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# The Citrus Industry in Europe

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As part of a tour of several citrus areas during 1970, the author visited the three principal citrus-growing countries in Europe: Spain, Italy, and Greece. This article records the different approaches adopted by the citrus industries in these areas in tackling the important problems they face, both individually and collectively.

An earlier article (Chandler 1972) gave impressions of the citrus industry in Israel which has, within three decades, emerged as one of the principal citrus areas of the world, in terms of production, research, and technology. The growth of this industry has had an indisputable effect on the citrus industries in European countries, which find their traditional markets for fresh and processed citrus challenged by a progressive and even aggressive competitor, at a time when the world's citrus production is approaching a crisis of over-supply (Anon. 1970; Anon. 1971a; Turpin 1971). The general problems of the citrus industries in these European countries have been discussed in more detail by Burke (1967), and recent assessments of their position, and particularly their contributions to world trade in citrus, have been made by the U.S. Department of Agriculture (Anon. 1971b) and by Romero and Carles (1971).

## Greece

### *Citrus Industry*

One of the countries most affected by the expansion of Israel as a citrus exporter is Greece, and from conversations and personal observations its citrus industry is clearly not in a healthy position. It is based on small family orchards, widely scattered around the coast of Peloponnesus and the mainland to its north-west. These orchards have become increasingly fragmented with each generation, and frequently carry deciduous fruits, vine fruits, and vegetable crops interplanted with the citrus, mainly Navel oranges and lemons. While yields have improved in recent years, there is little current expansion in the acreage under citrus apart from an increase in mandarin cultivation. Thus Greece is one of the few countries where plantings of the principal citrus varieties are only just keeping pace with

the removal of old trees and the conversion of orchards to other uses.

Of all the countries visited, Greece comes closest to Australia in the volume of fruit produced, but there the comparison ends. Navels, which grow well under Greek conditions, with good yields of large high-coloured fruit, are the dominant crop, and there is so much grower resistance to planting any other orange variety that the Government has in hand legislation forbidding new Navel plantings. The need to cultivate other varieties, and so spread the season, arises from the unfavourable tariff against Greek exports to the EEC, and the inroads made by Shamouti and Valencia oranges from Israel into the traditional market for Greek Navels in Eastern Europe and Russia. Bilateral trade agreements still operate with Eastern Europe but such exports, though still substantial, no longer account for 20% of the Greek citrus crop and 70% of the total exports, as they did 10 years ago, even though citrus consumption in this region has quadrupled in the meantime. The industry is thus finding it increasingly difficult to dispose of unprocessable Navel oranges, maturing within a short season from late winter to early spring, particularly since, even now, the domestic consumption of oranges per head is about twice that of Australia, accounting for about two-thirds of the crop.

Faced with this situation, and accepting that Navels cannot be processed, the authorities, besides placing the ban on new Navel plantings and subsidizing orange exports by up to \$9 per ton, have embarked on a programme to improve fruit quality and lower production costs in an endeavour to win back Eastern European markets, and the facilities for citrus research in Greece will be devoted to this end for some time to come.

### *Citrus Research*

The centre for Greek citrus research is the Technological Institute for Plant Products, which is located in the Licrovissi country district about 8 km outside Athens. It occupies a large three-level building of about 1400 sq m with high ceilings and echoing corridors in the grand European style, and a basement which houses pilot-plant facilities; a small associated horticultural area provides material for trial processing operations. The Institute is a state-financed body with a staff of about 12 investigators and a few assistants under Dr C. D. Exarchos as Superintendent; their work is concerned entirely with the technology of processed fruits and vegetables, and with the testing of such products forwarded by other Government agencies. It is well equipped for this purpose though some of the processing equipment has required improvisation.

The Institute's citrus expert, Mr Jack Aspididis, has been mainly concerned with the utilization of oranges and with variations in the quality of the juice according to origin of the fruit. However, for the reasons discussed above, he has recently been directed to horticultural research aimed at improving fresh fruit quality, and was therefore recently transferred to the main Greek citrus area, the Argolis region in north-eastern Peloponnesus, where the Ministry of Agriculture operates a Citriculture Research Station at Poros.

### *Citrus Processing*

Because the period in Greece was brief and out of season, only one factory visit was made—to a citrus processing plant that was operated by a subsidiary of an English beverage manufacturer, and so cannot be regarded as typical of Greek citrus processing. This plant was comparatively small (about 950 sq m) but very modern, set up to handle lemon juice (single strength, concentrate, and comminuted) and lemon oil; waste materials were dried and sold to other processors for pectin recovery and cattle feed. It was a most scrupulously clean and efficiently run food processing plant with a particularly smooth flow-through of material from arrival of fruit to removal of dried waste. It demonstrated how comparatively easy it is to attain efficiency with a small modern unit, specially designed to process specified amounts of a limited range of products.

The establishment in overseas countries of such subsidiary factories is a recent development for British beverage suppliers, who annually sell about 112 million litres of citrus juices (60% in the form of frozen concentrate), more than 180 million litres of comminuted cordials, and more than 90 million litres of 'normal' cordials—the last two products for dilution 1:4 with water. These companies used to operate their own plants for the extraction of juice and the preparation of both types of cordials (squashes) from fresh fruit imported principally from Spain and South Africa. Nowadays, however, most of them import the citrus products they require in the form of frozen concentrate or comminuted fruit base from overseas processors who either are independent or function as subsidiaries of the parent company.

Thus, a complementary orange products plant similar to the Greek lemon products plant was operated by the same company near Valencia, Spain. In such situations, most of the quality control and formulation is done in Britain, while the local processor, who operates strictly according to instructions laid down by his English principals, looks after such factors as fruit supply, juice yield, and plant sanitation. Technical personnel from Britain visit the overseas plants regularly, and also whenever something goes wrong with the process or the product, as shown by sample batches routinely air freighted to London.

Some companies, on the other hand, contract with completely independent processors for their citrus supplies, changing supplier when desirable for reasons of cost or quality. All technical problems are left to the processor and his product is simply accepted or rejected according to whether it conforms to the terms of the agreement.

Under still a third system, the British company contracts for the supply of citrus products from established processors with whom it tries to develop long-term associations to ensure continuity of quality and supply; financial relationships between the parties may be complicated but, legally at least, the overseas processor is independent. There is no overall direction or control from London, but technical personnel are frequently called upon to advise and assist the processor. Quality control is carried out at both ends,



but most of the formulation is done by the processor, who supplies a range of products according to the cost and quality specifications of the British company.

## France

### *Citrus Research*

Unlike Britain, France has been actively associated with citrus research in the past through the Government-supported French Overseas Fruit Research Institute (IFAC) which carried out horticultural and technological studies on citrus in North Africa. This Institute had units scattered throughout the French overseas territories but with the dissolution of this empire its work has been gradually contracting until only a few of these scientific outposts remain active. Its activities have become increasingly consolidated around Paris, where M. P. Dupaigne directs an efficient technical service to the fruit products industry and where the Institute publishes the well-known international journal *Fruits* (in French with English summaries of original papers).

The reorganization of the Institute has resulted in an overall reduction in its studies relating to the citrus industry which now occupies a less important place in the French economy. Thus, one of the principal French citrus workers, M. R. Huet, who was originally stationed in Morocco, has returned to France to expand the Institute's chromatographic studies on fruit flavours, oils, and essences. His laboratories are located within the National Centre for Agronomic Research (INRA) at Versailles, and a closer integration of the Institute's work into that of this larger body is a future possibility.

Meantime, the Institute's interest in citrus is concentrated in the French island of Corsica with extensive trials in collaboration with INRA aimed at determining the varieties and clones most suitable for the island which, despite its frosts and winds, is France's only reasonable domestic source of citrus. The Corsican plantations, mainly oranges and mandarins, have expanded from 1200 ha in 1963 to 3500 ha in 1970, and are expected to approach 12,200 ha in 1975. With present plantings in Morocco alone standing at about 48,500 ha, the contraction in French interest in citrus research was inevitable, despite these recent developments in Corsica.

### *Citrus Industry*

Nevertheless, through such bodies as the Citrus Committee for the French Zone, the Institute maintains close contact with the North African citrus regions, particularly Morocco, which enjoys (with Spain and Israel) tariff preferences from the EEC. Here some work begun by the French is being continued. Until the French withdrawal, the Moroccan industry was quite well advanced, with a high level of horticultural practice and profitable fresh and processed fruit exports, but it declined seriously shortly afterwards, especially following the establishment of the EEC.

Recently the Moroccan government decreed the conversion of about 25% of the country's citrus acreage to the production of other crops, and with greater efficiency in the remaining orchards and consolidation of both fresh and processing operations the industry has improved considerably. Thus in 1969/70 fresh citrus exports reached a record 10 million kg (81% of production), compared with 1964/65 figures of 6.5 million kg (70% of production). Although the decline in the importance of the French market to the Moroccan industry is demonstrated by the fall from 46% to 35% in the proportion of citrus exports going to France over this five-year period, in 1970 Morocco still provided France with 9 million litres of orange juice and 2.7 million litres of grapefruit juice, respectively 64% and 28% of her requirements.

On the other hand, the Algerian industry is not important in the world citrus scene, except for mandarins which constitute about one-third of the crop. Some citricultural research of mainly local interest is still carried out in Morocco and Algeria, but it will be some time before the research effort in North Africa reaches the standard set by IFAC when it was active in the area.

## Italy

### *Citrus Industry*

Although Italy is one of the world's largest citrus producers, the fragmentation of all aspects of the industry—cultivation, marketing, and processing—has in the past deprived the country of the maximum economic benefits possible from its citrus crop, which amounted to 41 million kg in 1970/71. In the last two decades, the Government has spent a reported \$30 million dollars on



attempts to modernize citrus packing and processing operations as part of a general programme to improve the economy of the 'depressed' south (the industry is entirely located in the southernmost provinces) and in particular to demonstrate to Sicilians the advantages of being part of Italy. Economically, these efforts are justified by the favoured position Italy holds as preferred citrus supplier to the EEC but they are not welcomed unanimously by the independent packers and processors, who feel that the problems of the industry are largely concerned with establishing regular supplies of fruit at a reasonable cost.

Certainly Italian citriculture could benefit from Government intervention but again the highly individualistic attitude of Italian orchardists impedes progress. Thus, growers continue to plant orange varieties not in demand as export fruit, and refuse to uproot less productive groves despite government subsidies to do so. Although orange exports are heavily subsidized, Italy sells far less of the crop abroad than other Mediterranean countries. The record exports in 1969/70 amounted, for instance, to only about 13% of the orange crop, a little more than double the quantity that was withdrawn from sale by the Italian Government and reportedly destroyed.

Particularly in the southernmost mainland province of Calabria, where cultivation is often attempted right down to the seashore, the older Italian orchards are very hard-grown and thickly planted and carry a number of other crops. Growing conditions in Sicily are generally better than those in Calabria and some of the newer orchards (lemons near Palermo and lemons and oranges near Catania) follow modern cultivation practices, although terracing is undertaken in areas where Australian and American growers would contour their plantings.

The Italian citrus industry is unique in the importance of citrus oils and lemon products to its operations. In 1970, oil exports from Italy were valued at about \$6 million, 95% coming from Sicily. Sales were apportioned almost equally between the EEC, Britain, and the rest of the world, mainly the U.S.A. With lemons making up more than 30% of its citrus crop, Italy is the world's leading lemon producer and accounts for 60% of the world trade in this fruit. Oranges, on the other

hand, do not grow as well as lemons in most Italian regions, except the red-pigmented varieties which constitute 60% of the sweet orange crop. These 'blood' oranges, highly favoured as dessert fruit in central and northern Europe and commanding the premium 'winter orange' price in the U.S.A., would be a very attractive proposition except that fruit of uniform appearance is only available for a limited period and weakly pigmented fruit yields muddy-coloured juice on processing.

It appears to be virtually impossible for Italian orange juice processors to control the quality of their raw material, since most orchardists do not differentiate between fruit varieties cultivated, and supply to the processors a mixture of *Citrus sinensis* types, including blood oranges and heavily seeded common oranges (Biondo), with a sprinkling of mandarins. About the only form of control many processors exercise is the separation of the best blood oranges from the 'blond' types for the fresh fruit market.

#### *Citrus Processing*

Five citrus processing plants were visited in Italy, two in Calabria on the mainland and three in Sicily. Both the Calabrian plants were comparatively modern, 5–10 years old, each covering about 4600 sq m, with plans to expand to twice that area in the coming season, and producing single-strength and concentrated orange and lemon juices, beverages, oils, and peel products with a throughput of about 46,000 kg fruit per day. The Italicitrus Company on the west coast about 16 km north of Reggio operates an active research and development laboratory under Dr Alp Kunkar who has many publications on citrus quality control to his credit. Thirty-two km further up the coast was one of the several plants owned by W. Sanderson and Co., possibly the oldest and largest citrus processor in Italy; it had a remarkably high level of automation, particularly in the concentration units, but there were obvious problems in raw material control and consequently in plant sanitation despite (or because of) an associated fresh fruit packing house.

Even more recent in construction were the Sicilian plants of the IDOS Company in Palermo, and the Etna Company in Giarre at the foot of Mt. Etna. Both were established with considerable government support, especially the latter, in an effort to modernize the



citrus processing industry in their respective areas. The IDOS plant, the smaller of the two, is about the same size as the Calabrian plants and produces a similar range of products, principally from lemons, the major crop in the area. It is associated both financially and technologically with Assis Ltd., one of the largest citrus processors in Israel.

The Etna plant, occupying about 13,800 sq m, is the biggest single citrus operation in Italy, with an associated and very well-equipped fresh fruit packing house and extensive cold store facilities. The company recently launched concentrated frozen blood orange juice on the European market under the Ruby brand. Made from good-quality fruit, this juice had a pigment content high enough to ensure a bright deep red colour after processing and storage, in direct contrast to the usual product incorporating fresh fruit rejects in which the pigment content is too low to maintain a good colour. It is noteworthy that many experts think the Ruby product tastes more like raspberry juice than orange juice, and perhaps it provides a remarkable example of the effect of colour on taste response, since the fresh fruit, though distinctively flavoured, are recognized as oranges by the palate.

Some Italian processors resent the Government subsidy available to new companies like Etna, claiming that it encourages extravagance and provides opportunity for unscrupulous manipulation. A good example of the effective operation of a citrus processor without direct Government subsidy is the San Lorenzo Company, with a lemon products plant at San Lorenzo, a few kilometres outside Palermo, and a large, very modern orange products plant on the mainland about 40 km north-west of Reggio which is notable for a glass-enclosed control laboratory, centrally located in the processing area.

The much older San Lorenzo plant, despite an inconvenient layout resulting from numerous extensions, was particularly well run, manufacturing about 20 different types of lemon beverages. In an unusual method of fruit-handling, the lemons, delivered at a rate to match utilization, were dumped into large (12 m × 6 m × 4.5 m) water tanks from which they were drawn by bucket elevators. This procedure was claimed to eliminate damage to fruit on unloading, and the tanks served for both washing and holding the fruit

for the day's operations. In a unique process for comminuted products, FMC extractors were set up as if for oil recovery but with fruit juice as the rinse, and the wash liquors, containing oil and small chips of peel broken from the fruit during squashing, were then milled to provide the comminuted base. For their fine-quality oils, the company used an uncommon process in which cold-pressed oils were treated with pectinase before 'wintering', to precipitate resins and waxes and so give a product of high stability on storage and utilization.

Since oil recovery is usually more important in Italy than juice extraction, the local Fratelli Indelicato company manufactures citrus processing equipment specially designed for Italian conditions, which, nevertheless, finds world-wide use (Chandler 1971). Their two-year-old, 8000 sq m factory at Giarre is a very well-laid-out plant, with design and engineering laboratories, foundries, and several assembly lines which incorporate many modern principles in machinery manufacture while still making use of the individual craftsman.

### *Citrus Research*

Further evidence of the importance of essential oils to the Italian industry is provided by the fact that the major research institute in Italy concerned with citrus products is the Experiment Station for Essential Oils, at Reggio in Catania. The laboratories, located in the main street of Reggio overlooking the Straits of Messina, occupy a two-storey building of about 450 sq m which, though recently constructed, is in the conventional continental style with high ceilings, courtyard, and broad winding staircase. This unit of the Chemistry Division of the National Research Council of Italy was set up for quality control and research on Italian essential oils produced for export, including floral and folial oils. Oil from a non-processing citrus, the bergamot, receives the greatest attention.

Under the directorship of Professor Angelo di Giacomo, the staff of the Station includes Dr Maria Calvarano and her brother Dr Ignatio Calvarano, all three recognized authorities in the field of citrus oils and their properties. The Station does much routine testing of oils and has amassed a tremendous number of data on their properties and con-



stituents. Most of their research effort is directed to the development of methods, mainly instrumental, for assessing the purity of essential oils, work for which they are very well equipped, with gas chromatographs and ancillary apparatus, and pilot plant for oil recovery and purification. In addition, the Station publishes the quarterly journal *Essenze Derivati Agrumari* (in Italian with annual English summaries of original work), which prints about 80% of the citrus research papers from Italy, about 80% coming from the Station itself. The scope of this journal extends beyond the indications of the title and covers work on citriculture and citrus technology as well as general reports and surveys of the industry.

For an industry so important to the economy of large areas of the country (Calabria is almost entirely dependent on its citrus and oil industries), the paucity of citrus research in Italy is surprising. At Palermo in north-west Sicily there is a small industry-supported Centre for Experimentation on

Processed Foods and Citrus Products; a Department for Citrus Cultivation functions within the Faculty of Agriculture at the University of Catania on the east coast of Sicily; and at nearby Acireale the Ministry of Agriculture operates an Experiment Station for Fruit and Citrus Cultivation. The latter is reported to be the only orchard with certificated citrus plantings in Italy, and in the assessment of data on Italian citrus it must be recognized that they are most probably derived from heterogeneous material, particularly those relating to commercial orange products.

Apart from activities previously described, Italian citrus research reported in the literature is limited to spasmodic isolations and identifications of constituents carried out in the universities. Greater governmental promotion of citrus research and extension work would help to ensure that the country benefited fully from its current investments in the industry and from its advantages as principal citrus grower in the EEC.



Terracing of new orange plantations in the stony hill district south of Valencia, Spain, with older orchards in the lower background.



## Spain

### *Citrus Industry*

The Spanish citrus industry, one of the oldest in the world, is also one of the largest: Spain is the second largest citrus producer and the largest exporter of fresh fruit in the world. Its prosperity is indicated by the sizable, well-cultured Spanish orchards, which are devoted entirely to citrus, unlike those of Italy and Greece, and by the considerable recent increase in the area under citrus. Many of these new orchards were established at considerable cost since the land required special preparation, which included first removing the existing 'topsoil' of rocks and stones and then replacing it with soil from other areas such as river deltas. Terracing, with a built-in irrigation system fed by gravity from the adjacent hills, is prevalent, even in areas where Australian and American growers would use gently contoured slopes, and often with only two rows of trees per terrace.

Most new citrus groves in the Valencia area are located in the hills, and although such new plantations are undertaken under Government advice the frost hazards are very great. Indeed, the citrus industry of Spain is probably the most frost-threatened in the world; for example, in the 1970/71 season, below-freezing temperatures in December and January reduced the estimated crop by about 15% or 5.7 million kg. The greatest problem faced by Spanish growers, however, is the virus disease *la tristeza* which affects roughly one-third of the country's total citrus area and has already destroyed about 2 million trees since 1956, with another 20 million threatened.

Considerations of frost and virus susceptibility, together with high alkalinity and calcium level in the soil, greatly restrict the citrus varieties grown and the rootstocks used in Spain. Sweet oranges account for 90% of the citrus crop, about 40% each of blood oranges and seedless types, principally Washington Navel, and 5% each of Valencias and heavily seeded types; mandarins, lemons, and Seville oranges make up the remaining 10% of the crop.

The sour orange is still the dominant rootstock in Spain. Trial plantings on Troyer citrange and Cleopatra mandarin stocks are currently under study, but any change in rootstock is seriously circumscribed by risk of virus infection as well as by soil quality;

for instance, trifoliate orange and sweet orange have proved unsuitable rootstocks for Spain, though widely used in other countries.

Because Spanish orchards show great variability in their resistance to virus diseases and frost damage, it is common practice to cultivate two varieties or rootstocks side by side in the one block to determine *in situ* which is the more profitable. To reduce the dependence on Washington Navels and blood oranges, recent Spanish plantings have favoured the Navelate and Navelina late-maturing Navel types, and Satsuma and Clementine mandarins, with some Valencias to extend the range of citrus available, so that the Spanish industry can provide excellent-quality fresh fruit for 9 months of the year.

### *Citrus Processing*

Exports of fresh fruit, amounting in 1969/70 to two-thirds of the crop, dominate the industry, more than 80% going into the EEC. Unfortunately, the expansion in exports and the increase in home consumption have not kept pace with the growth in citrus production, and in 1969/70 there was an estimated surplus of 3.2 million kg, about one-eighth of which was purchased by the Government and resold to the processing industry at an unknown loss. Juice production may, in the future, be expected to absorb more of the surplus crop, with an expansion of trade in chilled juice, sometimes partly preservative and sometimes pasteurized, exported to France, Germany, and even further north in refrigerated 90,000-litre tankers. This product is either sold as fresh juice or processed for less immediate marketing, and finds particular use in countries such as France which do not permit the sale of reconstituted concentrate as the equivalent of single-strength juice. In the last season, 800 million kg of oranges were consumed as juice in Western Europe, and an increase of 50% in this amount is expected within the next 5 years.

Two government agencies, the Spanish Fruit Syndicate and Spanish Export Service, provide a double check on the quality of citrus products for export, with the power to enforce the specifications they set, and it is significant that a high proportion of world research into the detection of adulteration of citrus products emanates from Spain. Comminuted juices are regarded with suspicion by the authorities as inferior products whose



quality is difficult to regulate in the way that they are able to control normal juices and concentrates.

Quality control for Spanish processors would indeed be difficult because of the number of different types of oranges utilized. For instance, at the time of this visit, one plant was operating on 60% Valencias, 20% bloods, and 20% seedy types, with a premium price paid for Valencias: it was claimed that they would process juice from December to May with such mixtures without recourse to Navels. Bitterness in Navel orange juice would be a serious problem in Spain since, for reasons discussed above, sour orange was still the dominant rootstock, but there was no indication that processors were encouraging the planting of later-maturing Navel types which are less susceptible to bitterness development in the juice. Despite these problems in raw material supply, standards of efficiency and hygiene in Spanish plants compared favourably with those in Australia.

One unique feature of the Spanish citrus industry is the freeze concentrator operated by Vital Citrus Processors at Gandia, south of Valencia. It follows the Linde-Krause principle, with a rotating refrigerated drum partly immersed in orange juice. The frozen juice is scraped off the drum and separated into ice and concentrate in a screw press. Separation is not complete and the intermediate fraction is diverted to another line for conventional evaporation. After two stages of freeze concentration, a product containing 42% sugar is obtained which costs about 40% more than evaporated concentrate but is regarded as economically competitive by virtue of its high quality. The freeze concentrate finds its major use in Germany where it is usually mixed with cheaper concentrates to give a better-flavoured product on reconstitution.

#### *Citrus Research*

The citrus industry in Spain, along with other branches of the food industry, benefits greatly from the activities of the Institute of Agricultural Chemistry and Food Technology which is the leading research centre for food science in Spain. The Institute, one of 22 groups within the Technical Research Section of the Council for Scientific Research, is located across the road from the University of Valencia with which it enjoys close asso-

ciation. They collaborate in operating a School for Agricultural Engineering (including Food Technology) for post-graduate students, and the Institute has the use of the University's facilities such as computer and NMR services.

Located in a modern five-storey building of about 1400 sq m, the Institute, under the direction of Dr E. Primo, is concerned with the production of food crops and the technology of their utilization. It operates its own orchards and farms where crops are grown under supervision, maintains close liaison with industry, and undertakes a limited but stimulating teaching load, all desirable features in a food research institute. In addition, it publishes the quarterly journal *Revista de Agroquímica y Tecnología de Alimentos* (in Spanish with English summaries of original work) which contains almost all the food research papers from Spain, with special sections (including one on citrus) devoted to technical reports, surveys, and information on various aspects of the food industry.

However, since the food processing industry in Spain is generally not so far advanced as in most other Western countries, there is much emphasis in the work of the Institute on problems of immediate practical application. Thus, a major project aims to improve the quality of Spanish canned foods by comparisons of local and imported brands, identification of the cause of quality deficiencies, and rectification of these deficiencies by studies in the Institute's well-equipped pilot plant.

The breadth of the Institute's research may be gauged by reference to three long-range studies related to citrus: fertilizer trials throughout the citrus regions of Spain with the cooperation of the Ministry of Agriculture under the supervision of Dr Antonio Piero; a study of the variations in protein, flavonoid, and limonoid constituents of Spanish citrus with variety and region under Dr Royo Iranzo, with particular reference to his well-known work on detection of citrus adulteration; and an examination of virus-infected trees for constituents that may be used for early chemical diagnosis of infection.

There are two other major centres for citrus research in Spain. At Murcia, 160 km south of Valencia, another unit of the Council for Scientific Research but within the Agri-



cultural Sciences Section, the Institute for Soil Science and Agrobiolgy, cooperates with the Department of Agriculture of the University of Murcia in an extensive programme of citricultural research under the direction of Dr Octavio Carpena. At Burjasot, 16 km north-east of Valencia, another Government agency, the National Institute for Agronomic Research sponsored by the Ministry of Agriculture, operates the Eastern Citrus Station which has sections devoted to most aspects of citriculture, including rootstocks, viruses, and nutrition. The Station also has a Crop Utilization and Preservation Section under Dr A. R. Feliu, which is concerned with citrus products, both fresh and processed. The processing area of about 230 sq m combines an FMC packing-house unit for such operations as washing, waxing, and wrapping, with pilot-plant facilities for juice extraction, canning, and concentration, while the associated laboratories are mainly devoted to routine analytical and quality testing for the Government-controlled Spanish Export Service mentioned above.

Under a national plan for coordination of citrus investigations promoted by the Committee for Scientific and Technological Research, which is the Spanish Government's supreme advisory council in this field, the three centres at Valencia, Murcia, and Burjasot have recently undertaken a cooperative, comprehensive, four-year research programme designed to increase the wealth of this traditional Spanish industry by studies on varieties, rootstocks, soils, fertilizers, and methods for virus and frost control.

### Conclusions

With common problems resulting from an expanding world production of citrus, particularly in South America (Pitcher and Tisch 1970; Anon. 1971*d*), against a background of mounting competition from synthetic 'citrus products' (Myers 1969), European citrus countries face individual difficulties, which they are making strong efforts to solve: Greece with its dependence on lemons and Navel oranges, Italy with its need for industrial coordination, research, and extension, and Spain with its threats from frost damage and virus infection. A recent survey (Romero and Carles 1972) indicated a surplus citrus production in the Mediterranean area of

700 million kg by 1976/77, which, though amounting to less than 10% of the total crop, could pose very severe marketing problems. Thus, although a 50% increase in the consumption of orange juice could be expected in Western Europe by that time, the European citrus areas could expect to intercept only a quarter of this increase because of strong competition from other areas.

Nevertheless, each country is increasing its investment in citrus growing and processing because of the importance of the industry in their total economy and because each is determined not to lose its position in the world citrus trade. The work on new citrus varieties recently reported to the Eighth International Congress for Mediterranean Citriculture (Anon. 1971*c*) provides striking evidence of this determination.

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# A Study of the Microstructure of Bread Doughs

By R. Moss

Bread Research Institute of Australia

Now that new methods of sectioning and staining samples have been developed, microscopy can prove a valuable tool in studying the structure of bread doughs at various stages of mixing.

Microscopy is a technique that is often neglected when a suitable method to study some food research problems is being selected. The technique is particularly suitable where a knowledge of the physical structure and composition of the material is important to the understanding and solution of the problem. In the 1920s and 1930s many food scientists used microscopy as an integral part of their research programme, but later it became relatively neglected. This was because many workers assumed that they had an adequate picture of the structure of the materials with which they were involved. Often this was based partly on earlier work, but also partly on deductions resulting from their own research findings. However, new and improved methods of sectioning have recently been developed which, together with new staining techniques, allow clearer, more accurate, and more detailed studies of the microstructure to be made. These developments are largely due to the pressure and demands of medical research where histological and histochemical studies have always played an important part. These advances are now being implemented by workers in other fields to give a clearer understanding of their own research problems and thus assist in their solution.

The sectioning and staining techniques to be described have been specially developed for the study of dough microstructure but they could easily be adapted to a wide range of other foodstuffs.

When a dough is mixed several changes in its external appearance can be discerned with the unaided eye. These changes provide some useful information to the baker and can indicate to the skilled operative whether the dough is under or over mixed or when it has

received sufficient mixing to produce an acceptable loaf. The extent of this information is, however, very limited; it is also imprecise, and much of it is lost. What is of greatest importance is that the operative cannot describe to an observer exactly what information he gained from looking at and feeling the dough, although it is by this that he judges the duration of the mixing. The light microscope, with its resolution limit of  $0.2\ \mu\text{m}$ , can reveal many of the changes taking place not only on the surface, but also *within* the dough. Moreover, with the photomicroscope, such changes can be recorded, thus enabling comparisons to be made that do not rely on subjective assessment.

In the traditional mixing process the doughs are mixed for 20 to 30 minutes in a slow-speed mixer, after which time the dough is too resistant to stretching and not sufficiently elastic to undergo subsequent mechanical processing. These structural defects are rectified during bulk fermentation. In high-speed mixers sufficient energy may be imparted to the dough in a much shorter time, resulting in a structure having the desired properties of elasticity and extensibility. Slow-speed mixers can produce a dough having these desirable characteristics immediately after mixing with a suitable combination of reducing agents (cysteine or sodium metabisulphite) and oxidizing agents (dehydroascorbic acid, bromate, or iodate).

After completion of mixing, a dough appears to the unaided eye to be a homogeneous mass, but when viewed through a microscope the magnified image enables individual components to be recognized. The flour proteins undergo many changes as mixing commences, and the formation of the gliadin and glutenin proteins into a con-



tinuous gluten network can be studied for a wide variety of mixing actions. The marked effect on the appearance of the gluten network caused by the addition of oxidizing and reducing agents used in activated dough development is also clearly discernible.

Starch granules, which are the other main flour component, can be observed with the aid of a differential staining technique (Flint and Moss 1970). The distribution of the starch granules within the dough and the manner of their association with the gluten greatly modifies the physical behaviour of the dough and yields much information about the flour and the mixer. The water absorption of the flour is considerably affected by the amount of damaged starch granules present, and it is possible to differentiate both milling- and weather-damaged starch granules from undamaged granules. The presence of non-flour ingredients, fat and yeast, is also plainly revealed. The distribution of the latter gives a good indication of mixing efficiency. The microscope also provides information about the changes in the microstructure that cause the transformation of the dough surface from rough and 'woolly' to smooth and 'clear'.

Compared with microscopy in medical and zoological fields relatively little has been published on the microstructure of doughs and baked goods (Bahlsens Keksfabrik 1956; Baker 1941; Burhans and Clapp 1942; Butterworth and Colbeck 1938; Carlin 1944; Francis and Groves 1962; Jooste and Mackey 1952; Pohl, Mackey, and Cornelia 1968; Sandstedt, Schaumburg, and Fleming 1954; Strandine *et al.* 1951), and of the papers available the majority are concerned with bread. They are scattered widely through the literature and frequently do not give sufficient information on the composition of doughs and their production. When production details are given they are often very different from those used in current commercial practice.

### Equipment

To show structural details under the light microscope, sections must allow light to be transmitted through them. Investigations on dough structure require the section thickness to be in the region of 10  $\mu\text{m}$  (0.001 cm). The apparatus used to cut these thin sections is known as a microtome.

The structure of a dough is too fragile to enable thin sections to be successfully cut

without an embedding medium to support it. Paraffin wax has been traditionally used to infiltrate histological specimens, but the preceding clearing stages completely remove the lipid component from the dough. Gelatin, water-soluble waxes, ester waxes, celloidin, and plastics have all been used as alternative embedding media, but are unsuitable for use with dough as either the temperature or the solvents used would alter the distribution of one or more of the dough constituents. The only embedding medium that appeared to be suitable was ice, preferably derived from the naturally occurring dough water.

Freezing microtomes are essentially similar to ordinary microtomes but are equipped to freeze the sample on the microtome stage. The freezing is usually achieved with liquid carbon dioxide. Provision for cooling the knife with an additional carbon dioxide jet was introduced by Schultz-Brauns (1931). More recently freezing has been by electrical means, using Peltier cells. The greatest disadvantage of all these methods is their dependence on ambient conditions; temperatures above 18°C and excessive humidity tend to prevent successful sectioning.

The alternative to the ordinary freezing microtome is for the specimen, microtome, and surrounding atmosphere to be at a low temperature. This may be achieved in a cryostat.

### Cryostat Microtome

The first cryostat was developed in Denmark by Linderstrøm-Lang and Morgensen (1938). In the original, cooling was by blocks of solid carbon dioxide, but these were soon replaced by refrigeration coils which enabled the temperature to be accurately controlled.

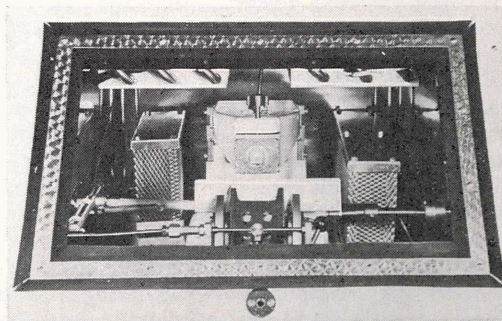


Fig. 1.—Interior of cryostat microtome, with front perspex window raised.



A Slee-type HR cryostat (Fig. 1) was chosen as the most suitable for the investigations at the Bread Research Institute. It is fitted with a rotary retracting microtome and the sample, when mounted on the microtome, retracts from the knife during the return half of the cutting cycle. This prevents frictional heat being generated by the sample rubbing against the back edge of the knife and therefore minimizes the possibility of smearing the shortening across the sample face. The cryostat operates at a selected temperature between  $-5$  and  $-30^{\circ}\text{C}$  with an accuracy of  $\pm 1^{\circ}\text{C}$ . The controls for operating the microtome are situated on the outside of the cabinet and access is via the heated perspex window or via the central 9-cm-diameter porthole.

The sections remain frozen after being cut, which is not the case when handling dough sections of similar thickness from a simple freezing microtome. If the sections were allowed to thaw prior to being placed on glass slides, the structure would completely collapse.

#### *Microscope*

The Zeiss WL Research microscope, fitted with a series of apochromatic objectives, permits structural details in the sections to be recorded photographically using either black-and-white or colour film. Transmitted bright field illumination and polarized light are used, the latter being particularly useful for studying the structure of starch granules and the distribution of damaged granules in a dough.

#### **Materials and Methods**

Samples of dough, approximately 20 g, are taken from the mixer at selected intervals as mixing proceeds. They are rapidly frozen by immersion in liquid nitrogen or by quenching with liquid carbon dioxide, to prevent further changes to the structure and to render the dough less liable to damage by handling. The rate of freezing is sufficiently rapid to avoid any observable disruption of the structure due to ice crystal growth. Samples of frozen dough (0.5-cm cubes) are cut from the centre of the frozen samples whilst in the frozen state. The cubes are fastened onto stainless steel holders by freezing a special supporting medium with liquid carbon dioxide, and the holders are clamped onto the microtome arm. The sections are orientated on chilled glass

slides and, when flattened and fully in contact with the slides, allowed to thaw. Slides to be examined for fat distribution are kept below  $4^{\circ}\text{C}$  at all times to prevent migration of the fat.

The sections are allowed to dry on the slides for at least 24 hours before being stained with the following specific reagents to reveal the various ingredients.

(1) *Wheat gluten and other proteins*—a 0.1% aqueous solution of ponceau 2R (C.I. No. 16150), acidified with 4 drops of 1N sulphuric acid per 50 ml of staining solution. Ponceau 2R is an anionic, azo dye that is bound to the positively charged sites on the protein molecules.

(2) *Damaged starch*—a 0.5% aqueous solution of chlorazol violet R (C.I. No. 22445), after pretreating the section with a 1% aqueous solution of the anionic dye-resist agent Taninol A.D.R. to prevent the protein being stained by the dye. Details of this technique have been published elsewhere (Flint and Moss 1970). Chlorazol violet R is an anionic disazo dye that penetrates and stains the damaged starch granules, but cannot penetrate the undamaged granules and therefore leaves them unstained.

(3) *Yeast cells*—a 0.01% aqueous solution of methylene blue (C.I. No. 52015), buffered to pH 4.6 (Fink and Kühles 1933). Methylene blue is a cationic thiazine dye that is bound to negatively charged substrate groups. At neutral pH many dough constituents are stained, thus making it difficult to identify the yeast cells, but at pH 4.5 only the yeast cells stain as they have many free carboxyl groups.

(4) *Lipids*—a 70% ethyl alcohol solution saturated with respect to Sudan IV (C.I. No. 26105) and oil red O (C.I. No. 26125) (Flint, Moss, and Wade 1970). These molecules are not dyes but lipid-soluble colourants, the lipid being stained by virtue of their greater solubility in lipids than in the hydro alcoholic solvent.

(5) *Bran*—a 0.05% aqueous solution of toluidine blue (C.I. No. 52040) buffered to pH 4.4 (O'Brien, Feder, and McCully 1964). Toluidine blue is a cationic thiazine dye closely related to methylene blue. It stains lignin a blue-green and metachromatically stains polyuronides of the cell wall purple.

Undamaged starch is shown by choosing a mounting medium of suitable refractive



index (G. T. Gurr's Xam, improved, white, neutral medium, R.I.1.49) slightly different from that of the starch, thus ensuring sharp and distinct outlines of the starch granules.

The series of photomicrographs has been selected from one mixing study to demonstrate the changes that take place in the microstructure of a dough made from a commercial New South Wales bread flour mixed in a Farinograph recording dough mixer. This consists of a rectangular containing vessel, the base of which is shaped to take two sigma mixer blades rotating in opposite directions. The force required to turn the blades at constant speed through a dough is measured by a dynamometer and recorded on a strip chart recorder. The development of a continuous protein network from the diffuse protein present in the endosperm is followed through the early stages of mixing to the stage at which breakdown due to over-mixing occurs. The distribution of the yeast, shortening, and damaged starch is shown at various stages during mixing and changes in distribution are discussed.

### Results and Discussion

As can be seen in Figure 2, the starch granules can be divided into two main groups by their size and shape: the large, elliptically or irregularly shaped *A* starch granules (*A*, long diameter approximately 30–40  $\mu\text{m}$ ) and the small, round *B* starch granules (*B*, diameter less than 10  $\mu\text{m}$ ).

At the left of Figure 2 a mass of tightly packed starch granules is enveloped in a diffuse, palely stained protein. This is an endosperm cell (*E*) and the thin, unstained cell walls (*W*) can be seen above and below the cell.

The dough has been mixed for only 1 min and as the mixing proceeds these cells are broken apart. The proteins become hydrated and the gluten develops into large masses (*M*) that bind starch granules in their midst and stain intensely. The formation of these masses depletes the remaining starch granules of the enveloping protein that surrounds them in the endosperm cells.

Little gluten development has taken place because of the short mixing time and in Figure 3 isolated streaks of compressed yeast cells can be seen throughout the dough. The methylene blue technique stains only the yeast cells, which are clearly visible as densely

stained, small dots (*Y*). The majority of the dough mass contains few or no yeast cells. As a gluten network is formed the yeast becomes evenly distributed throughout the dough either as single cells or in small groups of less than six cells each.

After 3 min mixing no diffuse, undeveloped protein remains, as shown in Figure 4. The discrete starch-protein masses have been stretched out by the mixer blades into sheets, which in section appear as long, thin fibrils (*F*). These fibrils contain only the small starch granules since the diameter of the larger granules is greater than that of the fibrils. Some of the larger granules are embedded in the surface of the fibrils.

The fibrils link together to form a continuous network. At this stage of mixing some large starch-protein masses (*M*) are still incorporated in the network. There are large spaces between adjacent fibrils, containing starch granules free from enveloping protein. The network has many discontinuities, but these are reduced as mixing proceeds, and the spaces between fibrils are also reduced in size.

Three minutes' mixing under these conditions is sufficient to distribute the lipid evenly throughout the dough, mainly in the form of globules. The lipids are intensely stained by the Sudan IV/oil red O mixture and in Figure 5 can be seen as small, black masses or globules (*G*). The diameter of the globules is less than 10  $\mu\text{m}$ , with the majority less than 2  $\mu\text{m}$ . Very few of the larger lipid masses and streaks observed earlier in the mixing process remain.

Subsequent mixing does not alter the lipid distribution or reduce further the size of the globules. The light grey areas (*L*) in which some small starch granules are embedded are gluten masses, palely stained due to lipoproteins.

The structure of the protein fibrils (*F*), however, is changed by further mixing, and Figure 6 shows their appearance after 7 min. They are stretched, resulting in a decreased diameter. There is more cross linking between adjacent protein fibrils and more of the starch granules are intimately associated with the protein. Some of the fibrillar protein has been converted into an enveloping mantle (*EM*) flowing around the starch granules. This results in a marked decrease in staining intensity.





Fig. 2.—Dough section, thickness 10  $\mu$ m. Mixing time 1 min. Stain ponceau 2R.

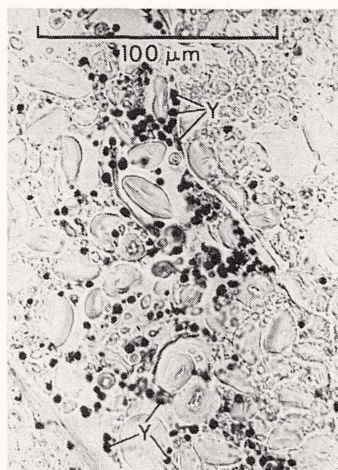


Fig. 3.—Dough section, thickness 10  $\mu$ m. Mixing time 1 min. Stain methylene blue.



Fig. 4.—Dough section, thickness 10  $\mu$ m. Mixing time 3 min. Stain ponceau 2R.

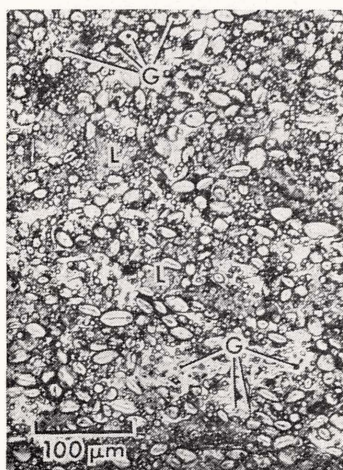


Fig. 5.—Dough section, thickness 10  $\mu$ m. Mixing time 3 min. Stain Sudan IV/oil red O.

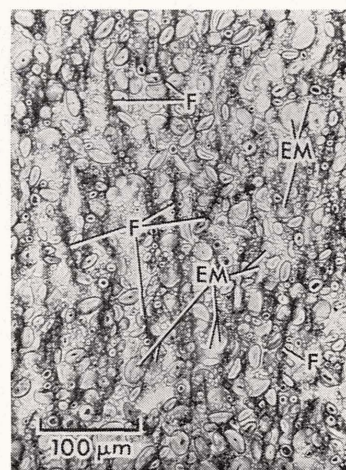


Fig. 6.—Dough section, thickness 10  $\mu$ m. Mixing time 7 min. Stain ponceau 2R.



Fig. 7.—Dough section, thickness 10  $\mu$ m. Mixing time 7 min. Stain chlorazol violet R.

Dough mixing does not increase either the degree of damage or the number of damaged starch granules. The distribution of damaged granules (*D*) after 7 min mixing is shown in Figure 7. Only those starch granules damaged either enzymatically or physically during milling are stained with chlorazol violet R.

Generally it is the larger *A* starch granules which are damaged since they are subjected to greater pressures when the endosperm

particles pass through the mill rolls. Starch granules that are stained are not severely damaged because, in addition to being unbroken, the staining is not intense and the granules have distinct outlines.

Mixing was stopped after 11 min, at which stage the dough was very difficult to handle, being extremely extensible and very sticky. This represents a gross degree of overmixing and no fibrillar protein network remains, as



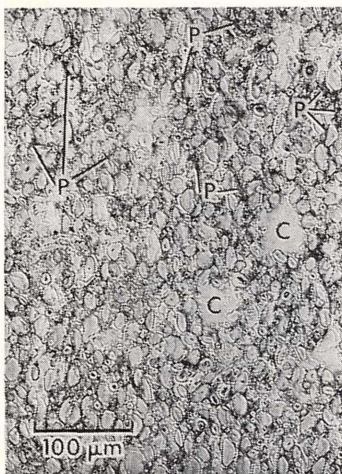


Fig. 8.—Dough section, thickness 10  $\mu$ m. Mixing time 11 min. Stain ponceau 2R.

can be seen from Figure 8. The gluten has been converted into a veil-like mantle that surrounds all the starch granules in the dough. This mantle is very palely stained and appears to be homogeneous, no structural detail being resolvable. Some more intensely stained protein (*P*) is present in isolated areas, being the last vestiges of the fibrillar network. The small round spaces (*C*) in the photomicrograph are air cells incorporated into the dough by the mixer.

The photomicrographs illustrate the changes undergone by one particular flour and dough formulation when mixed in a selected high-speed mixer. Addition of one of the oxidizing or reducing agents used in bread manufacture would alter both the manner and the rate at which these changes occur.

Oxidizing agents increase the strength of the gluten and make it more resistant to stretching into fibrils, later to be interconnected to form the continuous gluten network necessary for retaining gas during fermentation and for forming a desirable crumb structure in the finished loaf. Once the fibrillar network has been established, however, it is more stable than that formed from the same flour in the absence of oxidizing agents, and requires much longer mixing to convert it into a veiling mantle of protein to surround all the starch granules. Reducing agents have the opposite effect, and increase

the extensibility of the protein. This results in the more rapid formation of a fibrillar network but one which is very easily over-mixed into a veiling protein mantle that has poor gas retaining properties and cannot be successfully machined.

Currently at the Bread Research Institute this technique is being used to examine the effect of these additives not only during the mixing of doughs but also during their relaxation, moulding, and proofing. This is then related to the microstructure of the crumb from the finished loaf.

### Summary

The changes that take place in the microstructure of bread dough during mixing have been studied using a specially developed light microscopy technique. Dough samples are frozen by immersion in liquid nitrogen and sectioned on a cryostat microtome, and the distribution of wheat protein, damaged and undamaged starch granules, yeast cells, and lipids is demonstrated by the use of selected, specific stains.

The mixing destroys the protein matrix that, in the wheat, encloses all the starch granules, and develops the gluten into compact masses surrounded by starch granules free from any supporting protein. These masses are then stretched out to form a continuous network throughout the dough which appears fibrillar up to the optimum mixing time. Upon further mixing the fibrillar network is converted into a veiling mantle of protein that surrounds all the starch granules.

The distribution of yeast and added lipids is initially uneven, but quickly becomes even when the continuous fibrillar network has been formed. Mixing does not alter the amount of damaged starch granules, which remains the same as that present in the flour.

Certain oxidizing and reducing agents are used in some commercial bread manufacturing processes, and their effect on the microstructure is discussed.

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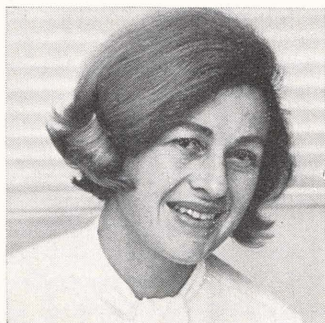


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## News from the Division

### New Appointments

Miss Josephine Bastian joined the Division as Assistant Editor, to work with Mr G. J. Walker in the editing of papers and reports; she will be taking a particular interest in the *Food Research Quarterly*. Miss Bastian graduated B.A. from the University of Syd-



ney in 1952 with first-class honours in English Literature and M.A. from London University in 1958. Her appointments include Teaching Fellow in the Department of English Literature, University of Sydney, and Acting Principal of the Women's College at the same University. Miss Bastian has worked as a freelance journalist for some years.

Mr J. E. Algie, Experimental Officer, and Mr R. Gamble, Technical Officer, transferred to FRL from the Division of Textile Physics on 3 July. They will join the Physics Section to study the properties of food by dielectric methods.

### Retirement

On 4 July, Dr W. J. Scott retired as Officer-in-Charge of the Meat Research Laboratory, Cannon Hill, after a career with CSIRO spanning 39 years. A two-day symposium was held in mid June in commemoration of Dr



Scott's contribution to meat science and technology, and this will be reviewed in the December issue of this *Quarterly*.

Dr Scott's successor as Officer-in-Charge of MRL is Dr D. J. Walker, formerly of the Division of Nutritional Biochemistry.

### Visiting Workers

Mr Derek D. Oudit of the Department of Agriculture, Trinidad and Tobago, West Indies, returned to a research position in Trinidad on 22 June. He was in Australia for two years on a Commonwealth Scholarship, attached to the Fruit Storage Section at FRL. During his stay he completed the requirements for an M.Sc. degree from the University of New South Wales, the subject of his thesis being 'Effects of composition of the atmosphere on chilling injury in selected fruit after harvest'.

Two UN/FAO Fellows spent several weeks each at the Division. Mr Aloisio Enoka from Western Samoa received training in food processing techniques and Mr Nadir Isik from Turkey studied analytical instrumentation and methods, with a view to introducing some of these at the Canning Research Institute in Bursa, Turkey.

### Awards

Mr G. Loftus Hills, retired Chief of the former Division of Dairy Research, has received the gold medal of the Society of Dairy Technology (U.K.).

Mr F. D. Shaw of MRL has been awarded the M.V.Sc. degree by the University of Queensland for his thesis 'A study of some physiological and biochemical indices of fattening in cattle being fed rations of wheat'.

### General

The Australian and New Zealand Institutes of Food Science and Technology held their first joint convention from 21 to 25 May at Surfers' Paradise, Queensland. Several papers by members of the Division were presented. Mr J. F. Kefford, who is President of the Australian Institute, spoke about the training of personnel for the food industry. Other papers from the Division dealt with temperature effects in fish processing, vacuum tumble blanching, vacuum concentration of

passionfruit juice, the flavour of passionfruit juice, production of a sterilized dairy-based carbohydrate-free infant food, nitrate in foods, the properties of rock lobster muscle, and heating with humid air.

Mr M. V. Tracey, Chief of the Division, has been appointed a member of a committee established by the CSIRO Advisory Council to review the present relationships between secondary industry and the Organization, including the transfer of up-to-date information (in both directions), the letting of contracts, granting of patents and licences, and, in general, to assess the effectiveness of CSIRO vis-à-vis secondary industry, as defined in the Science and Industry Research Act 1949-1968. The committee includes members from industry, the universities, and CSIRO.

### New Periodical

A new quarterly journal has appeared, published by the Food and Agriculture Organization of the United Nations. *World Animal Review* plans to deal with all aspects of animal production, animal health, and animal products (meat, milk and milk products, eggs, wool, etc.), with special reference to work being done in these fields in the developing countries.

Although it is written primarily for a technical audience, it should also be of considerable interest to anyone in government, teaching, or research who is concerned with primary industry.

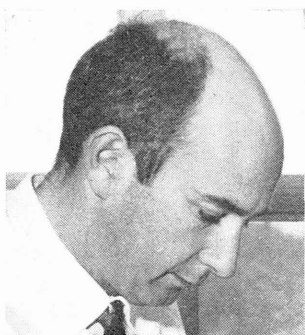
Enquiries about and requests for *World Animal Review*, or any other FAO publication, may be sent direct to The Chief, Distribution and Sales Section, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy.

### AIFST/CSIRO Flame Sterilization Workshop

Production of the book of lectures from the Specialist Course for the food industry ('News from the Division' in *CSIRO Food Research Quarterly* Vol. 32, No. 1) was delayed. It is now ready, however, and copies are available from Mr G. Fisher, Technical Secretary, CSIRO Division of Food Research, P.O. Box 52, North Ryde, N.S.W. 2113, at \$5 each. Cheques should be made payable to 'AIFST Food Engineering Group'.



## Iliano Mario Coggiola, 1935-72



Mr I. M. Coggiola, known to all in the Division as Ili, died suddenly at his home on 23 April 1972.

Ili joined the Division of Food Preservation at Homebush as a Technical Assistant in 1954. He was promoted to Experimental Officer in 1960 after receiving the Diploma in Applied Biology from the University of New South Wales. He obtained his B.Sc. in 1961 and M.Sc. in 1968.

While an assistant Ili showed considerable competence and increasing initiative. His research calibre became apparent at the time of his promotion when he undertook the solution of problems encountered in the experimental fumigation of oranges with ethylene dibromide to kill the eggs and larvae of the Queensland fruit fly. In the fumigation experiments at Gosford a large proportion of the ethylene dibromide was not recovered from the air and the oranges, and it was not known whether it was absorbed by the equipment or decomposed. Ili obtained the answer by studying absorption in all-glass equipment of his own design from which the introduced ethylene dibromide could be recovered quantitatively. He obtained many data that could be applied to commercial fumigation and gave considerable help to the fumigation programme. His aptitude for applied as well as fundamental problems became apparent.

He worked on the anaerobic decomposition of ascorbic acid while an assistant and later undertook the identification of the major products of decomposition in the pH range of foods. His identification of 2,5-dihydro-2-furoic acid was the result of some very difficult and painstaking work.

His work with me on the chemistry of

$\alpha$ -farnesene and its role in the storage disorder superficial scald of apples is still very fresh in my mind. In this work a mature Ili was a true colleague, contributing equally in ideas and in the experimental work. He was involved in the preliminary work that led to the breakthrough. We were under considerable pressure at the beginning of 1964 when I had just arrived back from overseas and learned that Dr K. E. Murray had isolated and identified farnesene. We decided to do everything possible to exploit this result in the fast-approaching apple storage season. There was much to be done in developing techniques, but Ili gave himself wholeheartedly to this problem and we were ready when the fruit was due. He had already begun his own studies on the oxidation of  $\alpha$ -farnesene when I retired, and I felt that further progress was in the very capable hands of Dr E. F. L. J. Anet and himself.

His friendliness and sterling character endeared him to everyone in the Division at North Ryde and I am sure that all, including his young widow, will support this tribute.

FRANK E. HUELIN

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# Selected Publications of the Division

## From the Food Research Laboratory

Copies of most of these papers are available from the Librarian, CSIRO Division of Food Research, Food Research Laboratory, Box 52, P.O., North Ryde, N.S.W. 2113 (Telephone 888 1333).

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Copies of most of these papers are available from the Librarian, CSIRO Division of Food Research, Meat Research Laboratory, Box 12, P.O., Cannon Hill, Qld. 4170 (Telephone 95 2122).

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### From the Dairy Research Laboratory

Copies of most of these papers are available from the Librarian, CSIRO Division of Food Research, Dairy Research Laboratory, Box 20, P.O., Highett, Vic. 3190 (Telephone 95 0333).

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