cement Chemicals (Aust.) Pty. Ltd.

The Chemical Society and the University of Nottingham announce the establishment of the CHEMICAL SOCIETY RESEARCH UNIT IN INFORMATION DISSEMINATION AND RETRIEVAL at Nottingham as from 1 August 1966.

LEWIS BERGER (GREAT BRITAIN) LTD. has announced a new product, Bergermaster acrylic primer/undercoat, marketed in either one-gallon or half-gallon sizes. In addition the company has announced a revision of the Bergerflek colour range, which amounts to a complete replacement of the existing 20-colour range with the same number of new colours.

The CHURCHILL INSTRUMENT Co. has announced the introduction of their falling rod viscometer. The company says this instrument is a falling coaxial cylinder type, with water jacket facilities for maintaining constant temperature conditions of test. The sample fills the annular gap between rod and orifice, governing the time of free fall of the rod between two reference marks 100 mm apart. The measured fall time is proportional to viscosity and the effective range is 20 to 2,000 poises. STANHOPE-SETA LTD., sole agents for Gardner Laboratory Incorporated, of Bethesda, Maryland, USA, have announced the availability of the new Gardner portable 60° gloss comparator. This instrument was developed for use by a large firm of manufacturers of farm machinery and equipment in the United States. It does not conform exactly to ASTM specifications and is a comparator only, but it is claimed to be a truly portable, very practicable device for checking gloss on the production line within reasonably close limits.

THOR CHEMICALS LTD. has announced conclusion of an agreement with A.F. Hechler SA, of Basle, for the manufacture of organo fluorine compounds in the UK. The range of compounds is biologically active and is used in industrial preservatives and botanical applications. One of the compounds is used as a low toxic preservative for emulsion paints, another as a systemic insecticide. The existing selling concession in the UK, held by M. E. Dougherty Ltd., a member of the Tar Residuals Group, will continue.

"Careers in Chemicals," a film made in co-operation with the Appointments Boards of eight universities, has been produced by DISTILLERS CHEMICALS AND PLASTICS GROUP and is available on free loan from this company.

A plant for the chemical removal of old paint from paint-mixing vessels (or pots), said to be ten times faster and much cleaner than the manual method commonly employed in the paint industry, has been designed and put on the market by the GENERAL DESCALING CO. LTD., of Worksop, Nottinghamshire.

PFIZER LTD. has announced that the extension to their pure synthetic iron oxide plant at Stalybridge will be in operation during the autumn of this year. This plant has been designed to meet in full the future demands in this country for high purity iron oxide, both for ferrites and for electrophoretic paints.

The first stage of the ICI (EUROPA) LTD. move to its new headquarters at 43 and 44 Boulevard du Régent, Brussels I, took place when the ICI (Europa) Plastics Group became operative there on Monday 4 July. The Chemicals Group became operative there on 18 July and the other sections of the organisation due to move will start operations in Brussels in the coming months. ICI (Europa) Ltd. is a new company formed by ICI in October 1965 to supervise and co-ordinate ICI selling and manufacturing activities throughout Western Europe.

A special unit where the paint application systems of all the big car manufacturers can be simulated has been constructed by PINCHIN JOHNSON AND ASSOCIATES LTD. at their works at Silvertown, London, E.16.

MINNEAPOLIS CARGILL INC. has announced plans to build Western Europe's largest soyabean processing facility in the Port of Amsterdam.

# Proceedings of the Annual General Meeting

The Fourth Annual General Meeting of the Incorporated Association was held on 30 June 1966 at 6.30 p.m. at Painter Stainers Hall, Little Trinity Lane, London, E.C.4, with the President (Dr. S. H. Bell) in the chair.

The notice convening the Meeting was read.

# Apologies

Apologies for absence were received from Mr. G. A. Campbell (President 1934-36), Mr. A. J. Gibson (President 1938-40), Mr. P. J. Gay (President 1959-61) and Mr. S. A. Ray.

#### Minutes

The President asked the meeting to take as read the Minutes of the Third Annual General Meeting held on 28 May 1965, as printed and circulated in *JOCCA*, 1965, **48**, 762-766. There being no comments, the adoption of the Minutes was put to the meeting and carried unanimously. The President then signed the Minutes.

## **Report of the Auditors to the Members**

The Report of the Auditors to the Members was read.

#### Annual Report of the Council for 1965

Mr. I. C. R. Bews (Honorary Secretary) moved the adoption of the Annual Report of the Council and the statement of Accounts for 1965.

Mr. F. Sowerbutts (Honorary Treasurer) seconded the adoption of the Annual Report of the Council and statement of Accounts for 1965. He pointed out that although in 1965 the income of the Association increased by over  $\pounds 3,400$ , the deficit on the year's working was  $\pounds 2,415$ . As this was the second successive year in which a deficit had been incurred, the most careful scrutiny had been made of the financial forward planning of the Association and he would have more to say on this aspect under a later item.

The Chairman asked if any member wished to comment on the Annual Report of the Council and the Statement of Accounts.

In relation to the Biennial Conferences, Dr. L. Valentine felt that the Association was too generous in respect of the payment of expenses not only for the chairmen of sessions and lecturers but also for their wives. This was unique in his experience and he suggested that Council should consider in future paying the expenses only of wives of lecturers and chairmen of sessions.

The Chairman stated that this suggestion would be noted; every attempt was being made in recent years to make the Conferences self-supporting.

Mr. H. A. Newnham asked if an explanation could be given for the relatively high expenses per head of the New South Wales and Victorian Sections compared to the London Section.

Mr. F. Sowerbutts stated that this was partly attributable to the help which the United Kingdom Sections received from the headquarters offices at Wax Chandlers' Hall and in which the Australian Sections could not share; nevertheless, the Australian Sections were expensive to operate.

There being no further comments or questions on the Annual Report of the Council and Statement of Accounts, these were formally adopted by the meeting.

#### Election of President-Designate of the Association

Dr. Bell stated that it was not easy for a President to say what a President ought to be, yet it was only from a knowledge of the activities involved that one could attempt to consider the man, the time and the circumstances. His

successor as President would hold office during the Jubilee Year and it gave him great pleasure to put forward the name of Mr. F. Sowerbutts as the Council's nomination. Mr. Sowerbutts had had over 40 years of continuous service to the Association. He was a founder member of the Manchester Section, Honorary Treasurer of that Section for over 14 years and for a time its Honorary Secretary. In 1948 he was the first Chairman of the Midlands Section. He had been a Vice-President of the Association twice and since 1960 has been Association Honorary Treasurer and Chairman of the Exhibition Committee; he was now a member of the London Section.

This was a remarkable record and the President formally proposed to the meeting that Mr. F. Sowerbutts be elected President-Designate.



F. Sowerbutts, President Designate

This was carried unanimously and with acclamation.

Mr. Sowerbutts thanked the meeting for the honour bestowed upon him. This was an honour to which he had never aspired and he would do his utmost to merit the confidence of the members.

#### Election of Vice-Presidents of the Association

The Chairman asked the meeting to take the nominations of the Council as

printed on the agenda en bloc and this was agreed. The following were then elected as Vice-Presidents :

(a) Vice-Presidents who have not been President :

(i) Mr. J. C. Anner
(ii) Mr. P. B. Hunt
(iii) Mr. H. A. Newnham
(iv) Mr. J. Smethurst
(v) Dr. H. W. Talen
(vi) Mr. V. C. Thompson

(b) Vice-President who has been President :

Mr. N. A. Bennett

#### **Election of Honorary Officers of the Association**

The Chairman asked the meeting to take the nominations of the Council as printed on the agenda en bloc and this was agreed. He pointed out that, owing to the appointment of Mr. Sowerbutts as President-Designate, it was necessary for Council to nominate a successor to him as Honorary Treasurer. He felt sure that with Mr. A. S. Fraser as Honorary Treasurer the Association would be well served.

It was unanimously agreed to elect the Honorary Officers as follows :

Honorary Secretary	••	• •	• •			Mr. I. C. R. Bews
Honorary Treasurer	••	••	••	••		Mr. A. S. Fraser
Honorary Editor		••		••	••	Mr. A. R. H. Tawn
Honorary Research an	d Dev	elopme	ent Offi	cer	•••	Mr. A. T. S. Rudram

#### Announcement of Election of Three Elective Members to Council for 1966-68

The President read the following report which had been received from the Auditors :

We have scrutinised the voting papers for the three elective members of the Council received from the members in the United Kingdom and General Overseas Sections, and certify that the votes cast, including those notified by letter from the South African and Victorian Sections, show that the following obtained the largest number of votes :

1. G. H. Hutchinson 2. A. W. Blenkinsop 3. L. Tasker

24 voting papers were rejected as not being in order.

Cooper Brothers & Co., Chartered Accountants.

London, 22 June 1966

The President then declared the three members listed elected to Council.

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#### Chairmen of Sections for the Coming Session

The names of the Section Chairmen for the coming year were given as follows :

Auckland					Mr. P. B. Hunt
Bristol					Mr. R. J. Woodbridge
Hull	• •	• •		• •	Mr. L. W. Wynn
Irish	• •	• •	• •		Mr. J. H. A. Quick
London		• •			Mr. C. R. Pye
Manchester	••	• •	••		Mr. I. S. Moll
Midlands	• •	••	••	• •	Mr. C. H. Morris
Newcastle			• •		Mr. E. L. Farrow
New South W	ales		• •		Mr. D. M. D. Stewart
Queensland		•	•••	• •	Mr. R. N. Lewis
Scottish		• •	•••	• •	Mr. I. S. Hutchison
South African	• •			• •	Mr. K. R. McDonald
South Austral	ian	••	••		Mr. M. D. Gilham
Victorian		• •			Mr. F. Marson
Wellington	• •		••		Mr. R. D. Bell
West Australia	an	••	••	••	Mr. P. Murphy
West Riding	• •		••	• •	Mr. N. Cochrane

#### **Membership Subscription Rates**

The Chairman reminded the meeting that, under Article 11 of the Association's Articles of Association, the membership subscriptions were determined at two successive Council meetings and confirmed by the members in general meeting.

Mr. Sowerbutts proposed that with effect from 1 January 1967 the annual membership subscription rates in the various categories of membership shall be as follows :

Ordinary and Associate Memb	pers	• •	 £5	5s.	0d.
Retired and Junior Members		• •	 £1	1s.	0d.

In proposing this motion, Mr. Sowerbutts reiterated the points outlined in the memorandum circulated to members of the Association on 5 May with the agenda for the Annual General Meeting. He said that a small Committee had first examined the financial position of the Association and their conclusions were considered in detail by the Finance Committee, which put forward recommendations which were debated at two successive Council meetings in November 1965 and February 1966. As a result Council determined that the subscription rates for 1967 should be increased and, in accordance with Article 11, this motion was before the General Meeting for confirmation.

Mr. J. Smethurst seconded the motion, saying that as a member of the Finance Committee he felt that the financial participation by the membership needed to be improved and that, after a lot of consideration, he was sure that the proposal was one which must be supported.

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The Chairman stated that, before asking for comments, he felt he should outline the position of the Association in Australia, where the Association had five Sections with a co-ordinating Federal Committee, and in the last few weeks a considerable volume of correspondence had been received objecting to the subscription increase. The Australian Federal Committee had sent a letter to the President asking for it to be read to the Annual General Meeting. In the unavoidable absence of the Australian Sections' Representative on Council (Mr. P. J. Gay), the letter and other communications were read and amplified by the Immediate Past President, Dr. J. E. Arnold.

Having explained the position in Australia, the Chairman then asked for comments from the Annual General Meeting. The following members contributed to the debate :

Mr. H. A. Newnham, Dr. F. M. Smith, Mr. C. Butler, Mr. G. L. Lewis, Mr. J. J. Pemberton, Mr. N. A. Bennett, Mr. G. D. Topping, Dr. F. W. Stoyle, Mr. A. E. Skinner, Mr. L. H. Silver and Mr. M. H. M. Arnold.

A motion was proposed for the adjournment of the debate on this item, but the Chairman, sensing the feeling of the meeting and in consultation with the Association's Solicitor, declined to accept this.

The motion to confirm the subscription rates for 1967 as printed on the agenda was then put to the vote and the Chairman declared it to be carried.

#### Re-appointment of Auditors and fixing the remuneration thereof

It was proposed by Dr. S. R. W. Martin that Cooper Brothers & Company (Chartered Accountants) be re-appointed Auditors of the Association and that their fee be increased from 100 guineas to 150 guineas (inclusive of the fee paid for the auditing of the accounts of the Australian Federal Committee). This was seconded by Mr. R. N. Wheeler and carried unanimously. With the consent of the meeting the remaining items on the agenda were deferred until after the Foundation Lecture and Dinner.

#### Vote of thanks to retiring Council Members

Mr. C. W. A. Mundy (President 1955-57) moved a vote of thanks to those members retiring from Council for their services to the Association and this was carried with acclamation.

#### Vote of thanks to Honorary Officers

Dr. F. M. Smith proposed a vote of thanks to the Honorary Officers. As a Council member he had been able to appreciate the hard work done for the Association by the Honorary Officers. Since this was the only opportunity for the members to show their appreciation of their services he asked them to join him in the vote of thanks, which was carried with acclamation.

#### Vote of thanks to Chairman of Meeting

Mr. I. C. R. Bews (Honorary Secretary) called upon Mr. N. A. Bennett (President 1957-59) to move a vote of thanks to the President as Chairman

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of the meeting. Mr. Bennett said that he felt sure all members present would appreciate that the Annual General Meeting had been a most difficult one at which to take the chair and that the President deserved the congratulations of all on the way in which he had conducted the proceedings. He called on the members to show their appreciation by a warm vote of thanks, which was carried by sustained applause.

#### Any other business

Mr. F. Sowerbutts asked the meeting to record its thanks to the General Secretary (Mr. R. H. Hamblin) and his staff and the meeting responded in the customary manner.

The President then declared the proceedings closed.

### New South Wales

#### Particle size distribution and assessment using the disc centrifuge

At the May meeting, Mr. Brawn, of ICIANZ, presented a paper on "Particle Size Distribution and Assessment Using the Disc Centrifuge."

The assessment of pigment dispersion by ordinary gauge does not provide any information on the particle size distribution and more complicated techniques are necessary to obtain this information.

Mr. Brawn said that there are some 75 techniques known for measuring particle size distribution, e.g. optical and electron microscopes, electronic counters, sedimentation, etc.

Mr. Brawn then described in full detail a novel apparatus, the disc centrifuge, which is used for measuring particle distribution by a comparatively simple method. The apparatus is used to separate dispersed pigment particles into fractions which are then analysed by colorimetric or conventional analytical methods.

A lively question time followed the lecture and the vote of thanks was proposed by Mr. M. J. Leahey.

A. A.

### Scottish

#### Eastern Branch

#### International test methods for paints

"International Test Methods for Paints" was the title of an illustrated lecture given by Mr. T. R. Bullet to the fifth meeting of the Branch. This was held in the North British Hotel, Edinburgh, on 28 February 1966.

Outlining the universal organisations which were concerned with the technical performance of paints, Mr. Bullet disclosed some of the items that were currently under investigation at the Paint Research Station.

The requirements to make an international test method successful were :

1. Ease of operation. 2. Good reproducibility of results. 3. Inexpensive apparatus.

Mention was made here of tests such as the American Ford Cup (B.4 and No. 4) and German DIN Cups for viscosity/consistency measurements.

A newer instrument than these traditional ones was the Rotothinner due to Bushing, which complied with the three requirements given above.

Drying time and film hardness testers were discussed, and the use of aluminium v. steel as a substrate for mandrel tests mentioned.

With adhesion tests the European cross-cut grid tests were illustrated, and the PRS tensometer method described.

Flash point, gloss and contrast ratio/hiding power methods were given in some less detail.

It was stated that durability and artificial weathering were still under study, the proposed BS method being well thought of. Some rusting studies and plate preparation were illustrated.

Such a wide range of technical tests provoked much interested discussion after the lecture, and among those who participated were Mr. Holmes, Mr. Hutchinson, Mr. Gummerson and Mr. Cochrane.

Mr. Soames proposed the vote of thanks to the lecturer and this was well accorded.

### 8th Australian OCCA Convention



The Hotel Victor, Victor Harbour. Headquarters for the 8th Australian OCCA convention held 14-17 July 1966

The 8th Convention, held at Victor Harbour, South Australia, from 14-17 July, proved to be another very successful Australian Federal Committee project. The Convention organisers, namely the South Australian Section Committee, can rest assured that the function well maintained the standard of previous OCCA Conventions in every way. The total attendance, including daily delegates who did not require accommodation, was 192. Full-time delegates, totalling 160, were accommodated in the three Victor Harbour hotels, The Victor, The Grosvenor and The Crown. Attendances at the technical sessions remained fairly constant at well over 100 persons throughout and 172 delegates



A section of the audience attending one of the technical sessions

attended the Official Convention Dinner on the night of Saturday 16 July.

The following papers were presented at the technical sessions held on Friday 15 July and Saturday 16 July :

"The Importance of Physical Effects on the Action of Catalytic Surfaces in Vinyl Polymerisation," by Dr. P. E. N. Allen (University of Adelaide, South Australia).

This paper put forward some theories on what happens during vinyl polymerisation, these theories being based on careful kinetic measurements and direct physical observation.

"A Study of the Optical Behaviour of Titanium Dioxide Pigments and the Interactions Resulting from Concentration Effects," by Dr. W. E. Craker and F. D. Robinson.

The lecturer (Dr. W. E. Craker) presented a detailed interpretation of the results obtained from the measurement of the optical properties of paint films at various thicknesses and PVCs.

"The Formulation of Fungus-Resistant Paints," by Dr. E. Hoffmann.

This paper described the work carried out for the Victorian Housing Commission by CSIRO in connection with the development of a reliable fungus-



Mrs. E. M. Southwell, the first lady to present a paper at an Australian OCCA Convention

resistant paint. Zinc oxide alkyd paints and zinc oxide latex paints were shown to provide fungus-resistant properties much superior to paints containing fungicides as additives.

"Some Aspects of Recent Paint Research at Defence Standards Laboratories," by M. Camina and Mrs. E. M. Southwell.

Mr. Camina and Mrs. Southwell jointly described three aspects of research being carried out at DSL, Maribyrnong : the relationship between solar radiation variances and paint film durability, a study of heterogeneous clear varnish systems and the assessment of fungalresistant paints.

Mrs. Southwell is the first lady ever to have lectured at an Australian OCCA Convention.

"Factors Affecting Surface Profile in Abrasive Impact Cleaning," by R. J. McIntyre.

This very detailed paper dealt with the study of the many variables associated with sand and grit blasting techniques and the desirability of controlling all these factors in order to achieve uniformity of surface texture within a certain specification of required characteristics to provide suitable paintholding properties.

"The Testing of Zinc Dust Paints," by D. G. Thomas and B. A. Richardson.

The lecturers related the performance of various zinc rich paints to formulating factors, including metallic zinc contact in the dried paint film, vehicle type, size of zinc particle, film thickness and surface topography.

"Tar Epoxy Protective Coatings," by A. Adomenas.

This paper described the laboratory work carried out over the last four years to assess the relative merits of the various types of coal tars available in Australia and the performance of solvent based tar epoxy coatings was compared with high solids and solvent free types. The advantages and disadvantages of polyamide cured coatings against amine cured coatings were also discussed in detail. "New Applications for Polyurethane Surface Coating Materials," by Dr. L. Havenith.

A brief description of theoretical considerations was given for the selection of isocyanates suitable for lacquers. The reaction mechanism of an isocyanate prepolymer with polychloroprene was reviewed as a possible means of producing elastomeric coatings. The properties of an aliphatic isocyanate of a biuret structure were discussed and some fast curing polyurethanes were described.

#### Social

The two official social functions held during the Convention were a buffet dinner on the night of Thursday 14 July and the Convention Dinner on the night of Saturday 16 July. The quality and the quantity of delicious food prepared for those two functions were outstanding. At the Convention Dinner a toast to OCCA was proposed by Mr. K. Ellis, a member of New South Wales Section and a foundation member of the Australian Federal Committee. Mr. E. T. Backous, Chairman OCCA in Australia, responded to the toast and spoke briefly on OCCA in Australia.

The guest speaker at the Convention Dinner was Mr. A. M. Ramsay, General Manager of the South Australian Housing Trust. Mr. Ramsay talked in a lighthearted manner about the work of the Housing Trust as well as many other topics of direct or indirect interest to OCCA members.

Unofficial social activities included golf and swimming in the heated pool attached to Hotel Victor.



Mr. A. M. Ramsay, guest speaker at the official dinner. In the background (l. to r.) are Mr. M. D. Gilham, South Australian Section Chairman, Mr. E. T. Backous, Australian Federal Committee Chairman and Mr. B. Lourey, AFC Honorary Treasurer

### **OCCA Biennial Conference**

20-24 June 1967: "Interfacial Behaviour"



A view of St. Nicholas Cliffs showing the Grand Hotel on the left and the St. Nicholas Hotel on the right

It is felt that those intending to be present at the next Association Conference and who were not present at the last Scarborough Conference in 1963 would like to see the relative positions of the Grand Hotel and the St. Nicholas Hotel. Most of the delegates will be accommodated in the Grand Hotel, where the Technical Sessions and the Official Functions will be held, and the remainder of the delegates will stay at the St. Nicholas Hotel.

The theme of the Conference will be "Interfacial Behaviour" and it is expected that on this occasion there will be four Technical Sessions, each of which will be devoted to a particular aspect of this theme. Following their successful introduction at the Torquay Conference in 1965, there will also be "Workshop Sessions," where topics that are of interest to the delegates but not connected with the main theme of the Conference will be discussed.

There will be a full social programme with a Civic Reception on the Wednesday and the Association's Dinner-Dance on the Friday evening. Tours to places of interest will be arranged and there will be opportunities for delegates to take part in various sporting activities.

Details will be circulated to members early in 1967, but others wishing to receive details should write to the General Secretary at the address shown on the front cover.

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### **Midlands Section**

#### Day Symposium

For this second Day Symposium, organised by the Midlands Section, on Thursday 20 October 1966 at Regency Club, Stratford Road, Monkspath, Shirley, Solihull, Warwickshire, a controversial subject of topical interest has been chosen under the title "Solvent or Solventless." The present day trend of coatings, from the end user point of view, will be dealt with by specialists in a number of fields and it is hoped that the interest of delegates will be aroused and a stimulating discussion result.

Registration of delegates must be made on a form obtainable from the Honorary Secretary of the Section (D. J. Silsby, 356 Baldwins Lane, Hall Green, Birmingham 28) not later than 17 September. Accommodation is limited. Applications will be accepted in strict order of receipt.

The fees, which include coffee and lunch, will be:---

Members of OCCA£1 10s 0dNon-Members£2 0s 0d

Delegates will receive synopses of the lectures at least two weeks prior to the

Symposium together with a numbered programme and list of delegates.

The programme will be as follows :

- 10.30-11.00—"Painting at Rolls-Royce; Present and Future," B. J. Wilmin (Rolls-Royce Ltd.).
- 11.00-11.30—" The Emulsion, Solvent and Solventless Paints in Railway Service," F. G. Dunkley (British Railways).
- 11.30-11.45—Coffee.
- 11.45-12.15—" Electrical Insulating Varnishes and Compounds," F. Meyer, B.SC., M.I.E.E. (The Sterling Varnish Co. Ltd.).
- 12.15-1.00—Discussion.
- 1.00-2.30-Lunch.
- 2.30-3.00—"The Influence of Solvent on Choice of Coatings for HM Ships," J. Smith, B.SC. (Central Dockyard Laboratory).
- 3.00-3.30—"Solvent, Room to Manoeuvre," V. A. Moore (Pinchin, Johnson & Associates Ltd.).
- 3.30-4.30-Discussion.

### **Newcastle Section**



The winners of the British Titan Cup, Mr. T. W. Robinson (left) and Mr. B. G. Anderson

#### **Golf tournament**

The fifth annual tournament for the

British Titan Cup took place at Brancepeth Castle Golf Club on 25 June 1966.

The competition, a fourball, betterball against bogey, was jointly won by :

Mr. T. W. Robinson (International Paints, Felling) and Mr. B. G. Anderson (British Titan Products Co. Ltd.) (three down).

Twenty people took part in the competition in conditions which were very good, dry and sunny.

It is perhaps worthy of note that last year's cup holder, Mr. R. G. Carr, of Camrex Paints Ltd., Sunderland, also finished three down, but the rules stipulate that in the event of a tie the pair with the better second half will win.

JOCCA

### **New South Wales Section**



Committee for 1966. Seated (left to right): M. J. Leahey (Immediate Past Chairman), D. F. Crossing (Hon. Secretary), D. M. D. Stewart (Chairman), D. Thompson (Hon. Treasurer) and A. Adomenas (Hon. Publications Officer). Standing : J. A. Scott, J. C. Gibson, R. Drummond, P. A. York, D. I. Townsend and B. T. Clarke

#### **Past Chairmen's Dinner**



Part of the table showing (left to right): T. Backous (Federal Chairman), D. M. D. Stewart (Section Chairman), M. J. Leahey (Immediate Past Chairman) and S. L. Leach, D. Crossing, E. Haimann and D. Thompson

NSW Section's Past Chairmen's Dinner was held at "Jonroe" on 2 June, being attended by 15 Past Chairmen, the present Chairman and all members of the Committee. It was an enjoyable evening, each Past Chairman making a few comments on topics of interest concerning the Oil and Colour Chemists' Association.

There was considerable discussion over the proposed increase in subscriptions and the possible consequences of reduced membership.

It was emphasised that it was not the increase alone that was being objected to by NSW Section, but also to the fact that Australian Sections have no opportunity to vote in the UK on important matters such as this that could have a profound effect on the Section.

#### 1966

#### Wine-tasting evening

A most successful wine-tasting evening was held at "Ye Olde Crusty Taverne" on Tuesday 10 May. A record number of some 150 members, their wives and guests attended this very popular event amongst OCCA social functions.

A comprehensive range of wines was made available by the Wine and Brandy Producers' Association. Mr. A. L. Moore,

**Courses in Surface Coatings Technology** Bradford Technical College has organised the following course in surface coatings for the 1966-67 session designed to cover the CGLI No. 250 Printing Ink Technician's certificate, part 1, syllabus, over two years.

Should there be sufficient demand, a course for CGLI No. 9 Paint Technician's certificate for 1968 (last year of old scheme) will be run.

Further information about these courses is available from :

Mr. R. E. Todd, Bradford Technical College, Great Horton Road, Bradford, 7.

#### The Proposed University of Surrey Battersea College of Technology

Two courses in rheology have been organised by the Department of Physics as follows :

Measuring Techniques and Analysis of Observations—a course of about 20 lectures on Tuesdays, 6.30 p.m., 8.30 p.m., commencing 4 October 1966.

Mathematical Theory of Stress and Deformation—a course of about 20 lectures on Wednesdays, 10 a.m.-12.15 p.m., commencing 5 October 1966.

These lectures form part of a series preparing students for the Post Graduate Diploma or M.Sc. Degree in Rheology of the University of Surrey.

Particulars are available from : Mr. R. W. Whorlow, Department of Physics, Battersea College of Technology,

London, S.W.11.

Wine Advisory Officer, discussed various wines and advised on choice of wines suited for different occasions. "Ye Longe Boarde" smorgasbord meal was well prepared to suit all tastes.

The event was very ably organised by the new social convenor, David Townsend, and members can look forward to other successful social events later this year.

Course in Chemistry and Technology of Surface Coatings

A Course in the Chemistry and Technology of Surface Coatings has been arranged by the Department of Chemistry and Metallurgy, Rutherford College of Technology, Newcastle upon Tyne. The course, which begins in the third full week in September and extends over three terms, will be part-time, attendance being required one day each week (Thursday). Entry qualifications are HNC Chemistry or Applied Chemistry and candidates successful at the examination taken at the end of the course will qualify for an endorsement to the HNC. Successful candidates who also satisfy the require-ments of the ancillary subjects and industrial experience would normally be eligible for the licentiateship of the Royal Institute of Chemistry.

Requests for further details and applications for enrolment should be sent to :

The Principal, Rutherford College of Technology, Ellison Place, Newcastle upon Tyne, 1.

#### **News of members**

Mr. A. R. H. Tawn (Hon. Editor), Research Manager of Cray Valley Products Ltd., has been appointed an Associate Lecturer in the Department of



A. R. H. Tawn

Chemistry and Chemical Technology of the Borough Polytechnic, London. Like Dr. S. H. Pinner, who was appointed some years ago, he will assist Dr. S. Cotson, Head of the Department, in industrial liaison and the provision of courses on polymers and surface coatings at the postgraduate level. Associate Lectureships are bestowed by the Board of Governors on a limited number of distinguished industrial scientists whose part-time and advisory services they wish to retain.

### **Obituaries**

#### John H. Hesketh

John's many friends and past associates in the surface coatings industry were saddened to learn of his death on 22 February, at the age of 68.

An Overseas Member of OCCA, he was a prominent figure in the formation of the Australian Section and its Victorian Branch in 1946. He served on the early Branch Committee, and was elected vice-chairman (1953) and chairman (1954) of the Victorian Section. He was also vice-president of the Association (1954-55).

Educated at Trinity Grammar, and Melbourne University, he was an early (1923) Associate of the Royal Australian Chemical Institute, and was elected a Fellow in 1948.

His early life in the chemical industry began as a partner of Laughlin, Drake and Hesketh; in 1926 he joined Hardie Trading Ltd. Responsible for his company's entry into the nitrocellulose automotive finish field in this period, he also introduced alkyd technology from the United States in the early 30's. After having been technical director of Hardie Trading Ltd. ("Spartan") for some 14-15 years, he retired from the paint industry in June 1962.

His personal interests included troutfishing, golf, pictorial photograph, flowerraising and recorded music. One was wary of discussing with him Rosencrantz and Laertes, stereophony, Weingartner's Beethoven, dry fly vs. wet fly, or corms. His later years were spent raising hothouse plants on quite an extensive scale at his country home at Olinda.

John is survived by his wife and two married daughters.

To have worked with this gentle man was a privilege.

D. A. K.

#### E. A. Bevan

We are sorry to report the death of Eric A. Bevan, President of the Research Association of British Paint, Colour and Varnish Manufacturers, at his home in Pott Shrigley, Cheshire, on 2 August. He was an Ordinary Member of the Association attached to the Manchester Section, and Managing Director of Styrene Co-Polymers Limited. A full obituary will appear in a later issue of the *Journal*.

### **Register of Members**

The following elections to membership have been approved by Council. The Sections to which the new members are attached are given in italics.

#### **Ordinary Members**

BENN, MICHAEL GEORGE, B.SC., Reckitt & Colman (Africa) Ltd., PO Box 8247, Johannesburg, South Africa. (South African)
CAFFERY, GEORGE FRANCIS, "Oakford Cottage," Island Wall, Whitstable, Kent. (London)
CARTWRIGHT, JEFFREY, 9 Woodside, Knutsford, Cheshire. (Manchester)
CHWEIDAN, ROY, B.SC., 94-15th Street, Orange Grove, Johannesburg, South Africa. (South African)
GILMOUR, SAMUEL THOMPSON, B.SC., British Paints (NZ) Ltd., Rosebank Road, Avondale, Auckland, New Zealand. (Auckland)
GRANDAGE, DAVID BRIAN, 17 Royal Oak Drive, Bishopswood, Brewood, Stafford. (Midlands)
KAPOOR, KUL PRAKASH, B.SC., 15 Rosehill Road, London, S.W.18. (London)
KORMOS, ANDRES, Koral Petroquimica SACIFIA, Paseo Colon 1216, Buenos Aires, Argentina. (Overseas)
PAGAN, DAVID ROY, B.SC., Herbert Evans & Co., PO Box 1386, Durban, South Africa. (South African)
SEFTON, REUBEN JOSEPH, B.SC., A.M.C.T., 23 Hillside Road, Southport, Lancs. (Manchester)
SIBLEY, DEREK JOHN, 16 Preston Avenue, Newport, Mon. (Bristol)
STYNES, ALBERT, 3 Ranworth Road, Gt. Sankey, Warrington. (Manchester)
TELANG, DINKAR BHALCHANDRA, M.SC., International Paints Ltd., Felling-on-Tyne, Gateshead, 10, Co. Durham. (Newcastle)
TILLYER, RICHARD BRIAN, Expandite (NZ) Ltd., PO Box 38079, Petone, Wellington, New Zealand. (Wellington)
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Associate Members
BAYNES, ROBERT CYRIL, 93a Valley Road, Mount Eden, Auckland, New Zealand. (Auckland)
BAYNES, ROBERT CYRIL, 93a Valley Road, Mount Eden, Auckland, New Zealand.
BAYNES, ROBERT CYRIL, 93a Valley Road, Mount Eden, Auckland, New Zealand. (Auckland) BOLT, NIGEL LANGSTON, PO Box 341, Dar-es-Salaam, Tanzania, East Africa.
<ul> <li>BAYNES, ROBERT CYRIL, 93a Valley Road, Mount Eden, Auckland, New Zealand. (Auckland)</li> <li>BOLT, NIGEL LANGSTON, PO Box 341, Dar-es-Salaam, Tanzania, East Africa. (Overseas)</li> <li>BROWNELL, PETER C., Flat 4/27, Laperouse Street, Manly, New South Wales, Australia.</li> </ul>

GRIMMER, DAVID FRANCIS JAMES, Geigy (UK) Ltd., Melton Road, Thurmaston, Leics. (Midlands—Trent Valley Branch)

PRINCE, RICHARD JOHN, PO Box 3747, Wellington, New Zealand. (*Wellington*) SACCO, MANLIO, 19 Salford Street, Salisbury, Queensland, Australia. (*Queensland*) BROWN, DAVID MACDONALD, 7 Glendevon Square, Glasgow, E.3. (Scottish) HEATH, DAVID MICHAEL, 20 Leicester Road, Sharnford. Leics. (Midlands) MCMAHON, JAMES, 16 Bellfield Street, Glasgow, E.1. (Scottish) PINSON, MAXWELL DAVID, 40 Cavendish Gardens, Wolverhampton. (Midlands) ROMMENS, KEITH, General Chemical Corp. Ltd., PO Box 21, Jacobs, Durban, South (South African) Africa. SNELL, DAVID GEOFFREY, Blundell-Permoglaze Ltd., James Road, Tyseley, Birmingham, (Midlands) 11. WICKEN, MICHAEL WILLIAM, 93 Bracondale Road, Abbey Wood, London, S.E.2. (London)

### **Forthcoming Events**

Details are given of meetings in the United Kingdom up to the end of the month following publication, and in South Africa and the Commonwealth up to the end of the second month.

#### **Tuesday 6 September**

Manchester Section. Junior Lecture : "Basic Rheology—What It Can Tell You," by Mr. A. R. H. Tawn (Cray Valley Products Ltd.), to be held at the Manchester Literary and Philosophical Society, 36 George Street, Manchester, 1, at 4 p.m.

#### Monday 12 September

Victorian Section. "Security Printing," by Mr. W. Steggles, to be held at Union House, Melbourne University, Parkville.

#### **Tuesday 13 September**

West Riding Section. Symposium on Dispersion : "High Speed Mixing," by Mr. J. de Jong (Morehouse International) ; "Ball Milling," by Mr. I. R. Sheppard (Steele & Cowlishaw) ; "Sand Milling," by Mr. D. P. Sullivan (D. H. Industries).

#### Wednesday 14 September

London Section. "Problems of Paint Users," by Mr. D. A. Bayliss (CEGB), to be held at the Physics Department, Imperial College of Science and Technology, South Kensington, London, S.W.7, at 6.30 p.m.

#### **Thursday 15 September**

New South Wales Section. "Silicone Copolymers-Their Manufacture and

Use," by Mr. J. H. Vinson and Mr. H. G. Stephen. Further details from Section Secretary, Mr. D. F. Crossing, 10 Richard Road, St. Ives, New South Wales.

#### Friday 16 September

Midlands Section. "Continuous Strip Coating," by Mr. E. E. V. Sharpe, to be held at Chamber of Commerce House, 75 Harborne Road, Birmingham, 15, at 6.30 p.m.

#### Friday 23 September

*Manchester Section.* Works Visit : The Wallpaper Manufacturers Ltd., Holmes Chapel Branch and Jodrell Bank.

#### **Thursday 29 September**

London Section—Thames Valley Branch. "Grinding," by Mr. A. C. B. Mathews, to be held at the Royal White Hart Hotel, Beaconsfield, Bucks., at 7 p.m.

#### Friday 30 September

Bristol Section. "Trichlorethylene Paints," by Mr. A. H. McQuillan (ICI Ltd.), to be held at the Royal Hotel, College Green, Bristol, 1, at 7.15 p.m.

Irish Section. "PVA Pigmentation," by Mr. L. Tasker, to be held at the Clarence Hotel, Wellington Quay. Dublin, at 8 p.m.

#### Monday 3 October

*Hull Section.* "Colour," by Mr. W. B. Cork (Reckitts Colours Ltd.), to be held at the Royal Station Hotel, Hull, at 7 p.m.

#### **Thursday 6 October**

Midlands Section—Trent Valley Branch. "Versatic Acids and Their Derivatives," by Mr. M. V. Moore (Shell Chemical Co. Ltd.), to be held at the British Rail School of Transport, London Road, Derby, at 7.30 p.m.

*Newcastle Section.* "Metallo-organic Primer Coatings," by Mr. R. N. Faulkner (PRS), to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 p.m.

#### Monday 10 October

London Section—Southern Branch. "Polyurethanes—Progress in the Surface Coating Industry," by Mr. G. Sutno, to be held at the Royal Hotel, Southampton, at 7.30 p.m.

#### **Tuesday 11 October**

West Riding Section. "Modern Developments in Printing Inks and Paper Varnishes," by Mr. J. A. L. Hawkey, to be held at the Great Northern Hotel, Leeds, at 7.30 p.m.

#### **Thursday 13 October**

Scottish Section. "Fish Oils, Their Composition, Processing and Some Industrial Application," by Mr. S. A. Reed (Marfleet Refining Co. Ltd.), to be held at More's Hotel, India Street, Glasgow, at 6.30 p.m.

#### Friday 14 October

*Hull Section.* 23rd Annual Dinner-Dance, to be held at the Eden Hotel, Willerby, Nr. Hull, Yorks, at 7 p.m.

Manchester Section. "Strip Coating," by Mr. D. S. Newton (Richard Thomas & Baldwins Ltd.). Joint meeting with the North-West Branch of the Institute of Metal Finishing, to be held at the Manchester Literary and Philosophical Society at 6.30 p.m.

#### Saturday 15 October

Scottish Section—Student Group. "Colour Consciousness," by Dr. F. M. Smith (Geigy (UK) Ltd.), to be held at the Lorne Hotel, Argyll Street, Glasgow, at 10.30 a.m.

#### Thursday 20 October

London Section. "The Behaviour of Printing Ink on Rollers," by Mr. C. C. Mill (The Printing, Packaging and Allied Trades Association), to be held at the Physics Department, Imperial College of Science and Technology, South Kensington, London, S.W.7, at 6.30 p.m.

*Midlands Section.* "Solvent or Solventless Symposium," to be held at the Regency Club, Shirley, at 10.30 a.m.

*New South Wales Section.* "Metal Protection with Vinyls," by Mr. B. Godard (Union Carbide Inc., USA). Further details from Section Secretary, Mr. D. F. Crossing.

#### Wednesday 26 October

Scottish Section—Eastern Branch. "Tall Oil and its Products," by Mr. Porter (British Oxygen Chemicals Ltd.), to be held at the North British Hotel, Princes Street, Edinburgh, at 7.30 p.m.

#### **Thursday 27 October**

London Section—Thames Valley Branch. "Paints to Meet Motor Car Manufacturers' Requirements," by Mr. I. Walker (Ford Motor Co.), to be held at the Royal White Hart Hotel, Beaconsfield, Bucks., at 7 p.m.

#### Friday 28 October

Bristol Section. "The Strip Coating of Steel with Paint and Plastic Finishes," by Mr. D. S. Newton (Richard Thomas & Baldwins Ltd.), to be held at the Angel Hotel, Cardiff.

Irish Section. "Developments in Media for Printing Inks and Paints," by Mr. G. H. Hutchinson, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8 p.m. Manchester Section. Annual Dinner-Dance, to be held at the Midland Hotel, Manchester.

#### Thursday 10 November

New South Wales Section. "Modern Methods of Milling Phthalocyanine Pigments," by Mr. H. G. Cook (ICI Ltd.). Further details from Section Secretary, Mr. D. F. Crossing.

#### Monday 14 November

Victorian Section. "Some Aspects of Pigment Dispersion," by Mr. H. Cooke, to be held at Union House, Melbourne University, Parkville.

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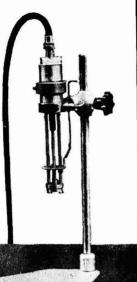
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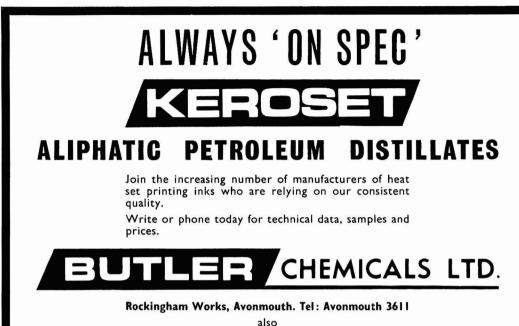
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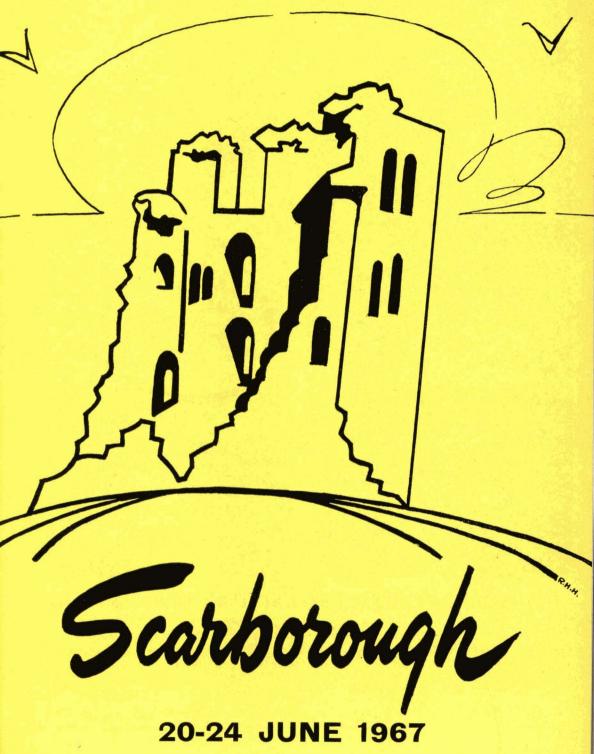
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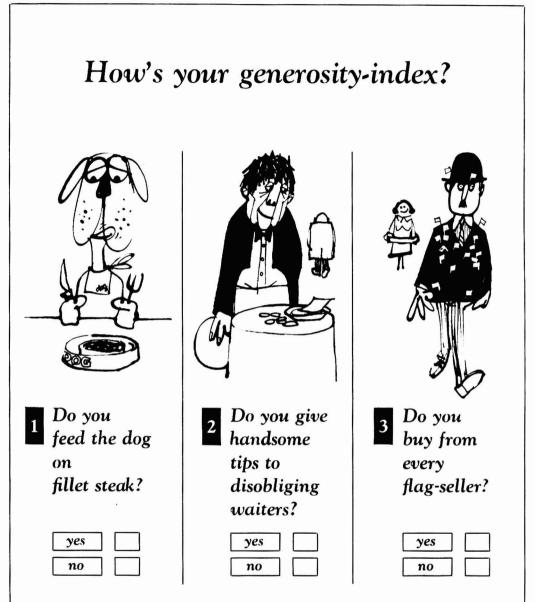
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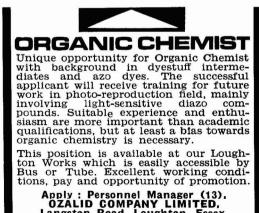
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Australian Sections 0 il & Colour C hemists' A ssociation Vol. 3, No. 2. FEBRUARY, 196 35 **Proceedings** and News

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