Chemistry and Industry

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Chemistry and Industry

Society of Chemical Industry

The Society was founded in 1881 and incorporated by Royal Charter, 1907

Offices of the Society
14 Belgrave Square, London SW1X 8PS
Telephone 01-235 3681

The Society was founded in 1881—
'To advance applied chemistry in all its branches. To afford its members opportunities for the interchange of ideas with respect to improvements in the various chemical industries and for the discussion of all matters bearing upon the practice of applied chemistry and the publication of information thereon.'

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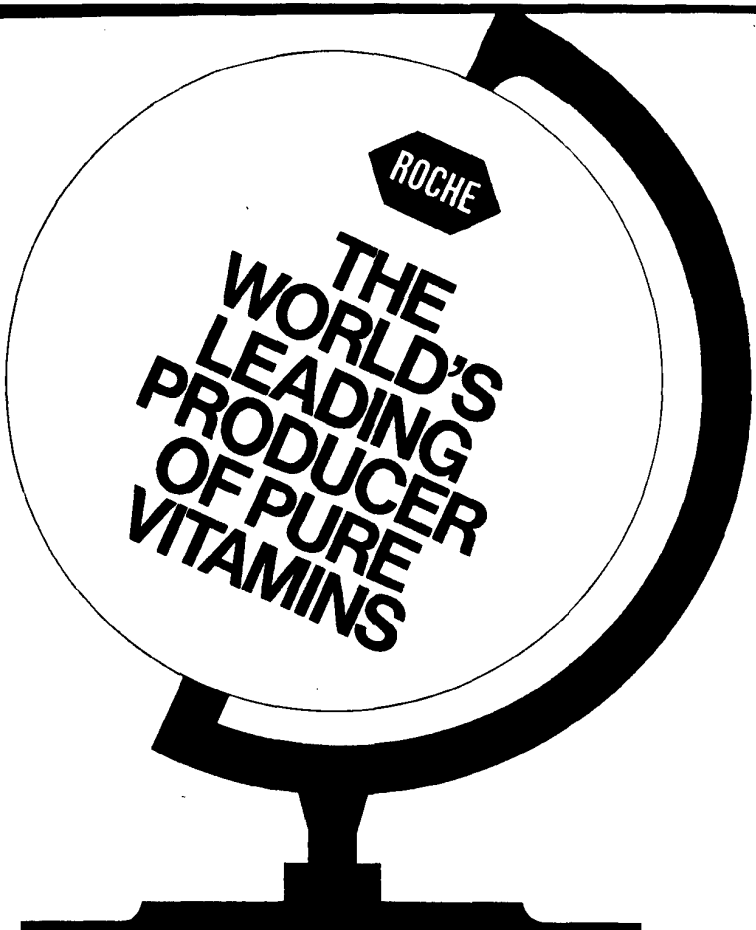

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Editorial

What price priorities?

In the issue of 18 September 1971, p 1051, this journal carried a picture of Lord Sherfield, formerly the distinguished diplomat Sir Roger Makins, shaking hands with Mr George Beeby. The occasion was the opening of BSI's new Sherfield Building and the two were together as president and deputy president of the British Standards Institution.

It is fair to say that the opening of a modest building was of little event in itself: it is what lay behind the opening ceremony which should be causing industry – and ours as much as any – to prick up its ears or adjust its bi-focals.

What Mr Beeby doesn't know about the economic effects of technical rationalisation, particularly internationally, would find plenty of space on a postage stamp: the significant thing is the reaction of Lord Sherfield in the short time that he has become president of BSI. Now a prominent figure in international finance, he has become an ardent propagandist for the Institution in which he holds the highest position. The two are correlated, for there can be little doubt in any one's mind that a major force in Lord Sherfield's enthusiasm is the very rapid realisation that the widespread collaboration which is achieved through BSI means, in the long run, real money, both to those who put standards into practice and to the country as a whole.

Sherfield Building is in the Hemel Hempstead Centre of BSI, a centre which is very largely devoted to testing and approvals within export demands. It holds, for example, the agency for testing electrical and other equipment on behalf of the Canadian Standards Association. These tests are a legal prerequisite to exports to Canada. It has authority from the USA automobile authority to approve a number of automobile parts for entry to the American market.

It is in effect the modern requirement in the rapidly growing trend towards certification and approval of foreign goods and is a decided lever in the pressure to ease the path of exporters throughout the world by establishing reciprocal approvals agreements. When such independent and authoritative centres are testing to internationally agreed test methods and certifying under international authority, the shunting of new designs backwards and forwards between a manufacturer and the country of his market, the time spent in constant trips to iron out technical differences, will be unnecessary.

There is a long way to go yet but the movement has started and gains momentum every year.

Which brings us to a parallel item in the opening ceremony – the establishment of a plaque in honour of the foresight of Mr H. A. R. Binney, now director general of BSI, who, in concert with leading figures in the standards movement like Mr Beeby, saw this turn of events a decade ago.

It was possibly a unique occasion in this field – the recognition of a man in such a permanent way within his own lifetime of service. Three years ago, *The Times* carried a picture of Mr Binney greeting Dr Boitsov, his counterpart in the USSR. Dr Boitsov is now a Minister of the Soviet Council of Ministers, and heads a grand network of standards centres, which includes over 1000 officers solely devoted to information. Like Mr Binney, Dr Boitsov sits on the council of the International Organisation for Standardisation in Geneva and expresses in his own energy in international collaboration the very keen interest of the Soviet.

There must be a lesson here somewhere when a learned standards man becomes a minister in the senior Soviet hierarchy. There must be something to be drawn from the fact that Japan maintains a permanent liaison man at ISO in Geneva. The lesson is not hard to find. In one year Russia saved 8 million roubles on crane production, in the same year 13 million roubles on paper and stationery and these are only two in a list totalling an astonishing financial return.

Yet BSI, and for that matter most of the other national standards bodies, staggers from April to March, cocking an apprehensive eye, first at the economic charts affecting their subscriptions and sales and then at the mood of the government of the day. Not only does it not know where next year's money is coming from, it does not know how *this* year's expenditure is to be met.

The comparisons, at a time when the international pressure is for harmonisation of effort which will save every country untold millions in avoiding dragging themselves around national technical obstacles, are ludicrous. BSI's annual report shows that the Institution took a leading role in the production of 53 ISO Recommendations strictly related to chemicals. The total effort including paints, pesticides, plastics compounds and chemical engineering is large and grows larger if one selects items such as the sampling and chemical testing of leather, petroleum and dozens of associated standards.

In the long run, when these ISO standards are firmly embedded in national standards they can only add up to conservation of skills, of time and of money.

Under the present financial strictures, individual companies can be forgiven for overlooking these facts. But in terms of concerted industrial effort and national concern, there is sufficient evidence that the considerable amount of voluntary effort expended on work of vital importance to industrial efficiency and the export trade is, if not exactly neglected, at least unappreciated.

This is the real caption to the picture of Lord Sherfield and Mr Beeby that was published a short time ago . . . and it is the proper emphasis on the simple building that was opened on that day.

News this week

Society reports and notices

Plastics and Polymer Group

'The physics and chemistry of fracture'

Professor E. H. Andrews, head of the Department of Materials at Queen Mary College, London will be giving a lecture on the above subject at 14 Belgrave Square, London S.W.1 on Tuesday 23 November 1971 at 6 pm. Professor Andrews has supplied the following abstract of his proposed talk:

The study of fracture phenomena in polymeric solids can be approached from any one of three different directions, which could be called the engineering, metallurgical and chemical viewpoints respectively. The engineering or mechanical approach, typified by the fracture mechanics treatment of the subject, considers the solid as a continuum with certain average or macroscopic mechanical properties such as elastic moduli, yield stresses, viscoelastic parameters and so on. Its great value is that it leads to predictive formulae which are useful to the engineering designer wishing to avoid fatigue fracture or stress-corrosion.

The 'metallurgical' or morphological approach recognises the importance of physical microstructure and heterogeneity in controlling fracture properties. For example, a spherulitic specimen of a given plastic may be very brittle, but one crystallised under strain may be tough and ductile. The relationship between morphology and fracture is a developing field of effort and some current work will be discussed.

Finally, fracture ultimately concerns the breakage of inter-atomic bonds, and can thus be regarded from a chemical viewpoint. Extensive studies of 'main-chain' rupture have been made in the general field of mechano-chemistry, but the relation of these events to the fracture of the bulk solid is only just emerging, largely as a result of e.s.r. and related investigations.

E.H.A.W.

Food Engineering Panel

Forthcoming lecture 'The drying and instantising of milk products'

The Food Engineering Panel, Food Group, are holding a meeting on 8 December 1971 at 14 Belgrave Square, London, which will be addressed by **Mr P. Barnard**, of the Milk Marketing Board on the subject of 'The drying and instantising of milk products', and not as may be elsewhere described. All members of the Food Group and their guests are welcome to this topical meeting, which begins at 6.15 pm. R.J.C.

Colloid & Surface Chemistry Group

Forthcoming lecture 'Colloidal aspects of printing inks'

A meeting will be held jointly with The London Section of OCCA on the above subject at 6 pm on Monday 13 December 1971 at 14 Belgrave Square, S.W.1. The Speaker will be Dr W. Carr of Ciba-Geigy. An abstract of the lecture follows:

Colloidal dispersions are characterised by the degree of sub-division of the disperse phase. The lower limit of size is accepted as being 1nm (1 millimicron); the upper limit as being between 0·2 and 1·0μm (0·2 and 1·0 microns).

Particle size measurements on the pigments in printing inks demonstrated that commercial inks are partly in the colloid range. With some inks, their storage properties are directly related to the degree to which they are true colloids. This work would indicate that the upper limit for true colloidal dispersion is 0·3μm.

Inks can be shown to improve in dispersion on dilution. This can be explained qualitatively in terms of modern colloid theory.

The flow properties of inks can be explained partly in terms of modern colloid theory. K.S.W.S.

Pesticides Group

Forthcoming symposium 'Increasing the biological contribution to the control of pests and diseases'

A major symposium will be held in Oxford on 5–7 January 1972 on the above theme. It is being organised on behalf of the Royal Society Sub-committee for Biological Control by the Association of Applied Biologists, British Ecological Society and SCI Pesticides Group. Scientific contributions will review developments in a variety of control techniques, including the use of conventional pesticides and other chemicals. Emphasis will be placed on the effective exploitation of these techniques with due regard to economic and environmental considerations.

Further particulars and application forms may be obtained from the Society of Chemical Industry, 14 Belgrave Square, London SW1X 8PS. D.P.J.

Solvent Extraction & Ion Exchange Group

Forthcoming visit to Permutit and Battersea Power Station

A visit has been arranged to the Permutit Co. Ltd at Isleworth, Middlesex and Battersea Power Station on Wednesday 1 March 1972 at 11 am. In the morning **Mr R. C. Williams**, British Nuclear Fuels Ltd, will give a lecture on 'Ion Exchange and its use in modern electric power stations' at Isleworth. The visit to Battersea Power Station will take place in the afternoon.

Places on this visit are limited and those who wish to attend should write to Mr M. J. Cristal, Head of Technical Information Service, The Permutit Co. Ltd, Permutit House, Gunnersbury Avenue, London W.4 before 31 December 1971.

If sufficient persons are interested an additional visit in the morning and a repeat of the lecture in the afternoon will be arranged. N.M.R.

London Section

Forthcoming visit to Harwell

The whole-day visit to the Chemistry Division (Pure and Applied) at Harwell has been arranged for Thursday 11 May 1972, commencing at 11 am. Further details will be given in the Bulletin about two months before the visit. P.A.S.

Heavy Organic Chemicals and Fine Chemicals Groups

'Impact of organometallics on the heavy organic chemical industry'

A joint meeting of the Heavy Organic Chemicals and the Fine Chemicals Groups was held in the Society's rooms on 19 October 1971 with **Dr J. A. Gardner** (Vice-Chairman, Heavy Organic Chemicals Group) in the chair. Dr Gardner introduced the lecturer, **Dr J. Faesel** of Metallgesellschaft.

Dr Faesel started his lecture with a brief history of the industrial production and application of organometallic compounds, dating from the introduction some 40 years ago of lead tetraethyl and of organic mercurials. The usage of organometallics in the petrochemical industry dates from

the pioneering work of Ziegler on the oligomerisation and polymerisation of olefins. The basic Ziegler chemistry was outlined; on the basis of Ziegler's work (particularly with aluminium alkyls) routes to a number of interesting compounds have been developed, *e.g.* long chain α-olefins, primary alcohols, propylene dimer, ethylene and propylene polymers, polybutadiene and polyisoprene. In 1970, production of aluminium alkyls was 25,000 ton, mainly used in the production of long chain primary alcohols from ethylene; the production of α-olefins by ethylene oligomerisation is not currently competitive with production from n-paraffins. Propylene dimer, the only product from propylene with aluminium alkyl catalysts, gives isoprene and methane on cracking, and about one third of the western world capacity of synthetic isoprene is based on this reaction. The Ziegler high density polyethylene process was described briefly, and the properties of low density and high density polyethylene were compared. The production of polypropylene was developed by Natta with catalyst systems composed of aluminium alkyls plus titanium salts; polypropylene has many applications, and has a rapid growth rate so that by 1975 it was forecast that 20 per cent of the total polyolefin market will be for polypropylene.

In the polymerisation of diolefins to give rubbers, different polymer structures are obtained with different catalyst systems. Lithium alkyls are useful catalysts, and in 1970 some 300 ton of lithium butyl were used in the production of stereo rubbers, mainly from butadiene; stereo polybutadiene is competitive with natural rubber in tyres and mechanical rubber goods. Aluminium alkyls are used in the production of ethylene/propylene terpolymer rubbers which have good properties in many applications. The versatility of the organometallic catalyst systems available allow many varieties of rubber to be produced, and it was forecast that stereo rubbers will grow much more rapidly than SBR, representing about 30 per cent of the market for synthetic rubber by 1975.

In the subsequent discussion, Dr Facsel said that the hazard of handling organometallic compounds were now well understood; lithium butyl was sold as a non-pyrophoric solution. In reply to a question on the use of synthetic rubbers in tyres, he emphasised that polybutadiene and terpolymer rubber were already competitive with natural rubber, but that polyisoprene was not so because of the high monomer cost.

The meeting concluded with a vote of thanks proposed by **Mr H. Hodge**, who complimented the lecturer on his able presentation in simple terms of a subject of considerable complexity. A.F.M.

Newcastle upon Tyne Section

'Safety aspects of natural gas'

A joint meeting with the RIC and the Institute of Fuel was held on Wednesday 13 October 1971 in the School of Chemistry, Newcastle upon Tyne when **Dr P. J. King** of UMIST gave an illustrated talk on 'Safety aspects of natural gas'.

Dr King, who was introduced by **Dr R. N. Parkins**, the Section Chairman, pointed out that the subject matter of the lecture was based on the results of the inquiry into the safety of natural gas as a fuel, which was conducted at the Government's request during 1970. After giving a brief historical survey of the discovery of natural gas and the conversion plans for Great Britain, Dr King spoke of the factors leading to the growth of public disquiet and the subsequent setting up of the inquiry.

The speaker compared the physical and chemical properties of town gas and natural gases and showed that while the two types of fuel were similar they did differ in many respects and the effect of these differences on their relative safety were discussed. Particular attention was placed on combustion characteristics and explosion hazards. The problem of adequate and efficient ventilation for all types of fuel was given special consideration and comments on the advent of natural gas in the north east were included.

The speaker concluded that all fuels were hazardous unless properly used, but natural gas introduced no new hazards when compared to town gas; on the contrary, it removed the greatest single hazard, that of carbon monoxide poisoning due to accidental escape of unburnt gas.

A lively discussion followed in which it was queried how the gas industry intended to overcome the ventilation problem, and whether statutory powers to enforce regular inspection of premises would be involved. Dr King replied that he thought adequate ventilation ought to be provided in the first instance by the gas industry. Dr King suggested that a joint consultative council for safety be set up for the gas, electricity, oil and solid fuel industries.

One speaker mentioned that there was evidence which showed that the chance of explosion with natural gas was greater than that of town gas and he asked what facts and figures were available to substantiate this. Dr King replied that statistics with regard to explosions were minimal and it could not be said that natural gas was more dangerous on the basis of such inadequate information. The gas industry was taking the problem seriously and information regarding gas explosions was recorded *independently by fire officers*.

The meeting concluded with a vote of thanks to Dr King by the chairman of the local section of the Institute of Fuel.

D.A.W.

Appointments and resignations

Mr Robert Carr, Secretary of State for Employment, has appointed **Mr S. H. Lawrence** to be a member of the board of directors of Industrial Advisers to the Blind Ltd.

Mr M. A. E. Hodgson and **Mr S. D. Lyon** have been elected deputy chairmen of the Board of ICI as from 1 April, 1972. At that time they will join **Mr M. J. S. Clapham** and **Mr R. S. Wright**, two of the three present deputy chairmen. **Mr P. T. Menzies** retires from this position on 31 March on taking up the chairmanship of the Electricity Council.

Mr M. A. E. Hodgson

Mr S. D. Lyon

The Badger-Tomoe Co. Ltd, Tokyo, Japan, has appointed **Mr E. H. Martel** as vice-president and managing director.

Mr R. M. Wells, development manager and a member of the executive board of Philips Filtration Ltd, has been invested as chairman of the Filtration Society for 1971-2.

Mr S. W. Gillies, director and general manager of Saro Products Ltd, Isle of Wight, has been appointed managing director of that company.

Mr C. A. Cannon and **Mr G. Gollings** have been appointed to the board of Gloster Saro Ltd, Gloucester.

A well-known authority in this field, **Mr W. A. Roberts** has been appointed field product manager (nuclear) for Beckman-RIIC Ltd of Glenrothes and Croydon.

Pennwalt Chemicals Ltd has announced that **Mr D. Macleod** has been appointed area representative for Scotland, calling on engineering outlets with a range of cleaning and pre-finishing chemicals.

Mr S. T. McGeary has been appointed general production manager of GEC-Elliott Process Instruments Ltd.

Dr D. Newman, chemistry master at Eton, has been appointed by the Salters' Institute to a Sabbatical Fellowship to be held at Imperial College in the Department of Chemical Engineering and Chemical Technology during the Spring term of 1972.

Mr V. H. Wentworth, senior technical specialist - plastics with Monsanto in London, has retired after 17 years with the company.

Mr S. A. Stewart retired as Director of the British Tar Industry Association on 31 October 1971. He is succeeded by **Mr H. Hayman**, Managing Director of Benzole Producers Ltd and currently President of the National Benzole & Allied Products Association.

Mr J. R. King, director of sales of Torpedo Marine Paints, the marine division of the Berger, Jenson & Nicholson Group of Companies, has retired.

Thomas Broadbent & Sons Ltd has announced the retirement of **Mr F. Broadbent** on 30 September 1971, due to continued and serious ill health.

Death

Professor Dr H. A. Boekenoogen, formerly head of the Unilever Research Laboratories at Vlaardingen, Holland, died on 26 October 1971. Professor Dr Boekenoogen gave the International Lecture to the Oils and Fats Group of SCI in 1966.

Company and industrial news

Conoco and Norsk seek Italian offshore acreage

Continentale Italiana S.p.A., the Italian subsidiary of Continental Oil Company, is seeking offshore prospective acreage. The company has announced that, in conjunction with Norsk Hydro Italiana, it is applying to the Italian government for exploration licences in thirteen blocks of Zone C, situated off the coast of Sicily. Norsk Hydro Italiana is the Italian subsidiary of Norway's Norsk Hydro, and will have a one-third interest, to Continentale's two-thirds, in the Zone C Venture.

Urethane elastomers from ICI

The Organic Division of ICI are introducing a new range of eight thermoplastic urethane elastomers which combine qualities of rubber and plastics. The product, 'Daltomold', has the elastic properties of rubber and the toughness associated with plastics produced from polymers. The new products, which mould like plastics and work like rubber, have a wide variety of uses in fields where flexibility and hard wear are the main requirements. They also show resistance to fuels, oils, oxygen, abrasion, tear and general mechanical abuse. The 'Daltomold' series are expected to compete with synthetic rubber and, it is hoped, to replace certain other materials, including metals, in some applications in the car, textile, footwear and engineering industries.

Rapid mineral identification

A service for the expert and rapid identification of a wide range of mineral substances is now offered by Yarsley Research Laboratories Ltd of Chessington in Surrey. Using modern *X*-ray powder diffraction analysis equipment, only a small sample of the unknown substance is required. Identification is assisted by the American ASTM index which records data on over 9000 compounds, in addition to Yarsley's own library of films giving data on over 5000 materials.

Expanded polystyrene sheet process

Omega Plastics Ltd of Canvey Island, with financial backing from the National Research Development Corporation, have produced expanded polystyrene as thin sheets of consistent quality, which can be used for a variety of purposes. Demand for the material is such that Omega is offering the complete process as a package deal and is willing to license patents both in the UK and abroad.

Phillips announces polyethylene licence in Yugoslavia

Phillips Petroleum Company has announced a new high density polyethylene licence agreement with Hemijska Industrija Pancevo, a Yugoslavian firm, who will erect a 50,000 ton/year plant at Pancevo, 20 km from Belgrade. Fifteen companies now have licences to use the Phillips process in the USA and in nine other countries. The licensee plants, together with the Phillips plant near Houston, Texas, now represent one million ton/year of high density polyethylene capacity.

New copper processing methods

Scientists in the Research School of Chemistry at the Australian National University have discovered methods of extracting and refining copper which could change the existing industrial processes. Patent applications have been filed. Announcing this, the Vice Chancellor of the University, Professor Sir John Crawford, said a small unit for developing the practical and commercial aspects of the discoveries had been established. Sir John said that a group of research scientists had found solvents which allowed copper to be processed by other than the conventional methods, together with a process which avoided the slow electro-refining in the purification of blister copper. Apart from possible economic advantages, he said, the new processes may help to reduce pollution. It was hoped that an Australian company would join with the National University in development. *Radio Australia News*

I.C. convert Fulham power station to oil

A contract to convert the boilers in the No 1 boiler house of the Central Electricity Generating Board power station at Fulham from coal to oil firing has been awarded to International Combustion Ltd of Derby. This contract is worth almost half a million pounds. The contract calls for the conversion of six boilers, the first to be completed during late 1971 and the sixth by early 1972. Under the terms of this contract, International Combustion will supply, erect and commission the complete oil firing plant and all conversion equipment. International Combustion have been working for the past two years on a number of oil and gas conversions for the Central Electricity Generating Board throughout the country.

Order for Douglas-Rownson

Douglas-Rownson of Basingstoke (Baker Perkins Group) have obtained a £20,000 order from Meyer Dumore for a palletising and conveying system which is to be installed at Daniel Thwaites brewery, Blackburn.

A.P.V. – Mitchell's Japanese contract

A.P.V. – Mitchell Ltd of Croydon have developed and patented a process for producing pure fluosilicic acid from vapours given off during the vacuum concentration of phosphoric acid. This pure fluosilicic acid, containing less than 100 p.p.m. of phosphoric acid, is then used to produce aluminium fluoride and cryolite, essential raw materials in the production of aluminium. This is a major breakthrough in using unwanted effluent for the production of valuable chemicals. The first plant, consisting of two units, was installed in Japan in 1969-70 for Tohoku Hirov KKK at Akita giving highly satisfactory results. The success of this plant has resulted in a further order being placed for Mitsubishi Chemical Industries for installation later this year.

BOC introduce aircraft engine starting system

A new method of starting aircraft jet engines is being introduced into the UK by the British Oxygen Company in co-operation with Aviation and General Consultants Ltd of Middlesex. The system is based on the 'Cryostart' jet engine starter

which is manufactured in the United States by the Systems Capital Technology Corporation of California. The system involves passing liquid nitrogen over hot aluminium oxide pellets. The unit has a tank of liquid nitrogen supercooled to −185°C. When the engine is to be started, the liquid nitrogen flows into a chamber containing a bed of aluminium oxide pellets that have been preheated to 232°C. As soon as the nitrogen contacts the heater bed it turns into gas, expands instantly and is ducted into the jet engine to turn the turbine air compressors. The 'Cryostart' system has a number of advantages over conventional methods using compressed air.

General

New combined treatment for liver fluke

After several years of trial work a Shellstar molluscicide called 'Frescon' is now available to farmers for spraying pastures to control the mud-snail which transmits liver fluke to cattle and sheep causing liver damage, unthriftiness and in acute cases, death.

'Frescon' is important because, for the first time, farmers can make a two-pronged attack by using it with existing oral treatments. The combination of both methods is calculated to give better results than either used alone. The material will be distributed by ICI Pharmaceuticals Division, Zanil and Nilzan and the marketing organisation necessary to get the integrated treatment into use.

Agricultural Science Institute calls for national policy on primary industries

The Australian Institute of Agricultural Science has called for a national policy on primary industries. The institute said in a special statement that the combined effort of farmers, agricultural scientists, economists, sociologists and politicians was needed to place the rural economy on a sound basis for the next 18 to 20 years. However, the immediate need was to solve short term problems facing the man on the land. It considers a thorough review is necessary. The institute announced that it is setting up a coordinating committee to consider a national policy on agriculture.

Radio Australia News

Willows Francis Group annual report

In his annual statement Mr A. J. Cornforth the chairman of the Willow Francis Group reported a profit before tax of £139,399, and stated that sales and trading profits had shown an encouraging increase during the first quarter of the current year. He stated that many of the company's immediate objectives had been attained, notably the restoration of an upward trend in the sale of speciality products in the veterinary, dental and chemistry divisions.

Glaxo Group annual report

In his annual report, Sir Alan Wilson, chairman of Glaxo Group Ltd, announced that, as had been predicted, sales had increased substantially but profit margins were reduced. Total Group sales, excluding wholesales, showed an increase of nearly 10 per cent over last year, while the trading profit at £24·1m increased by about 1 per cent. The profit after tax attributable to the ordinary stockholders was £12·8m, about 3 per cent higher than last year.

Associated British Maltsters annual report

The new chairman of Associated British Maltsters Ltd, Mr P. Parker, who took over from Mr D. L. Nicolson following his appointment to the chair of the new British Airways Board, announced in his annual report that profits attributable to ordinary shareholders amounted to £704,000, an increase of 67 per cent over the previous year. The annual general meeting of the company will be held on 11 January 1972, but the board is making a second interim dividend of 15 per cent payable on 10 December 1971.

Annual report of the Food, Drink and Tobacco Industry Training Board

The annual report of the Food, Drink and Tobacco Industry Training Board states that the year had been one of both consolidation and development. The major emphasis during the year was the improvement of standards of performance through individual advisory visits, meetings with groups of employers and the development of written guides and recommendations. The resulting improved standards would, it was hoped, form the basis for moving on through what the board has recognised as only the essential first stage of its work, the system based on levy/grant. During the year significant progress was made towards this goal. Discussions were therefore beginning on the feasibility of moving towards a dual role of assessment and advice based on charges more closely related to the amount of time spent with companies.

Turkey's largest filling station

In the recent *Shell Magazine* there is an article about a Shell filling station beside the Topkapi Gate of the ancient city of Byzantium. This station was discovered by Hugh Harvey, formerly of Trade Relations, SIPC.

Since the filling station is situated on the busy London Road it has been very successful and not only are there considerable gasoline and oil sales on the forecourt but also the station has five bays for lubrication service, a washing area that can handle up to 10 cars at one time and a fully equipped garage for repairs. The fuel oil business has also flourished and there are four tankers for customer delivery and five tankers are operated on behalf of Shell Turkey for distribution of Shell products to dealers.

Publications

Petroleum industry catalogue

The council of British Manufacturers of Petroleum Equipment has brought out its latest catalogue entitled *British petroleum equipment and services 1971/72*. The catalogue contains information about British companies who together can supply the whole range of equipment and services required for operations and installations in the oil, petrochemical and natural gas industries. Other literature from the CBMPE includes the weekly information bulletin *CBMPE information service* and the quarterly journal *Oil and petrochemical equipment news*.

Further information can be obtained from the Secretary, CBMPE, 118 Southwark Street, London SE1 0SU.

BSC progress report

The progress report on British Steel Corporation research and development, *Steelresearch* '71, describes some aspects of the Corporation's research for the period 28 February 1970 to 1 March 1971. Although not intended to be comprehensive the report spans for the first time the whole field of the Corporation's current research effort.

Copies of the report are available from the Information Officer, British Steel Corporation, Park House, Park Street, London W.1.

New literature from Nalfloc

New literature, describing a range of fuel and fireside treatments which will help users to pinpoint fuel utilisation and combustion problems, is available from Nalfloc Ltd. The company, jointly owned by ICI and the Nalco Chemical Company, makes a wide range of fuel and fireside treatments specially formulated to do specific jobs.

Further information may be obtained from Nalfloc Ltd, PO Box 11, Northwich, Cheshire.

Adhesives in the packaging industry

A series of 8-page colour booklets from National Adhesives and Resins Ltd, Slough, Buckinghamshire, provides a handy and useful reference for anyone concerned with adhesives for packaging. National,

which claims to be the world's leading supplier of adhesives products for the packaging industry, says that the primary concern is to help a user recognise, select and properly use the best adhesive for the job to be done.

Copies of the booklets can be obtained by writing to National Adhesives and Resins Ltd, Slough, Buckinghamshire.

Factory accidents

A new publication, *Factory accidents: their causes and prevention* (A5, 26pp – price 20*p*) has just been published by RoSPA. It replaces a previous publication, *The causes of everyday accidents in factories*.

Further information can be obtained from RoSPA, Terminal House, 52 Grosvenor Gardens, London S.W.1.

Organic synthesis design

A new booklet, *An introduction to the design of organic synthesis*, has been added to the series of free technical booklets produced by Koch-Light Laboratories Ltd. The publication, prepared by Dr S. Turner, analyses the approach to organic synthesis and also presents a simplified treatment of the analysis which will enable a chemist, using pencil and paper rather than a computer, to consider a specific synthesis in a general way which reveals many of the important problems to be surmounted when benchwork begins.

The booklet is available free on request from Koch-Light Laboratories Ltd, Colnbrook, Buckinghamshire.

Thiokol Chemicals Ltd report

A new technical service report produced by Thiokol Chemicals Ltd Technical Service and Development Laboratory entitled *Evaluation of thixotropic additives in two-part polysulphide sealants*, is now available from Thiokol Chemicals Ltd, Chemical Division, Station Tower, Station Square, Coventry, CV1 2GH. This report is based on two years' evaluation of thixotropic additives.

Meetings, Notices

Ciba Foundation meetings

A list of Ciba Foundation meetings, for the promotion of international cooperation in medical and chemical research, over the next few months can be obtained from the Ciba Foundation, 41 Portland Place, London W1N 4BN.

Chemical education conference

A three day conference entitled 'Developments in tertiary chemical education' is to be held at the Thames Polytechnic on 13-15 December 1971. The aim of the conference is to bring together chemists and educationalists to review and discuss current work in the areas of curriculum development, educational techniques and methods of assessment in chemistry at the tertiary level.

Further details and application forms are available from Mr B. A. T. Bleach, Academic Registrar, Thames Polytechnic, Wellington Street, London SE18 6PF.

Royal Society meetings

The Royal Society anniversary meeting will be held on 30 November 1971 at 2.30 pm. The programme of meetings for November and December 1971 is available from the Executive Secretary, The Royal Society, 6 Carlton House Terrace, London SW1Y 5AG.

Golden jubilee meeting

A meeting to commemorate the golden jubilee of the Department of Biochemistry of the Indian Institute of Science, Bangalore is to be held in Bangalore from 2-7 December 1971. The subjects on which papers are to be presented include lipids, proteins and enzymes.

Further information can be obtained from the Executive Secretary, The Biochemical Society, 7 Warwick Court, London WC1R 5DP.

Urban transportation conference

'Urban transportation' is the theme of the National Conference of the Institution of Highway Engineers, at Church House, Westminster on 9-10 December 1971 for which over 500 delegates have so far registered.

Further details and application forms can be obtained from The Secretary, The Institution of Highway Engineers, 14 Queen Anne's Gate, London SW1H 9AF.

National Physical Laboratory conference

A conference, entitled 'The measurement of high frequency dielectric properties of materials' will be held at the National Physical Laboratory from 27-29 March 1972. The frequency range covered will be approximately 1-300 GHz. A fee of approximately £20 will be charged to cover accommodation, food and conference expenses.

Further information can be obtained from Dr J. Chamberlain, National Physical Laboratory, Teddington, Middlesex before 15 December 1971.

First international packaging conference

Bookings are being taken now for the first International Conference on Packaging Technology, to be held at the London Hilton on 27-29 March 1972. This is the first international conference to be held on the technology of packaging. It is being organised by Pira (the research association for the paper and board, printing and packaging industries) in conjunction with IAPRI (the International Association of Packaging Research Institutes). The conference will provide a comprehensive picture of progress in various aspects of the many technologies involved.

Conference programmes and application forms are available now from the Conference Organisers, Pira, Randalls Road, Leatherhead, Surrey, England.

Pera conferences

Pera, the production engineering research association, is holding four conferences in Melton Mowbray. They are 'Selling technical products to industry' on 30 November-1 December, 'The modern concept of quality management' on 7-8 December, 'Non-destructive testing' on 9 December and 'Metrication – making it pay' on 15-16 December. Further details may be obtained from the Public Relations section, Pera, Melton Mowbray, Leicestershire.

Lighting seminar

A seminar on 'Using the Latest White Light Sources in Commerce and Industry', will be held at the Royal Commonwealth Society, Northumberland Avenue, London S.W.1. on Tuesday 30 November 1971 at 5.30 pm. It is sponsored by The Electricity Council and the Lighting Industry Federation.

Carbohydrate chemistry award

The Council of the Chemical Society, in association with the Carbohydrate Discussion Group, has announced the introduction of a new biennial award in carbohydrate chemistry, to be sponsored by Tate and Lyle Ltd. The award will cover all aspects of the subject. Applications or recommendations for 1970 must be received not later than 31 December 1971.

Copies of the rules of the award are available from Dr J. I. Gibson, The Chemical Society, Burlington House, London W1V 0BN.

Beilby Medal and Prize 1972

Awards from the Sir George Beilby Memorial Fund are annually made to British investigators in recognition of independent original work of exceptional merit carried out over a period of years in the fields of chemical engineering, fuel technology or metallurgy. Work of the nature indicated must be brought to the notice of the administrators not later than 31 December 1971 by letter addressed to the Convener of the Administrators, Sir George Beilby Memorial Fund, The Royal Institute of Chemistry, London WC1B 5DT. Applications made by the candidate should include the names of two referees. The letter should be accompanied by nine copies of a short statement on the candidate's career.

Chemical Industries Association annual general meeting and dinner

In his presidential speech to the annual general meeting of the Chemical Industries Association on 11 November 1971, Mr J. H. Townsend was pleased to publicly acknowledge the support that the members had given to the Association at a time when the industry was subject to outside pressures and influences, and individual members had to be ready to respond quickly and constructively to changing circumstances at home and abroad. This support had been well matched by the skill and effort of the permanent staff of the Association.

Mr Townsend went on to say that the industry must work harmoniously with many external bodies if chemical manufacturing in this country is to make its most effective contribution to national economic activities and to the wider aspirations of society. This means a continuing dialogue with government departments on all aspects of trade, employment and the environment and with trade unions on wages and conditions, and many discussions with the industries' counterparts in Europe in view of the growing involvement in European affairs.

On the economic front there have inevitably been serious problems for the industry as the UK economy has gone through another year without effective growth, and the powerful influences of inflation and trading difficulties have been strongly at work on the international scene. Some of these problems had been foreshadowed by trends established during previous years, but they had become more acute in recent months, and in the climate of uncertainty over international developments, the industry may find itself facing a tendency towards restrictive attitudes.

At home, the output of chemicals is currently only 3 per cent higher than it was a year ago. This is a serious slackening of growth compared with the average of 6·5 per cent/year throughout the 1960s. As a result of a market demand smaller now than was anticipated when the investment decisions were taken in the brisker economic climate of 1968-9 some surplus capacity is now evident, particularly in certain heavy organic fields. The industry is also subject to great pressures on costs of materials, fuels and wages, and increases in these have outstripped any improvements that can be made in productivity.

A more encouraging feature of the current scene is the performance of chemical exports. This is particularly gratifying at a time when surplus capacity in the major producing countries is tending to intensify competition. The volume of UK exports has continued to grow and despite keenness in prices their value for the first half of 1971 was £463m compared with £418m in 1970. Chemical imports showed less growth in the first half of the year and as a result the balance of trade in chemicals has improved.

The Association warmly welcome the decision of Parliament to join the European Economic Community, since it has strongly supported British entry in the belief that the balance of advantage would be decisively in favour of the UK chemical industry.

The Rt Hon. Peter Walker, Secretary of State for the Environment, with the President

As regards environmental problems, a substantial effort both nationally and internationally has been and continues to be made by the industry to overcome them. The industry has had over many years a deep consciousness of the problems of its own hazards, and Mr Townsend felt it significant that despite the hazards of work in the industry the incidence of injury to people working in daily contact with chemicals is currently slightly less than for the manufacturing industry as a whole. This was a subject on which there was a complete absence of complacency.

Mr Townsend concluded his speech by stating his conviction that the Association had the resources and the will to meet any challenges and to compete successfully with the rest of the world.

At the annual dinner held at Grosvenor House, Park Lane, London W.1. on the evening of 11 November 1971, Mr Townsend, in his capacity as President of the Association, welcomed the guests. In his response, the Right Honourable Peter Walker, Secretary of State for the Environment, was pleased to say that rivers and air were becoming cleaner each year, and that he shared in the optimism so far as the future of the environment was concerned. After a difficult period during which an Empire had disappeared, Britain was now entering a phase of growing industrial power supported by world-wide connections, fine institutions such as the City of London, and a resourceful people. Mr Walker then referred to a young generation who were deeply interested in the environment and felt that Britain had the opportunity of combining commercial greatness with a better standard of living. He said that he looked upon the chemical industry as a partner in achieving these aims.

The polychlorobiphenyls, their occurrence and significance: a review

by R. Edwards

H. J. Heinz Co. Ltd, Hayes Park, Hayes, Middlesex

Pollution of the global environment by man is rightly receiving considerable attention because of the increasing realisation of possible long term effects on both the physical environment and living organisms. Clearly it is necessary to distinguish between short term toxic effects and long term ones (*i.e.* reproductive and carcinogenic effects), these latter being more difficult to assess. The chlorinated hydrocarbons used as biocides are well documented in their short term toxic effects, and with knowledge of their facile accumulation in the environment due to their considerable chemical stability, more work is being done on the long term effects coupled with tighter control of their use. With the latter the main points of entry into the terrestrial environment are through crop spraying and seed dressings with consequent accumulation in the soil. From this point they can also reach the aerial environment secondarily by evaporation or 'co-distillation' and carriage in fine particulate matter. They are also systemic in plants to varying degrees. Aqueous run-off carries them through the drainage system together with industrial effluent sources to reach the marine environment above the continental shelves. It is thus possible for these compounds to reach the lower members of the many foodchains. Man's domestic and industrial use of biocides for hygienic purposes leads to entry at various other points in the food chains. The very large concentration factor (10^3) possible in these food chains is well known and in the final members (*e.g.* marine mammals, predatory birds, man) the concentrations can reach alarming proportions. Lipid solubility means that a large percentage of these residues will be stored in the fat depots of the body.

The polychlorobiphenyls (PCBs), with a very widely dispersed use throughout industry, have very similar properties to the chlorinated hydrocarbon pesticides, but are even more thermally and chemically stable. An important difference, however, lies in the routes through which they contaminate the environment. Emphasis here is on industrial effluents reaching the marine medium, and possibly the burning of industrial waste contributes relatively more to the aerial distribution. Although recommended for use in conjunction with pesticides, there is no documented example of this known to the author, so that distribution through crop spraying should not be a contributory factor. This fits the known pattern of distribution of PCBs in the environment, the highest concentrations coming from the heads of fresh water food chains, marine organisms and sea birds. High levels are also known in terrestrial avian predators, but only recently has it been shown[1] that in one region they can be found at levels comparable to those for organochlorine pesticides. While relatively little toxicological work has been done on PCBs and some of the results are superficially conflicting, this can be explained and there can be no doubt that they have very similar effects on mammalian and avian metabolic processes to those of chlorinated pesticides, and indeed that synergistic effects can occur between the two classes.[2] If any control over the use of PCBs is ultimately attempted this will be much more difficult than with pesticides because of the very large number of uses to which they are put.

There exists considerable difficulty in the analytical field because these compounds cannot be separated easily from pesticide residues, and this has probably resulted in quite a few reports in the past giving falsely high results for some pesticide residues.[3] Unknown peaks in gas chromatography analyses were early recognised[4–6] but it was not until Jensen[7] applied mass spectrometry to the problem that some of them were identified as PCBs.

Properties and uses

The PCBs received early notice back in 1881[8] and were in widespread industrial use by 1930.[9] The biphenyls, terphenyls, naphthalenes, and mixtures of these are chlorinated in varying degrees according to the use to which they are put,[10] and are marketed under trade names such as 'Arochlor', 'Phenochlor', and 'Clophen'. The products are complex mixtures of the various compounds and isomers. They range from mobile liquids to crystalline materials or hard resins, and while the lower members are readily soluble in non-polar solvents they become progressively less so with increasing extent of chlorination. They are extremely stable compounds, not hydrolysed by water, acid or alkali, and with a thermal stability such that they are used in fire-proofing. They find use in elastomers, adhesives, paints, varnishes, printing inks, putty and as general fillers. The excellent dielectric properties lead to widespread use for electric insulators and as coolant/insulators in transformers. This stability and their low vapour pressures make them ideal as lubricants, hydraulic fluids, liquid seals, cutting oils and vacuum diffusion pump oils. Employment in packaging materials has led to the contamination of food.[11] There are a host of other uses, but finally it may be mentioned that both the Monsanto Company and the USDH have reported that they act as effective vapour suppressants in insecticide formulations and thus prolong the active life of an application. However, it has not been acknowledged that PCBs have actually been used for this purpose commercially. As an ancillary fact, a patent for the production of PCBs from lindane residues has been granted. This multiplicity of usage has repercussions in the analytical area because

many solvents, some adsorbents and other analytical materials, and even the atmosphere of a laboratory can be contaminated by PCBs.

Occurrence in the environment

It is now well established that PCBs can be found in wildlife in many areas of the environment.[1-12, 26] The highest levels are generally associated with the industrial areas and the adjacent freshwater and marine food chains. The distribution is inevitably not quite so simple as this because of the migratory habits of some of the species involved[19,24] and the fact of widespread aerial distribution.[28] Cohen and Pinkerton[27] reviewed the latter in respect of pesticides, mainly over the North American continent, and found higher levels in dust than rainfall. Risebrough *et al.*[25] studied the carriage of pesticides in the trade winds coming from Europe and Africa by collecting dust from the air over the Barbados Islands, and detected from 1-64 p.p.b. organochlorine residues. Dust collected over the Californian coast contained some thousand times greater concentration of organochlorines, but none could be found in dust collected from over the Central Pacific, although traces were inferred from the nature of the particulate matter. While no PCBs were detected in any of these samples, it was concluded that they were probably present in trace amounts. Tarrant and Tatton,[28] however, who found organochlorine pesticides in rainwater samples taken from all over the British Isles showed that levels were highest in the summer months (*cf.* Risebrough *et al.*[25]) and in populous areas, also found PCBs in all of them although they did not estimate the concentrations. The importance of aerial distribution of pesticides relative to the hydrospheric distribution has been stressed by Risebrough *et al.*, and it is inferred that PCBs have a similar pattern of distribution. However, it would seem that with PCBs more emphasis should be placed on the hydrospheric medium, because an important part in the transfer to the atmosphere, *via* dusts used as carriers in agricultural applications, can be considered as low in this case. If the vapour pressures of DDT and PCBs are taken as being of the same order, it would then be expected that atmospheric levels of PCBs would be low relative to DDT. The relative concentration of pesticides and PCBs in the hydrosphere and atmosphere have yet to be convincingly demonstrated.

The outstanding feature of the distribution is the occurrence in marine organisms on the continental shelves round North West Europe and North America, and also the extraordinarily high levels found in some freshwater avian predators, all associated with effluent from industrial activity.[13,14,16,17,20,21,26] Along the west coast of the USA PCBs have been detected in estuarine and coastal waters in San Francisco Bay, San Diego Bay and Puget Sound (Washington). In areas remote from industrial activity round the Bay of California and parts of the Panama coast for example, birds' eggs contained 20-30μg PCB each, but near the above mentioned industrial locations the eggs contained up to several hundred and sometimes over a thousand μg PCB/egg. The east coast of the USA has yielded similar evidence for contamination by PCBs. Holden and Marsden[13] detected PCBs in seals from the Gulf of St Lawrence in shallow waters (Magdalen Islands) and in the deeper waters of the Cabot Straits. They have also been qualitatively detected in fish and shellfish from Texas and Georgia, and in the south, in the Gulf of Mexico, in estuarine waters at Charleston, S. Carolina, levels of less than 0·1 p.p.m. were found in fish and blue crabs.[20] Duke *et al.* also monitored the results of an industrial leakage into the Escambia River which was carried through several inner bays through to the Gulf of Mexico off Florida. Maximum levels at source ranged from 486 p.p.m. in sediment to 20 p.p.m. in fish, crustacea and sediment in the outer bays. The best indicators of contaminant levels were static organisms like oysters (up to 3 p.p.m.).

Round the coasts of North West Europe the situation is similarly documented. Jensen *et al.*[17] investigated levels in marine ecosystems in Swedish waters. The closed nature of the Baltic area perhaps leads to somewhat higher concentrations than those generally found on Atlantic and American coasts. Levels tended to be lower in waters off West Sweden and to the north of the Baltic proper, in the Gulf of Bothnia, and higher going south from the Stockholm Archipelago to the South Baltic. The levels ranged from 0·002 to 1·0 p.p.m. in mussels and fish up to 150-240 p.p.m. in white tailed eagles. The concentrations were equivalent to the total DDT levels found. Koeman *et al.*[16] qualitatively identified PCBs along the Netherlands coast from the Wadden Sea southwards to, and up, the Rhine, in mussels, fish and birds and their eggs. The uniformity of GC patterns suggested that they all originated from pollution of the Rhine. Holmes, Simmons and Tatton[12] reported the presence of PCBs in marine birds and their eggs collected from round the British Isles, and Holden and Marsden[13] showed them to be present in seals and porpoises round the coasts of Scotland, being highest in those from the east coast (see below). Bonner,[21] investigating the deaths of seal pups on the Cornish coast, found PCBs here (160 p.p.m. in blubber) higher than in comparable northern samples (34-48 p.p.m.), but less than in East Anglian specimens (350-520 p.p.m.). Presst, Jefferies and Moore, surveying PCB levels in British wildlife, reported an average of 4 (1-25) p.p.m. in the eggs of marine birds, and comparing the east and west coasts of Scotland in respect of two sets of twenty five eggs somewhat higher levels (7 as against 3·6 p.p.m.) in the western samples. No PCBs were detected in whales analysed by Wolman and Wilson,[29] and this is compatible with a distribution pattern confined to the continental shelves. However, this may be a rather naive use of the evidence because the water solubility of PCBs, especially the higher chlorinated ones, is extremely low and the bulk of the PCB is more likely to be associated with sediment on the bottom and entering into the food chains mainly *via* bottom living organisms. Most whales feed on plankton, and thus might in any case be expected to show low levels. A further point of perspective is also given by the fact that loss of lower PCBs from aqueous suspensions when aerated has been observed.[49]

Holden[26] has traced aqueous pollution back to one type of source. The work on seals in Scotland having shown that levels were highest in the Firth of Forth region, water samples from the surface and at depth from the estuary and river up to Glasgow showed no detectable amounts

and only trace quantities in trunk sewer effluents. The zooplankton contained less than 0·03 p.p.m., and fish up to 2·6 p.p.m. However, industrial effluents are usually passed to the sewage works together with domestic sewage, and so the sludge, which is dumped offshore, was examined. The fifteen samples analysed ranged from 1-185 p.p.m. PCB on dry weight, with two sewage plants giving consistently high levels (50-160 p.p.m.). Sludge from Manchester exhibited somewhat lower levels, and that from London less again, but taking into account the varying outputs, each area was estimated to contribute roughly 1 ton/year PCB contamination to the marine environment from sewage sludge. In the case of inland areas sludge can be disposed of by spreading over the land surface, and so offer a route to terrestrial food chains. The question of why it is that relatively low levels of PCB are found in man and his domesticated animals, posed in 1967[30] perhaps now has some answer.

Distribution in terrestrial and freshwater ecosystems has largely been assayed in terms of the avifauna because of the effects of organochlorine pesticides and almost certainly PCBs on some bird populations. The DDT/PCB ratio has been used by Risebrough *et al.* as an environmental index,[14,24,25] but when data is compared consideration must be made of the various biological factors, such as tissue distribution, species metabolism, migration, diet, health and age, which all affect residue concentrations. These authors have shown that the DDT/PCB ratio is low, below 2, near industrial areas, and increases with the distance away to become greater than 10 in remote regions. The geographical distribution in terrestrial and freshwater wildlife will be reviewed within the framework of these factors. Risebrough *et al.*[14] examined peregrine falcons and their prey, centring round the Bay of California. Falcons freshly migrated from the Arctic had low levels which increased with residence time in California; the levels in the Californian birds increased with age; and the indigenous adults showed the highest levels. The maximum values in Table I can be used to show the differences in tissue distribution.

The same authors[24] determined chlorinated hydrocarbon residues in breeding pelicans and cormorants in lakes to the north-west of the Great Lakes (Table II). As the birds used the same food sources the clear differences in results demonstrated a species difference probably due to variation in residue levels in the migration areas, but possibly metabolic differences in terms of rates of excretion of organochlorines into the eggs may be involved. Birds have also been studied in Britain[12,19] and the extensive survey (599 samples) by Presst, Jefferies and Moore[19] illustrates some of the factors involved; firstly habitat and migration (Table III). Secondly; the eggs from marine birds from two colonies widely separated from one another demonstrates very convincingly (Table IV) specific differences related to migration habits. The guillemot spends the longest time in coastal waters, the Kittiwake the shortest time there, for breeding purposes only, and the Shag is essentially sedentary. The effect of health on relative and absolute levels is seen in the report by Bonner[21] on seal pup deaths. (Table V).

Table I
Tissue distribution of DDT and PCB in peregrines and their prey

		Total DDT p.p.m.	BCP p.p.m.	DDT/PCB ratio
Peregrines:	unhatched egg	102	10·2	10·0
	muscle	100	98	1·0
	liver	77	57	1·3
	body fat	50	3·2	15·6
Prey:	egg	153	45	3·3
	whole body	59	9·8	5·5
	muscle	26·4	0·098	270

Table II
Chlorinated hydrocarbon residues in pelicans and cormorants

Eggs	pp'DDE p.p.m.	PCB p.p.m.	DDE/PCB ratio
Cormorant	10·4	8	1·3
Pelican	1·7	0·6	2·9
Fish (whole)	0·08	0·5	0·16

Table IV
Specific differences related to migration habits

	DDT/PCB ratio St Abbs Head	Galloway
Guillemot	0·233	0·250
Razorbill	0·500	0·455
Shag	0·625	0·727
Kittiwake	0·666	0·875

Table V
Effect of chlorinated hydrocarbon levels on seal pup health

		Total DDT p.p.m.	PCB p.p.m.	DDT/PCB ratio
Liver:	Cornish	2·4	46	0·05
	Northern (well fed)	0·6	4	0·15
	Northern (starving)	2·0	6	0·33
Blubber:	Cornish	11·1	160	0·07
	Northern (well fed)	9·8	34	0·29
	Northern (starving)	18·1	48	0·38

Table III
Habit and migration factors affecting chlorinated hydrocarbon residues

Bird class	Liver No samples	Liver Mean p.p.m.	Egg No samples	Egg Mean p.p.m.
(a) Terrestrial predators:				
static	75	4·5	115	1·7
partial migrants	49	4·3	6	2·0
migrants	9	1·0	5	1·0
(b) Freshwater (mostly static and mostly herons)	57	345	124	4·7
(c) Marine	4	2·5	96	4·0

One of the most striking features of freshwater species is the extraordinarily high levels in herons at the head of a food chain where up to 900 p.p.m. have been found[19] in British material. As the British heron is sedentary, occurs nearly all over the country, and there are few migrants from the continent, this species might be expected to be a good geographical index of PCB distribution. Unfortunately nearly all the samples obtained in the survey of Presst *et al.*[19] came from the Midlands and South East so that no clear pattern emerged. However, the age factor is well illustrated by this species as the average liver levels for nestlings was found to be 5 p.p.m. while that for adults was 98 p.p.m. Diet is of course an important consideration, and means that different species from the same area can exhibit very different PCB concentrations. Thus, while birds feeding on insects have shown 0·1 p.p.m. (liver), raptors feeding on mammals yielded 0·50 p.p.m. It is necessary then, when comparing or interpreting data, to take into consideration all these factors. Terrestrial restriction of PCBs to near industrial areas would seem to be reinforced by the failure of Holden[26] to find them in freshwater fish in Scotland, and by Davis[31] who failed to find them in beetles, earthworms, slugs and grasshoppers of one agricultural area in England.

To what extent are PCBs found in human beings and foodstuffs? Jensen in 1966 first reported[7] the presence of PCBs in human hair, and later reports[12,23] indicated that levels in man and his foodstuffs were lower than those found in birds, and below 1 p.p.m. One exceptional case has been recorded,[22] however. Recently a very extensive survey of foodstuffs in Sweden sampled between 1967 and 1969, and analysed for organochlorine pesticides and PCB's, has been published.[1]

In a wide range of foods and food materials, PCB levels were found to be below 0·1 p.p.m. (90 per cent of 1400 samples) and comparable to DDT levels. The higher concentrations came from freshwater and marine fish, especially those with a high fat content. Another case where relatively high levels were found was with milk. Cows excrete little DDT in their milk and here all eleven samples were below 0·03 p.p.m. DDT and 0·1 p.p.m. PCB on whole milk. In human milk relatively more DDT is excreted and the average over 22 samples was 0·107 p.p.m. DDT and 0·16 p.p.m. PCB. Other relatively high samples are eggs and butter. (Table VI)

Table VI

	No	*Total DDT*	*PCB*	*DDT/PCB (p.p.m. whole material)*
Human milk	22	0·107	0·16	0·668
Egg, yolk	20	0·21	0·027	7·78
Butter	12	0·1	0·03	3·33

Toxicology

Soon after the beginnings of industrial use of the chlorinated naphthalenes and biphenyls early in this century, human exposure was seen to lead to skin eruptions (chloracne) and in animal inhalation and feeding experiments (1919), it was shown that liver damage occurred with the chlorinated naphthalenes. As a result of three human deaths, investigations were continued in the 1930s, and it was further demonstrated in work on rabbits that in the naphthalene series the higher chlorinated mixtures were the most toxic. The work was expanded by Bennett *et al.*[32,33] who also used mixtures including PCBs, and PCBs alone. A PCB containing 65 per cent chlorine was used, and later one containing 55 per cent. The substances were applied to rats by inhalation, subcutaneous injection, and oral means, and it was confirmed that the high chlorinated naphthalenes were more toxic than the lower ones, but that the PCB was more toxic still. Here, feeding at a lower rate (0·05g/rat every other day) produced extensive mortality, and as with the naphthalenes resulted in yellowing and enlargement of the livers. Liver damage caused by these compounds had not disappeared two months after dosing had ceased. This damage caused the animals to be extremely sensitive to damage by other compounds, *e.g.* carbon tetrachloride (used as a solvent for these mixtures), and much smaller doses than normal were rapidly fatal. Miller in 1944[34] experimented with a PCB containing 42 per cent chlorine and worked on guinea pigs, rats and rabbits, using feeding, subcutaneous, skin and corneal applications. He used single doses ranging from 69 to 690mg and courses totalling up to 1380mg (over up to 54d) and found that guinea pigs were the most sensitive, rabbits less so, and rats least sensitive in respect of liver damage. Damage was largely confined to the liver but in rats there was also hypertrophy of the spleen. The occurrence of hyaline bodies in the liver was confined to the rat. Von Oettingen in 1955[35] found that even 0·05g/d fed to rats caused liver damage. Skin lesions were similar to those found in man.

Man's exposure to these chlorinated hydrocarbons leads initially to dermatitis, then liver damage with resultant jaundice, and Greenburg,[36] reviewing the situation in 1939, concluded that there had probably been at least six human deaths from this cause. Bennett *et al.*[32] had measured the air concentration at thirty industrial plants and suggested a maximum allowable concentration (MAC) of 0·5mg/m^3 for tetrachlorinated and higher chlorinated naphthalenes, with the observation that these levels had certainly been grossly exceeded over the previous twenty years. This work resulted in increased awareness of the hazards from vapours of these compounds and elementary improvements in industrial conditions has largely eliminated this source of danger (but see Biros[22]). The American Conference of Government and Industrial Hygienists in 1953[37] laid down an MAC for the lower chlorinated PCBs of 1·0mg/m^3, and for the higher chlorinated ones ('Arochlors' 1254–) 0·5mg/m^3. This appears to presume parallelism with the naphthalenes, and these figures are perpetuated in handbooks of industrial toxicology,[38] but recent papers[2,45] point to the reverse situation (see below). These MACs are to be compared with those for 'Aldrin' and 'Dieldrin' (0·25mg/m^3, tentative) and parathion (0·1mg/m^3). The oxidised chlorinated biphenyls are more toxic than the parent compounds.[38]

McLaughlin in 1963 published the results of toxicological studies on a number of chemicals which might be found in food[39] using a sophisticated method based on injection into fertile hens' eggs. 25,000 eggs were used over three years and strict controls set up. Among the compounds tested was the

'Arochlor 1242' injected at 0·025 and 0·1ml giving a 0 and 5 per cent hatch as compared with over 95 per cent in the control group. Teratogenic effects were also noted in some of the chicks which did hatch. These results may be compared with those for 'Heptachlor', where 0·05 and 0·10ml of a 1 per cent solution in propylene glycol (non-toxic) gave a 75 and 65 per cent hatch respectively.

Flick *et al.*[40] endeavouring to identify the factor causing the chick oedema disease, following reports that the factor was highly chlorinated and that a plasticiser (PCB) used as a cage wire coating fed at 0·01 per cent or higher caused oedema in chicks, investigated the effect of 'Arochlor 1242'. Groups of twelve chicks were fed on diets containing 200 or 400 p.p.m. over a three week period, changes in weight being noted. Weight gain was lower with the 400 p.p.m. diet and indeed loss took place at the end of the period. Three chicks on this diet died. Here the hydropericardium volume was ten times larger than with the 200 p.p.m. level or the controls, the pancreas, kidneys and adrenals affected, and haemoglobin levels were lower. The PCB produced similar effects to the oedema factor, but was not so potent, and gas chromatography demonstrated that it was not the same.

Two groups of workers, Risebrough, Peakall *et al.* in the US[19,24,41-3] and Jefferies, Moore, Presst *et al.* in Great Britain,[19,44] have actively pursued the effects of organochlorine pesticides and PCBs on birds. The decline in certain bird populations, especially raptors, has been attributed to the presence of organchlorine pesticides, but it is also possible that PCBs have played a part in this, not only because of their intrinsic toxicity but because it has been shown (in insects,[2] and below) that there are synergistic effects between some PCBs and DDT and 'Dieldrin'. Lethal toxicity to the adult bird is not the only factor because it has been shown[14,41] that organochlorine pesticides at the order of one p.p.m. are effective in inducing steroid hydroxylases in avian liver, thus leading to lowered oestrogen levels and reduced reproductive capacity. Added to this DDE has an entirely different effect on the reproductive system, that of causing eggshell thinning, which causes added failures in reproduction. Teratogenic effects may also occur because of the excretion/concentration factor in the eggs. It is thus necessary to review the effects of the organochlorine pesticides on birds in conjunction with those of the PCBs.

Following work by Peakall,[41] which demonstrated that pigeons fed with DDT at 10 p.p.m. and/or 'Dieldrin' at 2 p.p.m. for one week had higher levels of testosterone or progesterone metabolites than controls, Risebrough, Peakall *et al.*[19] compared the effect of DDE, DDT and PCB ('Arochlor 1262') in increasing the levels of polar metabolites in oestradiol metabolism, again using pigeon liver isolates in vitro. Their results are given in Table VII.

Table VII

	Polar metabolites formed (mμ moles)
Control	29
DDE (40mg/kg)	76
DDT (40mg/kg)	93
PCB (20mg/kg)	160

This PCB has about five times the capacity to reduce oestradiol levels than DDT or DDE. The effect of a single dose of DDT may last for over two months. This work was extended to compare the effects of two different PCBs in kestrels, again using liver microsome isolates.[43] The kestrels were fed at 0·5 and 5·0 p.p.m. levels with 'Arochlor 1254' and 'Arochlor 1262', and the results were as in Table VIII.

Table VIII

	Polar metabolites (nmoles/g microsomal fraction)	
	'1254'	'1262'
Control	0	0
0·5 p.p.m.	13·65	19·05
5·0 p.p.m.	60·5	47·95

While these figures do not clearly indicate the relative activity of these PCBs, it has been shown that 'Dieldrin' and PCB are much more active in reducing oestrogen levels than either DDT or DDE.[42] That pesticides reduce oestrogen levels in mammals has also been demonstrated.

Presst, Jefferies and Moore[44] investigated the lethal effects of 'Arochlor 1254' on Bengalese finches. Food containing higher levels of PCB (440 p.p.m.) was refused by the birds (100 per cent mortality not occurring) but it was still possible to calculate the LD_{50} as 253·6mg/kg/d. Seven out of the 38 birds died. Enlarged kidneys was the main feature, with in two cases increased volume of pericardial fluid. While there was a correlation between dose rates and liver concentrations, there was also considerable variation in levels (3-697 p.p.m.). Whether a particular concentration will be fatal will depend, as with organochlorine pesticides, on the condition of an animal. Considerable storage in fat is possible and until this reservoir is saturated, concentrations will probably not rise to lethal concentrations in susceptible organs. So danger can arise in times of stress (*e.g.* starvation or with the breeding cycle) when the fat reserves are mobilised releasing large quantities of PCB or whatever. The liver by itself in these birds may thus not be a good index because the liver fat reserves may vary considerably and will themselves be mobilised ultimately. The authors therefore suggest the brain/liver percentage as a useful index, being high in those birds that died (83·7 per cent and low in the survivors (26·4 per cent). The LD_{50} for DDT is some thirteen times lower than for this PCB, but consideration has to be given to the fact that while the DDT mortality curve is steep (no deaths at 1000 and all dead at 1200mg/kg/d), the PCB curve is shallow, so that at low levels PCB may be more toxic than DDT. The authors conclude on the basis of this experimental work, and reasonably assuming that no drastic change in useage of PCBs is likely to have taken place over the past two decades, that few bird deaths have been caused by PCBs. However they state, without citing evidence, that the PCBs do not vary markedly in toxicity and this is at variance with recent work[2,45,46] (see below), the PCB used here being of relatively low toxicity. Additionally the authors do not consider the possibility of synergistic effects.[2] It was recognised that the position with sublethal effects may be different.

Very little work[20,47-9] has been done on the effect of PCBs (and terphenyls) on aquatic organisms, despite the fact that it is known that some marine crustacea and fish are very sensitive to organochlorine pesticide residues.[44] For example *Crangon vulgaris* (brown shrimp) has an LC_{50}/48h/15°c for DDT of 0.003 – 0·1 p.p.m., and levels as low as 0·0001 p.p.m. may be detrimental to some species of mollusc. One difficulty in the study of toxicity to aquatic organisms is the extremely low solubility of these compounds in water and the difficulty of obtaining sufficiently stable emulsions.[47-9] The use of solubilising agents, of low toxicity, overcomes this. Zitko,[49] using 'Corexit 7664' to solubilise 'Arochlor 1221' and 'Arochlor 1254', carried out 192 hour tests with these compounds using Atlantic salmon parr. He found that concentrations above 2mg/l were lethal. Wildish[48] using 'Arochlor 1254' in emulsions, or solubilising with 'Corexit 7664', carried out 30d tests with *Gammarus oceanicus* and found lethal threshold values of 0·001 to 0·01mg/l ('Corexit') and 0·01 to 0·1mg/l (emulsions). Guthrie *et al.*[47] tested mixtures containing chlorinated terphenyls and their oxidation products and found lethal threshold values of about 5mg/l and pertinently that low oxygen levels in the water enhanced the toxicity. Duke *et al.*[20] who monitored PCB levels in biota and sediment in the Escambia Bay area of Florida as a result of an industrial leakage of PCB into the Escambia River, conducted both acute toxicity tests and 20d bioassays. 48h tests on pinfish and juvenile shrimps at the 100 p.p.b. level was not toxic to the fish but caused 100 per cent mortality in the shrimps. Oysters received a 96h test and the effect was measured in terms of arrest of shell growth. Here 1 p.p.b. resulted in 19 per cent decrease in shell growth and 100 p.p.b. 100 per cent. Resultant whole body concentrations for the three organisms were 17, 3·9 and 33 p.p.m. respectively. It should be noted that the oysters when replaced in PCB free water for three weeks recovered their normal growth rates. In the 20d assays using a concentration of 5 p.p.b., a 72 per cent mortality occurred in juvenile shrimps and 5 per cent in juvenile crabs. It would appear then from all this work that some crustacea and molluscs are as sensitive to PCBs as they are to organochlorine pesticides.

Some toxicological work has been effected using insects.[2,46] Moriarty[2] dosed fourth instar nymphs of *Chorthippus brunneus* (grasshopper) topically with 'Arochlor 1254' at 12·5, 50 or 200µg levels, but found that definite mortality was only associated with the 200µg group plus a possible sub-lethal effect associated with moulting (fat mobilisation). The maximum amounts of PCB in individuals of the latter group were 14µg (male) and 8µg (female). The work of Lichtenstein, however, is much more revealing as to the relative toxicity of the various PCBs and the synergistic effects with DDT and 'Dieldrin'. Groups of 50 *Drosophila melanogaster* were placed in contact with vessels coated with a layer of the substance or mixture of substances, and groups of 15 *Musca domestica* were dosed topically. Tests of the individual substances showed that 'Dieldrin' was some 25-30 times more toxic than DDT, and 1-8000 times more toxic than the PCBs. The higher chlorinated biphenyls, and the terphenyls, gave rise to no mortality in the 24 or 48h tests, but toxicity increased with decrease in chlorine content in the lower half of the series. These results raise doubts as to the relevance of the many papers which have used 'Arochlor 1254', which here has little or no effect under these conditions. The synergistic effects with 'Dieldrin' were positive with some of lower 'Arochlors' and uniformly positive with *pp'*DDT up to 'Arochlor 1254'. However, with the higher chlorinated PCBs and PCTs a reduction of toxicity was observed. While it is improper to transpose results from one phylum to another and short term toxicity may have little bearing on long term effects, nevertheless this paper is a strong indication that considerable care should be taken in planning these toxicological studies.

Work using mammals is equally sparse. A preliminary study by Sabotka,[50] using 'Arochlor 1254' and 'Arochlor 1260' or DDT in single doses of 1-10mg/kg ('Arochlor's) or 0-25mg/kg (DDT) carried out behaviour tests on mice over periods of 4 to 24h after administration. There is a clear difference between the two groups of substances. DDT probably attenuating and PCBs probably enhancing central inhibitory systems. The paper by Bitman and Cecil[45] surveying the oestrogenic activity of DDT analogues and PCBs in terms of the 18h glycogen response of immature rat uterus assumes considerable importance in relation to a lot of the work cited above. The stereochemical requirements are quite strict, and in general for diphenyl-methane and -ethane, and triphenylmethane compounds activity is restricted to those where either the *pp'* positions are vacant or filled by hydroxy or methoxy groups, being inactive where halogen or alkyl groups are present. So the *pp'* compounds perthane, kelthane, DDE, DDD and methoxychlor are inactive. However, *pp'*DDT is active and the *op'*isomer some sixteen times more so, while of the others mentioned only *op'*DDE gains a response. 'Arochlor's 1221-48' are about half as active at *pp'*DDT, but the higher members are inactive. (*cf.* Lichtenstein). In perspective though these compounds only have something like 10^{-3} times the activity of natural oestrogens, but this fits well with the observed pattern of steroid hydroxylase induction in birds, where DDT and PCB are very active but *DDE* is less so. Perhaps another significant point is that the *oo'* and *pp'*biphenols have an equivalent oestrogenic activity. The lower chlorinated PCBs could be degraded in the atmosphere as has been shown by laboratory experiments where the greatest diminution in one gas chromatograph peak was found where PCBs on an aqueous surface were subject to UV irradiation.[51] At the same time it has been observed[12,17,51] that the lower chlorinated PCBs tend to be absent in wildlife samples, particularly in birds, and it may be speculated to what degree there may be differential excretion or *in vivo* breakdown, and what effect this has. A recent study by Vos *et al.*[52] reveals another important aspect. They discovered that two out of three 60 per cent chlorinated PCBs contained highly toxic oxygenated impurities. These two commercial preparations ('Phenochlor DP6', 'Clophen A60') when injected into hens' eggs at the 3·5mg level caused almost 100 per cent mortality whereas the third sample ('Arochlor 1260') gave a relatively low mortality. A fraction containing no PCBs was isolated from one of them by 'Florisil' column chromatography and shown to be toxic.

Gas chromatography and mass spectra showed this fraction to contain tetrachloro and pentachloro-dibenzofurans which were, however, masked by hexachloronaphthalene and heptachlorobiphenyl respectively, thus limiting the accuracy of quantification. The pentachlorodibenzofuran was estimated to be at about the 5 and 20 p.p.m. level in the two samples. That PCB exerted a similar effect to that of the chick oedema factor (CEF) had been noted previously,[40] and that toxicological studies using PCBs had produced variable results. 1,2,3,7,8,9,-hexachloro dibenzodioxin has now been identified as one of the oedema factors[52] and it may be recalled that the toxic factor in the weedkiller 2,3,5-T was shown to be a tetrachloro dioxin.[53] In fact these chlorinated dioxins and dibenzofurans are very highly toxic groups of compounds. 0·2μg pentachlorodibenzofuran is needed to cause 100 per cent mortality in hen eggs as compared to 0·05μg of hexachlorodibenzo-*p*-dioxin. The occurrence of the dibenzofurans in PCBs is associated with the use of sodium hydroxide in the distillation of erude PCB. Pentachlorophenol (PCP) and 2,3,4,6- tetrachlorophenol are possible precursors of CEF and have been found to be present in toxic oleic acids.[54]

It is evident then that other compounds besides the PCBs themselves should be sought for in wildlife and that individual compounds as well as commercial preparations should be used in toxicological studies. Unfortunately the individual PCB compounds are not readily available.

Holden and Marsden[55] have already noted the presence of unidentified polar compounds in wildlife extracts. It is to be hoped that the current FDA programme[56] on the toxicology of PCBs takes into account all these factors.

Analysis

PCBs are extracted together with chlorinated hydrocarbon pesticides in routine residue analyses,[57] and being complex mixtures of compounds with GC retention times on non-polar columns almost identical to a number of pesticides, notably of the DDT group, present an analytical problem which is not completely soluble by gas chromatographic means alone. Chemical conversion of the pesticides, leaving the PCBs almost unchanged, provides a partial but not completely satisfactory solution, but group separation is fundamentally sounder, this of course providing additional evidence of identity. Both thin layer chromatography and column chromatography have afforded quite good partial separation of PCBs from pesticides and the best scheme[58] claims to leave only 'Aldrin' with the PCBs, with which there is very little interference by the PCBs. A number of workers[18,22,59,60] have confirmed the presence of PCBs in living tissues by means of mass spectrometry, but apart from this means the quantification of PCBs presents severe problems because of the large number of compounds involved and that the individual compounds are not readily available, so that most authors have only been able to calculate approximate figures.

Gas chromatography retention data for PCBs is now adequately documented[12,16,18,26,50,61] (and also[1,2,13,14,20,24,25,30,59]) and on non-polar columns the main region of interference of PCBs and organochlorine pesticide residues with one another may vary from the point of emergence of *pp'*DDE that of *pp'*DDT. PCBs may continue to emerge with retention times up to four or five times that of DDT. It has been noted in this laboratory that response to PCBs relative to organochlorine pesticides can be small on some columns, and from the viewpoint of determining pesticide residues, can ameliorate the situation. However, using more polar columns, *e.g.* 'QF-1' or 'XE-60',[12,23,59,62] PCBs emerge largely prior to the DDT group, which allows simple quantification of the latter, but may still leave an unsatisfactorily complex chromatogram. Improved resolution of PCB compounds has been claimed[63] using novel stationary phases.

One alternative has been to alter the retention time of pesticides by derivatisation, leaving the PCBs intact, and of course aiding the identification of the pesticides. Jensen[59] used a mild nitration technique adapted from Erro *et al.* and originating from the Schechter-Haller method, claimed that the PCBs remained unaltered while members of the DDT group were effectively removed, although other organochlorines (*e.g.* 'Toxaphene', heptachlor epoxide, lindane and BHC) are not removed. This scheme (1:1 HNO_3/H_2SO_4 for 5min at 0°c) was followed by Risebrough *et al.*,[14,50] but Reynolds[61] found that DDT derivative peaks ultimately emerged on the chromatogram making for an unprofitably long analysis time. Moreover, some lower chlorinated PCBs appeared to have been affected by the procedure. Saponification with alcoholic potash[13,14,50] leads to the dehydrochlorination of *pp'*DDT, *pp'*TDE, and 'Toxaphene', with the derivative peaks appearing earlier in the chromatogram, and the PCBs reportedly unaffected. BHC is destroyed by this procedure. PCBs have also been differentiated from DDT using carbon-skeleton chromatography.[64]

Separation prior to gas chromatography is to be preferred not only because simpler chromatograms can be obtained but because this will give nearly as much evidence as to identity as derivation. Thin layer chromatography, reverse phase paper chromatography[12] and column chromatography afford means of obtaining partial separations of organochlorine pesticides from PCBs and fractions can easily be obtained where only 'Aldrin', 'Heptachlor', and *pp'*DDE remain with the PCBs. The former two are not very important in respect of the gas chromatography separation because there is usually little interference between them and PCBs, but *pp'*DDE can emerge with the major PCB peaks using non-polar columns. Thin layer chromatography has given this sort of separation using silica gel[15] and alumina,[1,2] and in column chromatography Holden and Marsden[26,45] claimed good separation of PCBs using either alumina, silica gel, or both in series with long narrow columns. Reynolds[61] documented a separation with 'Florisil', while Bevenue and Ogata[62] commented on their failure to reproduce this work in terms of pesticide separations only, and noted the differences in activity between 'regular' and 'PR Florisil'. This reviewer has found no difficulty in obtaining this separation using 'Florisil'. Armour and Burke[58] in a carefully documented paper showed that it was possible to carry out a separation leaving only 'Aldrin' with the PCBs. For this they used silicic acid partly deactivated with 3 per cent water mixed with celite and eluted the

PCBs ('Arochlor's 1254 and 1260') with petroleum ether. 'Arochlor 1254' needed the larger volume of eluant and the separation was more critical. The cautionary note is added that any trace of polar solvent in the sample will make the separation impossible and *pp'*DDE will emerge with the PCBs. Also, the separation is only possible provided that the column (20 ± 5g) is not over loaded (*i.e.* not exceeding 20μg PCB). This then solves the main separation problem leaving on non-polar gas chromatography columns only a small PCB peak which may interfere with the estimation of 'Aldrin'. These authors have also documented the separation of chlorinated naphthalenes.[65]

Confirmation of identity has been achieved by combined gas chromatography/mass spectrometry,[18,22,59,60] but generally quantification is not simple because of the large number of compounds involved and because these are not readily available individually for reference purposes. If it can be assumed that a fraction contains only PCBs (*i.e.* in a column chromatography or gas chromatography effluent) and that the composition on a gas chromatography trace conforms to that of a commercial mixture, then microcoulometric estimation will give an answer. Jensen *et al.*,[17] using a combination of mass spectrometry, ECGC and microcoulometry obtained results which were only accurate to about a factor of two. With the same assumptions electron capture detection can be used, referring to commercial PCB mixtures[20] either by estimating the total peak area[19,59,63] or basing the calculation on one[1,16,19] or more[20] suitable peaks. *pp'*DDE or 'Dieldrin' may be used as an internal standard.[16,50] One empirical approach calculated one PCB peak as *pp'* DDT and multiplied by a factor of ten to obtain a result;[24] another[66] took the total area:weight ratio to be equivalent to that for DDE; and another [19,50] assumed the response of each peak to be equivalent to DDE and applied a correction factor from the use of microcoulometric detection (the authors did not have permanent access to a microcoulometric detector). Gregory[67] studying the electron capture coefficients of mono- and dichloro- and methylbiphenyls showed that there is over a twentyfold variation between the chloro compounds. Zitko[49] determined the concentrations of PCB in fresh and salt water by means of ultraviolet spectrophotometry and fluorescence. While the lower chlorinated PCBs can be prepared easily[68] and only simple mixtures formed when using the Ullmann reaction, the higher members are more difficult to make singly.

Discussion

The decline of certain bird populations was associated with the use of 'Dieldrin' seed dressings and has been attributed to lethal toxic effects. With increasing awareness of these dangers, better monitoring and usage control, at least in developed countries, lethal toxic effects are progressively less likely to occur. Rather, the significance of PCBs (and pesticides) to man and other organisms must now be seen in the context of sub-lethal effects having long term influence on populations through the media of reproductive processes (reduction in fertility, delays in breeding), teratogenic and carcinogenic effects on both the parent and later generations. LD_{50} tests, even over a long period, are irrelevant from this viewpoint. Certainly it is true that severe mammalian liver damage and death can result from high PCB levels but these are only reached in a few foodchain heads, and the normal levels found in man are not of this order. To be set against this, however, is the finding (in LD_{50} terms) that while with DDT there appears to be a definite 'no effect' level this is apparently not true for PCB. Precisely what compounds are responsible for the toxicity of PCBs is not yet clear, but it seems certain that it concerns the lower chlorinated compounds and probably the oxidation products *in vivo* or *in vitro*. It also emerges that impurities in some commercial mixtures are certainly highly toxic, and if the fact of synergistic effects, though demonstrated as yet in one area only, is taken into account then the picture becomes a very complicated one. Clearly it is extremely difficult to carry out strictly controlled toxicological studies at a feeding level of the order of 1 p.p.m. over a long period, and in terms of multigeneration tests. The need for multigeneration tests may be related to DDT, where, despite many years study, it has only recently been indicated that, with only the original parents dosed, carcinogenic effects may occur in later generations (6th generation mice). DDT may again be used to illustrate the sort of effect which may be looked for. In California, Oregon and Washington, genetic changes in the fruit fly population have been shown to result from heavy spraying programmes in these areas, and in the latter two states were tied to two specific forest spraying programmes. The pattern of these genetic changes can also be correlated with spread by winds over long distances. With PCBs the major dispersal medium is probably water, but otherwise there are no essential differences in the situation. The PCBs are spread all over the continental shelves which are fed by industrial effluents.

The toxicology of the weedkiller 2,4,5-T[53] and of mercury residues[69] has recently come to the fore and they present some parallels with the PCBs. While in contrast to PCBs there is a definite, but small, contribution to agricultural pollution, by mercury from seed dressings, the industrial contribution must lead to a similar marine distribution to PCBs, although as yet very little is known about this area. Mercury, in contrast to PCB, is only cumulative in living tissue to a limited extent, and there is a definite natural contribution from the sea floor. The massive aerial use of the weedkiller 2,4,5-T as a defoliant by the Americans resulted in teratological as well as direct toxic effects due to traces of a chlorinated dibenzodioxin. It is as well that PCBs are not used in such fashion because it has now been shown that some commercial preparations contain traces of chlorinated dibenzofurans which are also highly toxic.

Water pollution is receiving considerable attention within the traditional framework of oxygen deficiency, reduced and oxidised nitrogen, temperature and so on, but the type of pollution problem presented by PCBs, DDT and so on is largely ignored. In a recent official report on toxic chemicals, which recommends further work to appraise the situation in fresh, estuarine and coastal waters, there appears to be a large conceptual gap. Commenting on the presence of DDT in raw sewage, this is dismissed as a significant source of riverine pollution because DDT is solid attached and largely remains in the sewage sludge, but what happens to the sludge is blithely ignored. The ironic situation is of

course that the sludge is commonly dumped in estuaries or even spread out over the land so that pollutants such as the PCBs are recirculated through the food chains.

These are but a few aspects of environmental pollution which can ultimately lead to man's own detriment. In many cases the users of pollutant materials, industrial or domestic, have no conception of their own insidious contribution to pollution, and in the case of the very useful PCBs it is very difficult to see how their disposal can be controlled to minimise pollution. From the point of view of restricting types of use to those least likely to result in pollution, one producer, Monsanto, has made a very responsible move[70] in this direction, but as PCB manufacture is no longer covered by patents this may not be effective. It is obvious that with their ubiquitous presence the effects of PCBs and associated compounds must be investigated with great thoroughness. It is not only to preserve bird populations or other wildlife but to prevent long term changes in man himself that such searches are directed.

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Laser Raman spectra of drying oils and alkyd resins

by L. A. O'Neill and N. A. R. Falla

Paint Research Association, Teddington, Middlesex

Laser Raman spectroscopy is proving an important complement to infrared spectroscopy in the polymer field.[1] In this paper is reported a comparison of laser Raman spectra and infrared spectra of some drying oils and oil-modified resins used in the coatings industry.

The laser Raman spectra were recorded in the Division of Chemical Standards of the National Physical Laboratory.

Materials and experimental

The samples examined included linseed oil, tung oil and a pentaerythritol linseed *o*-phthalic alkyd resin.

The Raman spectra were recorded on a 'Spex' Model 1401 double grating laser Raman spectrometer, using a 'Spectrophysics' Model 140 argon ion laser with excitation at 488nm. The samples were examined in glass capillary tubes with 90° illumination.

The infrared spectra of the samples as thin films on KBr plates were recorded on a 'Unicam' SP100 spectrometer.

Results

The comparative infrared and laser Raman spectra are shown in Figs 1 to 3 and present the following features.

Linseed oil

The infrared spectra of unsaturated glyceride oils show strong bands at 2850 and 2920cm^{-1} which, together with medium intensity bands at 1460, 1375 and 720cm^{-1}, are assigned to aliphatic C–H vibrations. The ester groups present give rise to the strongest bands in the spectra at 1748, 1239, 1155, 1100 and 1020cm^{-1}. The methylene *interrupted cis* unsaturation in the chain does not give rise to any strongly active infrared modes, the major band being the medium intensity absorption at 3008cm^{-1}.

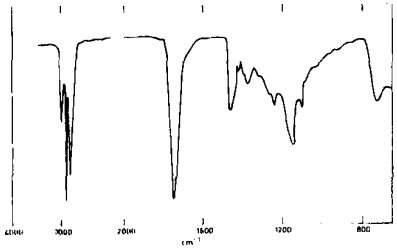

Fig. 1a *Infrared spectrum of linseed oil*

There are several bands in the infrared spectra of triglycerides which are common to the Raman spectra, although their frequency may be slightly different. These include the bands at 3008, 2920, 2850 and 1748cm^{-1}.

The ester bands are very much stronger in the infrared than in the Raman spectra; in particular the 1748cm^{-1} C=O stretch, the strongest infrared band, is only weakly Raman active, while the 1239, 1155, 1100 and 1020cm^{-1} bands are all absent in the Raman spectra.

The main feature in the Raman spectrum not found in the infrared is a very strong band at 1659cm^{-1} characteristic of unsaturation. There are, in addition, bands at 2966, 2906, 1442, 1304 and 868cm^{-1} due to aliphatic C–H vibrations and two ester bands at 1267 and 1077cm^{-1}.

The ratio of the intensity of the unsaturation band at 1659cm^{-1} to that of the aliphatic band at 1442cm^{-1} was determined for linseed in comparison with soya bean oil. From these ratios it was possible to obtain an assessment of the relative degree of unsaturation of the samples. The value obtained was in agreement with that determined by the Wijs' iodine method.

Tung oil

The infrared spectrum is similar to that of linseed oil. The main differences are associated with the *trans-trans-cis* conjugated triene structure present in tung oil. This structure gives rise to a strong infrared band at 990cm^{-1}, together with less intense bands at 960 and 3010cm^{-1}.

The effect of this conjugated structure is even more pronounced in the Raman spectrum. The strong unsaturation band occurs 20cm^{-1} lower than in linseed oil, at 1638cm^{-1}. The relative intensity of this band is also greatly increased (far more than would be expected from the

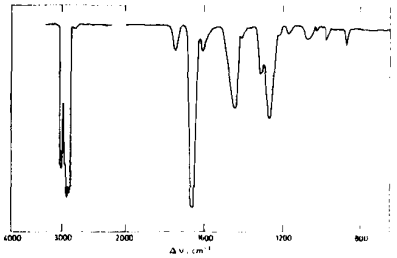

Fig. 1b *Laser Raman spectrum of linseed oil*

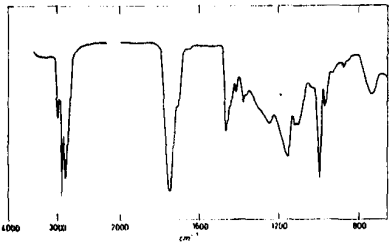

Fig. 2a *Infrared spectrum of tung oil*

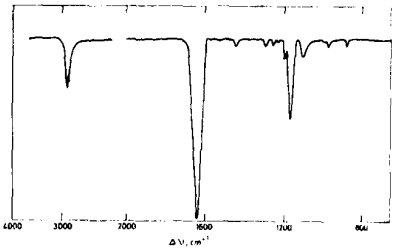

Fig. 2b *Laser Raman spectrum of tung oil*

higher total unsaturation of the oil).

Many more ester vibrational modes are Raman active than in the non-conjugated oils, giving medium intensity bands at 1291, 1255, 1230, 1195 and 1100cm^{-1}, together with a very intense band at 1165cm^{-1}.

Linseed oil alkyd

The infrared spectra of the linseed oil alkyd shows the characteristic features of the unsaturated oil. The principle changes that occur in the spectrum when the oil is incorporated into an alkyd are a frequency drop of the ester carbonyl stretch from 1748 to 1740cm^{-1}, and the loss of the 1239cm^{-1} ester band. The 1270cm^{-1} ester band, which is not infrared active in the oil, appears as a strong band in the corresponding alkyd.

The diagnostic infrared bands of the phthalic portion of the alkyd are at 3060, 1600, 1580, 1075, 1040, 740 and 705cm^{-1}.

The presence of pentaerythritol in the alkyd does not give rise to any major characteristic infrared bands. The 1385cm^{-1} band has, however, a slightly higher frequency than in the corresponding mode in a glycerol alkyd.

The differences between the Raman and infrared spectra of the triglycerides noted in the previous section are also seen in the spectra of the corresponding alkyd. As in the infrared, several changes are noted in the Raman spectra of the triglyceride portion of the alkyds. The very strong unsaturation band is shifted down a few wave number values to 1654cm^{-1}, while the 1165cm^{-1} ester band, which is inactive in the oil spectrum, is now present as a weak band in the corresponding alkyd.

The aromatic bands at 3060, 1600, 1580 and 1040cm^{-1} are common to the Raman spectra, those at 1600 and 1041cm^{-1} being of higher intensity than their infrared counterparts. The 1075, 740 and 705cm^{-1} bands are not Raman active.

The characteristic pentaerythritol modified band at 1385cm^{-1} is also Raman inactive.

Other alkyds examined included a saturated acid alkyd, in which the unsaturation band at ~1650cm^{-1} was essentially absent, and an alkyd prepared with sebacic acid in place of phthalic acid, in which the aromatic bands were absent.

Discussion and conclusions

The results show that in the field of drying oils and alkyd resins, the laser Raman spectra fill an important gap in the structural information given by infrared spectra.

While in the infrared spectra there is no characteristic band for total unsaturation, only for *trans* unsaturation, the band for total unsaturation in the Raman spectra is one of the strongest bands. For the non-conjugated glyceride oils the band can be used as a quantitative measure of unsaturation and the intensity of the band in the alkyd gives an indication of the residual unsaturation. With a conjugated

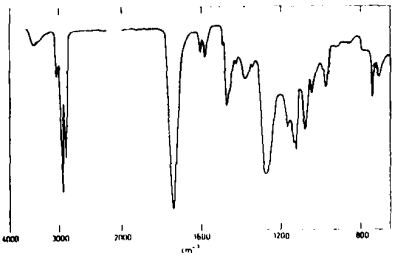

Fig. 3a *Infrared spectrum of pentaerythritol linseed o-phthalic alkyd resin*

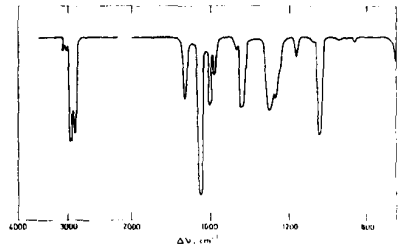

Fig. 3b *Laser Raman spectrum of pentaerythritol linseed o-phthalic alkyd resin*

oil the unsaturation band is greatly enhanced, dominating the spectrum, as it does on the ultraviolet region.

In contrast, the main ester carbonyl band, which is one of the strongest in the infrared, is weak in the Raman spectrum, and minor carbonyl bands are barely discernible.

Hydroxyl groups normally give only weak bands in the Raman spectrum and this region (around 3500cm^{-1}) has not been explored.

Several of the aromatic bands are common to Raman and infrared, and a few are stronger in the Raman spectrum, which may prove of value.

Reference

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British Pest Control Association annual dinner

The British Pest Control Association's annual dinner at the Park Lane Hotel on Tuesday 2 November 1971, attracted nearly 100 members and guests who came to hear this year's Principal Guest Speaker Sir Basil Engholm, Permanent Secretary of the Ministry of Agriculture, Fisheries and Food.

In the course of his address, Sir Basil referred to the changes in the work of the ministry which were announced in the government's white paper last January. With a view to reducing the burden on the tax payer, the ministry's pest control activities will be limited to those which can be carried out only by the government and which are essential to the public interest. Rabbit clearance societies were no longer eligible for grants and he thought that this would undoubtedly offer an opportunity for increased pest destruction activities for BPCA member companies. Furthermore, the white paper also announced a significant reduction in the ministry's advisory work on pest control and in future regional staffs will not be going out and about nearly as much to give on the spot advice on pest control. Although the ministry will keep on inspecting imported food cargoes for pests, because it has always been an important function of the government to defend this island against invasion, it will be up to commercial organisations themselves to pay for keeping pests under control in future. In the same way, the ministry will no longer normally go out to individual farms to advise on pest control unless they have statutory duties to perform.

In his reply, the President, Mr A. Fraser McIntosh, expressed his very great pleasure in being able to welcome Sir Basil Engholm to a BPCA Dinner for the third time, a fitting climax to what he described as a vintage year for the association. He referred to the annual dinner being this year made more glamorous by the presence of the ladies associated with the industry. Mr Fraser McIntosh, in referring to the recent major government decision on the EEC, also mentioned that during last month's British Pest Control Conference in Jersey he had started to investigate the formation of a European Federation of Pest Control Associations. He felt confident that Britain had a great deal to offer Europe in pest control techniques and standards of operation, and one of the main reasons for this was that during the last 25 years the industry has been in the unique position of pooling its knowledge with that of ministry officers.

Standing at the British Pest Control Association annual dinner is the President, Mr A. Fraser McIntosh, on whose right is the principal guest speaker, Sir Basil Engholm, the Permanent Secretary of the Ministry of Agriculture, Fisheries and Food. On his right is Dr A. G. Fisken, Vice-President of BPCA who proposed the toast to the guests, and on the President's left is Dr M. B. Green, the Chairman of the Pesticides Group of the Society of Chemical Industry, who replied on behalf of the guests.

The President ended his speech by expressing the sincere thanks of the BPCA to the ministry for the tremendous contribution to the Jersey conference by officials and scientific officers of the ministry, not only in the papers they presented, but in the vital part they played in discussions throughout the conference. This lent authority and emphasised the close co-operation and invaluable support given by HM Government and greatly impressed the overseas delegates.

C. J. KELBEL

Essay review

Synthetic methods of organic chemistry

by P. D. Magnus

Dr Magnus has been at Imperial College for the past ten years, first as an undergraduate, then studying for a PhD under Professor D. H. R. Barton. In 1966 he was appointed as an assistant lecturer in the second year of his PhD, and in 1969 he was appointed to the position of lecturer. During the past five years he has been working with Professor Barton and they have published several papers, notably in the area of organic synthesis. Dr Magnus' own research interests are in the study of new reactions and the synthesis of natural products, especially terpenes.

The latest volume of *Synthetic methods of organic chemistry** is number 25, completing the fifth series, and marking the documentation of synthetic procedures for the past 25 years. Before describing the achievements of this volume a brief glance at what synthetic organic chemists have been doing in the past, and more recently, might serve to illustrate the complexity and magnitude of the task that Dr W. Theilheimer has undertaken.

Organic synthesis, over the past hundred or so years has remained one of the main pursuits of organic chemical research. It must be said to be the life-blood of organic chemistry, supplying new reactions and the factual material upon which the theories of organic chemistry are based. Indeed, it would be no exaggeration to say, without organic synthesis there would be no organic chemistry at all.

Like the other branches of organic chemistry the objective of synthesis has not remained static in its outlook. It has changed since the early days of structural verification, now the domain of the *X*-ray crystallographer, to a search for new reactions that not infrequently (witness the Woodward-Hoffman rules) change the whole face of organic chemistry.

When we are asked to assess a paper or book concerned either with a specific synthesis, or the development of new general synthetic methods, the usual objective discipline of science can be extremely difficult to apply. We are approaching the subjective realm of aesthetic values and consequently many conflicting views may ensue. A particular synthesis may be said to be extremely useful in a commercial assessment, yet reveal nothing new or particularly stimulating to the academic scientist. Likewise some academic synthetic work that unfolds new reactions or enables fundamental steps to be taken in theory, are useless to the commercial world. Possibly there is only one real way of judging synthetic work and that is in its usefulness and generality. Does it enable one to do what could not be done before? Does it improve on what could be done before? Naturally the scientist's integrity must be absolutely honest in the assessment of what he has done. Any new synthetic methods that are reported in the chemical literature, to warrant serious appraisal, must attempt to define the scope, limitations and of course the yields of products obtained (preferably of isolated products). The practising synthetic organic chemist is then in a position to say whether this new synthetic method is useful to him personally or to synthetic organic chemists in general.

In the past the bulk of chemical reactions were discovered in the course of often long and tedious degradational work to elucidate structures. This wholly admirable pursuit uncovered a vast number of reactions, often unexplained at the time, and also provided the structures of a large number of natural products. The next most obvious step was to confirm the allotted structure by an unambiguous total synthesis. As the natural products presented more formidable structures the art of synthesis became more independent of the initial structural elucidation. Eventually with the advent of sophisticated spectroscopic methods and *X*-ray crystallography, synthesis has grown into an independent science.

This renaissance of organic chemistry has at once enabled the organic chemist to become more productive and creative. Synthesis is no longer a method of confirmation but a creative science seeking to understand and develop new methods of making and breaking bonds between atoms.

The documentation of the achievements of synthetic organic chemistry is an awe inspiring and monumental task. But no one can doubt the necessity of recording the reactions that have been used, with varying degrees of success by organic chemists, into a single series of books. It is one thing to see the necessity, quite another to execute it successfully. The necessity of documenting synthetic methods was recognised by Dr W. Theilheimer in the ambitious task of producing an annual coverage of syn-

* 'Synthetic methods of organic chemistry', 1971, Vol. 25, Yearbook 1971, by W. Theilheimer, pp xvi + 707, *John Wiley*, Sfr296, $71·05, DM296.

thetic methods. The first volume of 'Synthetic methods' appeared in 1946, and its title describes precisely what it is; a compilation of synthetic methods. The subsequent annual volumes have been divided into five series. Each five volume series is terminated with an index of reaction titles and a cumulative index. Consequently in 1971 we have arrived at Volume 25, the terminal volume of the fifth series. Throughout the twenty five volumes the coverage of the literature and format has maintained a consistently high standard, and over the years this encyclopedic work, in due recognition of the author's achievement has come to be more affectionately referred to as 'Theilheimer'.

When one is considering the synthesis of a molecule of any magnitude, or the operation of a single synthetic step, the question that is put, either consciously or subconsciously, is what bonds do I want to make, and how do I want to make them? Theilheimer is classified according to this simple idea. Each section is written under the heading 'Formation of the X-Y Bond', where X and Y cover the full range of atoms of interest to both organic and organometallic chemists. [H, O, N, Halogens, S, C and the remaining elements (Rem)]. When the various combinations of these elements are assembled the result is some twenty five different bond types. The sub-classification deals with the second question. How do I want to make these bonds? The bond formation types are symbolised as follows, addition (), elimination (), rearrangement () and exchange (). Furthermore the reactions such as electron pair formation on heteroatoms, and methods of synthesising nitrogen radicals and the heteropolar bonds are included. Volume 25 is completely up-to-date, containing references to some seven hundred papers that have appeared between 1968-1970. Each volume of 'Theilheimer' begins with a section entitled 'Trends in synthetic organic chemistry'. This section in volume 25 is as in previous volumes an extremely useful survey of the main synthetic achievements during the past year. Volume 25 includes references to the stereoselective juvenile hormone syntheses and prostaglandin syntheses that serve to remind us that natural products still provide a powerful incentive for the development of new synthetic methods.

The admirable achievement of 'Theilheimer' in collating the past twenty five years of synthetic organic chemistry can never be underestimated. Merely browsing through these volumes can be a source of many ideas and the acquisition of considerable knowledge. No active organic research school can afford to be without this vast store of information. Although prohibitively priced for the individual this should never deter libraries from apportioning part of their budget to the latest volume of 'Theilheimer'.

Book reviews

Understanding chemistry
1971
by G. C. Pimental and R. Spratley
Pp xiv | 974
San Francisco: Holden-Day $7·50

This is an excellent book into which an enormous amount of work and study has been put by the authors. It is a comprehensive text in general and physical chemistry which endeavours to return to fundamental chemical principles and bring order and logic to what in many similarly aimed text books is an unrelated collection of factual information. The book is ideally aimed for an introductory college course in the States which keeps the mathematical content to a minimum and adequately meets the need of 'A' Level chemistry courses in this country. In many of the topics for example the principles of chemical bonding orbital nomenclature and quantum theory the standard is carried beyond some 'A' Level syllabuses but the information is produced in such a palatable way that students using the book will be stimulated to see each topic through. The presentation is excellent and the index comprehensive. With 974 pages this is no small feat.

The book is to be thoroughly recommended to all students attempting 'A' Level or higher chemistry courses and it is a worthwhile addition to any chemical library.

J. D. THOMAS

Industrial crystallisation from solutions
1971
by J. Nyvlt Pp v+189
London: Butterworths £5·50

This short book is a translation of the Czech original of 1967, and it is described as having been extensively revised to increase its suitability for the English speaking market. The author has tried, with some success, to avoid excessive overlap with existing English texts, and he writes from the standpoint of the chemical engineer.

The first chapter, accounting for three fifths of the pages, covers the theoretical foundations of crystallisation as a preparation for the second chapter, which is a very welcome discussion of design calculations for crystallisers. This discussion is firmly based on the kinetics of crystallisation as well as on the usual mass bulances, and the use of numerous numerical examples confirms the general impression that this is a book by a practical man. Information about the solubility and crystallisation of many substances is presented in the appendices to the main text.

About thirteen hundred references are cited in groups, but the thoroughness of the literature survey varies markedly from one section to another. On p 77, for example, one sentence directs the reader to 50 references, and on p 156 no fewer than 66 references are introduced in this way. Since none of these references concern publications in the last ten years, a much shorter list of carefully selected recent references would have been much more useful.

The author has published a number of papers on crystallisation, not always easily accessible to western readers, and it is appropriate that his collected views should now be available in book form. Chemical engineers interested in crystallisation will certainly benefit from reading this successful translation, and it should encourage the replacement of the somewhat mysterious art of crystallisation by science-based engineering procedures.

D. A. BLACKADDER

Structure of organic solids. Main lectures presented at the Second Microsymposium 'Structure of organic solids', Prague, 1968
1969
Pp v + 86
London: Butterworths £2·90

The contents of this book have already appeared in *Pure and applied chemistry*, 1969, **18**, 4, pp 465 to 550. The Microsymposium referred to in the subtitle was held under the auspices of the Macromolecular Division of the International Union of Pure and Applied Chemistry, in conjunction with the Czechoslovak Academy of Sciences and the Czechoslovak Chemical Society. The contents consist of four excellent review papers in different fields. W. Hoppe and 11 colleagues described their experience over the last few years in the use of an automated four-circle diffractometer for the making of accurate *X*-ray diffraction measurements on an (if need be) unorientated single crystal. They illustrate this with a description of the structures and methods of structure determination of a number of complex organic compounds, including some of biochemical interest.

W. Ruland, in speaking of the structure of amorphous solids, gave a concise theoretical introduction to the idea of short-range order of particles of strongly anisotropic shapes such as a bundle of linear molecules or a stack of planar molecules, following this by experimental results on nylons and on carbonised organic materials.

G. E. Bacon, claiming that neutron diffraction studies will soon achieve first purity with and then pre-eminence over *X*-ray techniques for the determination of atomic positions and dynamics in organic crystals, gave an account of progress over the previous five years, illustrating this with examples of current studies of high accuracy of particular interest in chemistry.

Finally C. A. Taylor described optical methods (diffraction analogues) as an aid in structure determination, especially since the advent of the gas-phase laser has made it possible to rethink the design of optical diffractometers. By far the most useful applications of optical methods, he claims, are in studies of imperfectly crystallised material. His examples, copiously illustrated by well-reproduced diagrams, refer mainly to fibrous structures, which could have been from living or non-living material, but which are in fact 'shapes' producing various diffraction effects which could trigger off useful ideas relating to real structures.

The late KATHLEEN LONSDALE

Microwave molecular spectra. Volume 9, Part II of 'Technique of Organic Chemistry'
1970
W. Gordy and R. L. Cook Pp ix + 747
New York: Interscience Publishers
£15·50

Over the past twenty-five years microwave spectroscopy has contributed a wealth of information about molecular properties. Although it is probably most generally associated with precise structural determinations, it has also provided a wide range of other information about molecules. Included in this are molecular dipole and quadrupole moments, magnetic moments, nuclear quadrupole coupling constants, barriers to molecular inversion and internal rotation, conformations, molecular force fields and energy transfer in collision. The stage has now been reached where increasingly larger and more flexible molecules are being studied. Quite recently commercial instrumentation has been introduced which has enormously improved ease of operation. It is therefore opportune that a comprehensive and up-to-date book on the subject should be published at the present time. It appears as a member in the well-known 'Technique of organic chemistry' series, but its coverage and level of treatment will make it of interest to chemical physicists and physical chemists, and to spectroscopists in particular.

The book begins with a treatment of molecular rotation, in terms of angular momentum. The development of matrix elements of angular momentum operators lays the foundation for much of the book. Successive chapters give a systematic and comprehensive treatment of diatomic, linear polyatomic, symmetric rotor and asymmetric rotor molecules. Another chapter deals with molecules in which internal rotation occurs. The treatment of both high and low barrier approximations is set out and reference is made to numerous particular molecules.

Other chapters deal with line shapes and nuclear hyperfine structures. Stark and Zeemann effects for the various classes of molecules are treated in turn and again particular examples are considered. Centrifugal distortion in asymmetric rotors is clearly developed and its use in molecular force field determination is shown with examples. Particular attention is paid to the evaluation of molecular structures from rotational constants. A final chapter deals with nuclear quadrupole couplings, molecular dipole moments and the chemical bond. The whole treatment of the subject is well integrated, the quantum mechanical techniques used in the book following on smoothly from the development of matrix elements in the early part of the book. The reader is further helped by useful appendices. It is typical of the care that has been taken in the development that an appendix is devoted to the frequently used van Vleck transformation. In addition, the appendices include a wealth of information including molecular structures derived up to 1969. It is a book which is likely to be of wide interest for reference purposes on molecular structure, and will be indispensable for research workers with interests in rotational spectroscopy.

D. J. MILLEN

Aspects of adhesion 6
1971
by D. J. Alner Pp xii + 149
London: University of London Press
£3·45

This volume is one of a series dealing, as the title implies, with all aspects of adhesion. It is in fact the paper given at a conference held in the City University two years ago, but the fact that it is published somewhat later does not in any way detract from the topicality of the various articles in the book.

Since it is one of a series it deals particularly with only one aspect of adhesion, namely, the adhesion of thin films to various types of surface. Under these conditions, the interaction of the polymer with the substrate is of particular importance and there are, therefore, two papers dealing with the more fundamental aspects of this part of the subject and in particular one dealing with the effect of hydrogen bonding in adhesion. The remaining papers deal with quite specific aspects of the subject and include practical details of the instruments that have been designed to measure the adhesion of films under these particular circumstances. In addition, there are one or two articles dealing with adhesion of metal films to glass where the same general problems arise and in which the devising of suitable machines for measuring adhesion is equally important. As is usual in this subject, a great deal of the knowledge must be empirical in character though quite specific facts often emerge which are not explicable under the basis of existent theories. For those who are engaged in work of this kind the several chapters will be of great use in ensuring that films made under these conditions will be properly attached to the substrate.'

H. W. MELVILLE

Communications to the editor

Contributions should have novelty and should be brief. In order to expedite publication **proofs will not be circulated** *to authors unless requested when the manuscript is submitted or there are substantial queries by referees. Proofs not circulated will be checked against the manuscript and* **errata will not be published** *if the manuscript (including figures and formulae) is at fault.*

Structure of the pigments (Hansa colours) formed from acetoacetarylides with diazonium salts

*by P. Brady and J. H. P. Tyman**

Brunel University, Department of Chemistry, London W.3

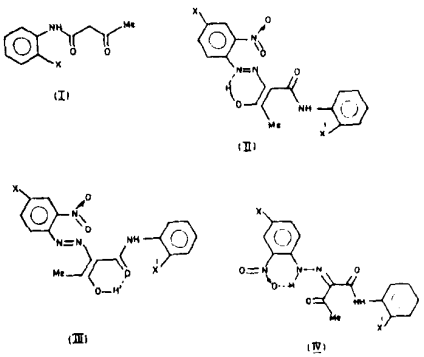
Recent preliminary work[1,2] on the crystal structure of certain analogues of HANSA 10G (C.I. pigment yellow 3) prompts the publication of this Communication on the related subject of the chemical structure of this class of substance.

In initial experiments, diazotised 4-amino-3-nitrotoluene coupled with the acetoacetanilides (I,X = F,Cl,Br,I) to produce yellow pigments having little difference in shade. The iodo and fluoro derivatives are new compounds.

Nitration of 4-fluoroacetanilide in concentrated sulphuric acid with methyl nitrate[4] rather than ethyl nitrate, followed by hydrolysis, diazotisation and coupling[3] with 4-fluoroacetoacetanilide and with compound (I,X = F) gave two new difluoro compounds which were orange in colour. The latter, the difluoro analogue of HANSA 10G, was obtained as orange needles from xylene or nitrobenzene, mp 248° C (Found: C, 53.2; H, 3.5; N, 15.4. $C_{16}H_{12}N_4O_4F_2$ requires: C, 53.1; H, 3.3; N, 15.4 per cent). The infrared spectrum (KBr disc) showed only a single carbonyl band at 1660-1670 (CONH), 1510cm^{-1} (NO_2), a broad band consisting of two maxima at 3050-3100cm^{-1} (NH and H-bonded OH) together with bands due to the N=N, C=C and phenyl groups. The n.m.r. spectrum proved to be difficult to determine owing to the insolubility of the pigment in the usual solvents ($CDCl_3$, $(CD_3)_2SO$, $DCON(CD_3)_2$, and C_6D_6). In hot nitrobenzene $C_6D_5NO_2$, absorption bands were present at τ 5·48 (*bs*, 1H, enolic OH), −1·55 (*bs*, 1H, CONH), 1·5-1·7 (*m*, 1H, ArH), 2·07-2·21 (*dd*, 2H, ArH), 2·8-3·0 (*m*, 4H, ArH) and 7·78 (*s*, 3H, Me). The enolic OH and CONH groups underwent exchange with D_2O. The dichloro (HANSA 10G) and the dibromo analogues[2] showed similar spectra.

Potentially there are a considerable number of hydrogen-bonded structures but only two (II and III, X = X' = F) need seriously be considered, incorporating a hydroxyl group associated with either an azo or a carbonyl group (of the CONH). The latter structure is reminiscent of the internal bonding occurring in acetylacetone. Since both benzeneazo-β-naphthol and acetylacetone (in nitrobenzene or $CHCl_3$) exhibit low field proton absorption (OH, τ-5) the n.m.r. spectrum does not afford a complete distinction between (II) and (III).

The existence of a tautomeric structure (IV) or the alternative in which the NH is associated with the carbonyl group can be excluded since in both cases neither the n.m.r. nor infrared absorption spectra would be in agreement. In *o*-aminoacetophenone τNH occurs at 6·12 (CCl_4). Likewise the n.m.r. spectrum of salicylaldehyde-2,4-dinitrophenylhydrazone, a double hydrogen-bonded structure showed τ-0·12 (*bs*, 1H, NH.... O, a feature characteristic of 2,4-dinitrophenylhydrazones generally) and τ −0·33 (*bs*, 1H, H-bonded phenolic OH). The infrared spectrum clearly exhibited a strong band at 1630cm^{-1} (C=N) absent in the HANSA pigments.



(I) (II) (III) (IV)

The bonded azo group in compound (II) would not be expected to undergo *cis-trans* isomerisation as would be the case with a free azo group. Chloroform solutions of the HANSA pigments irradiated with ultraviolet light for a prolonged period were like benzeneazo-β-naphthol completely stable whereas azobenzene rapidly isomerised. The difluoro compound before and after such treatment possessed, $\lambda_{max.}$ ($CHCl_3$), 260 (log ε4·40), 345 (log ε4·35) and 412nm (log ε4·31). The very fast properties of these pigments must indeed be associated with the very stable hydrogen-bonded structure (II) applicable to the solid states and to solutions.

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The presence of a fluorine atom *ortho* to the -NHCO- group might have been expected to lead to hydrogen bonding. No evidence of any significant shift in the n.m.r. absorption of the amide proton was found in the difluoro compared with the dichloro and dibromo compounds. 2-Fluoroacetoacetanilide, 2-fluoroacetanilide, and 2-fluoroaniline were similar confirming that the -NH- hydrogen-bonds comparatively less than the -OH group.

Although it was recognised[5] that only the enolic acetoacetanilide couples, numerous authors[6] have represented the structure of the products with the hydrazone grouping -NH-N=C<. In view of the present findings, the extensive formulation[7] of pigments of this type with such a structure is erroneous. Owing to simultaneous hydrogen bonding the enolic coupling product is stabilised as soon as it is formed.

The n.m.r. spectra of all the acetoacetanilides contain a small band at $\tau -3{\cdot}4$ (enolic OH) present to less than 10 per cent; tautomerism of the keto form to the enolic form readily occurs aided by the precipitation of the pigment as the coupling proceeds.

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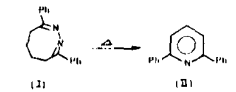
Pyrolytic reactions of cyclic azines

by G. Hasenheuttl, C. Opalka and J. G. Krause

Department of Chemistry, Niagara University, New York 14109, *USA*

Pyrolyses of several acyclic azines have been reported.[1] Now the first example of the thermolysis of a cyclic azine is described. The azine, 1,2-diaza-3,7-diphenylcyclohepta-2,7-diene (I),[2] was pyrolysed *neat and under nitrogen* at 250-300°C until there was no evidence of unreacted azine as shown by t.l.c. A considerable amount of ammonia was evolved during the pyrolysis, and a small amount (2 per cent) of liquid *identified as benzonitrile distilled* from the pyrolysate. Distillation of the residue at 0·1mm from an oil bath (220°C) afforded an oil (II) (70 per cent) which soon solidified and was recrystallised from ethanol, mp 81-82°C. Sodium *fusion confirmed the presence of* nitrogen, and the n.m.r. spectrum was devoid of any saturated C-H absorption. This suggested that the product could be 2,6-diphenylpyridine, and this was proved by comparison with an authentic *sample.*[3] Similarly, 1,2-diaza-3-5-7-triphenylcyclohepta-2,7-diene (III)[4] gave 2,4,6-triphenylpyridine (IV) (55 per cent), mp 137-139°C (picrate, mp 191-193°C) (Lit.[5] mp 137-138°C; picrate, mp 189-192°C).



(I) (II)

(III) (IV)

This remarkable reaction can be seen to involve the loss of only one nitrogen atom per molecule of azine as well as the loss of one hydrogen atom from each of the three sp^3 *carbons to give ammonia.* This contrasts with the pyrolysis of benzaldehyde azine where C=N cleavage with loss of nitrogen occurs to form stilbene.[6]

The conversion of compounds (I) and (III) into the *pyridines* (II) and (IV) under these conditions is probably a free radical process since the reaction displays an induction period (during large-scale pyrolyses on 50g of azine the reaction tended to go out of control at the beginning unless heating was stopped from time to time), and the yields are drastically reduced if oxygen instead of nitrogen is bubbled through the azine during pyrolysis.

Photolysis of the azines (I) and (III) with 3500Å light in methanol for extended periods of time resulted in no product formation and complete recovery of starting material.

The Niagara University Research Council is thanked for *partial support* of this work.

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