

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

Standard Methods for the Analysis of Oils, Fats and Derivatives

7th Revised and Enlarged Edition

Prepared by C. Paquot and A. Hautfenne



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International Union of Pure and Applied Chemistry
Applied Chemistry Division
Commission on Oils, Fats and Derivatives

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7th Revised and Enlarged Edition

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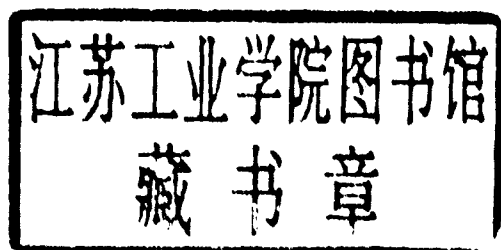
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International Union of Pure and Applied Chemistry

Commission on Oils, Fats and Derivatives

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Foreword

Since the publication of the 6th edition in 1979, the Commission on Oils, Fats and Derivatives has continued the work of adapting methods contained in the 5th edition which had not been included in the 6th edition. In addition the Commission has adopted many new methods and these have been published in *Pure and Applied Chemistry* (PAC) over a number of issues as '1st Supplement Part 1 . . . 6'.

Furthermore methods:

2.323 Determination of triglycerides by gas-liquid chromatography

2.404 Determination of total sterols content

2.431 Determination of the erythrodiol content

have been or will be published soon in PAC accompanied by a statistical report relating to the results of the collaborative study carried out before adoption of these methods. Method:

2.611 Detection of mineral oils in vegetable and animal oils and fats has not been published in PAC.

The present volume—the 7th edition—is the 6th edition enlarged and including these methods and the following main amendments:

- When drafting the 1st Supplement the intention of having a Part 1 devoted to Sections I and II, and a Part 2 devoted to the other Sections (as expressed in the foreword of the 6th edition published in 1979) was abandoned. Consequently a division of the 6th edition into several parts is no longer valid but the methods are grouped under specific subject headings, i.e. in sections.

- In order to have a more consistent numbering, two methods published in the 1st Supplement have had their number modified as follows:

No, 1st Supplement	No, 7th Edition
2.323 Solid Content Determination in fats by NMR (Low Resolution Nuclear Magnetic Resonance)	2.150

2.404 Identification and determination of tocopherols	2.411
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- An important correction has been made to method 2.209 Determination of polyunsaturated fatty acids with a *cis, cis* 1,4-pentadienoic structure.

The Commission on Oils, Fats and Derivatives of the IUPAC

It seems highly appropriate to reproduce here the following account, written in 1959 by J. Vizern and published as a preface to the 5th edition of the *Standard Methods for the Analysis of Oils, Fats and Soaps*, when the Commission was known as the Oils and Fats Division.

The oils and fats division of the IUPAC

Founded 29 years ago, since when it has had many names and many forms but has remained unchanged in spirit and purpose, the Oils and Fats Division—now within the framework of the International Union of Pure and Applied Chemistry—has won distinction which will again be enhanced by the publicizing of the methods which it has adopted so far.

Its task consisted essentially in the unification of the analytical methods indispensable to the manufacture of, and trade in, fat products; it has subjected the methods employed to a searching examination based exclusively on the results of analyses of identical samples carried out by its members. Some of these methods could be adopted rapidly, some had to be tested and refined by means of patient research, others are being, or will be, studied further, still others have finally been rejected.

Publications prior to and including that of 1954, comprising the texts issued and accepted over many years, needed re-editing in a more uniform style; further, new methods have been adopted since 1954 so that a new publication is called for. It cannot be a final version, since the work is continuing, but it would render the services which one may rightly expect from standardization.

The first attempt at unification was carried out in 1924. In April of that year Professor Fachini, Director of the Experimental Station for the Oils and Fats Industry, invited a number of specialists to Milan to discuss an analytical paper drawn up by an Italian commission whose work he had directed.

In addition to the Italian chemists, Mr G. Wolff and I represented France, and Dr A. Gruen, at that time Director of the Schichtwerke Laboratory, Aussig, represented Czechoslovakia. Germany was not represented, as Professor Helde was taken ill and had to abandon his journey. Great Britain's interest in the Conference was shown only by the submission of a paper from the Norman Tate Laboratory, Liverpool.

No decision could emerge from so small a meeting which, moreover, the participants attended in a private capacity. Nevertheless, in the course of the two-day meeting no fundamental differences had arisen between the points of view and, consequently, the path towards unification was clear.

In fact, no progress was made in the first few years, since the attempt made in Milan was not repeated. And yet it deserved to be, scientific language should be both unequivocal and international with regard to the definitions it uses as well as the techniques which these imply. Should this double character be lacking, difficulties arise in international contracts which both parties sign in good faith but interpret differently. For example, 'density' may be interpreted with respect to water at 15°, or as 'specific mass' related to water at 4°.

A contract stipulates the conditions of sale of oil after deduction of impurities: one party will determine the insoluble part in an organic solvent which leaves any soaps present on the filter, whereas the other party will regard only the mineral constituents of these soaps as impurities.

A contract specifies a fat product with an acidity not exceeding a certain limit. One party expresses acidity in terms of oleic acid, the other in terms of lauric acid. At first the product will not meet the specification, even though the other party is satisfied. No matter how improbable such discord may seem, it is not unknown, and I can recall a case of arbitration concerning the acidity of illipé seeds sold by a Dutch firm to a French firm. The arbitrators were unable to agree and the chief arbitrator then had to apply a Solomonian judgement to the effect that the mean molecular weight of the illipé fatty acids could be used to evaluate the acidity of this product in terms of lauric acid.

The same applies to the procedures used, for in every analytical determination carried out on complex organic mixtures there are empirical elements. The slightest modification in procedure will, under identical conditions, give different results, so that it is worth while to work out a standard procedure which, while paying due attention to theory, will give reproducible results.

In spite of these obvious facts, objections to this standardization have been raised by certain people who were under the impression that they had found the only correct method and found the retention of their monopoly profitable. Others again were sceptical. However, in the beginning there were a few people who realized that it would be advantageous to establish agreement not only about definitions but also about results, for analytical differences are harmful to the parties concerned and even more so to their experts.

Standardization does not cause loss of individuality. A procedure, however completely described, does not detract from the skill of those carrying out the determinations, and, moreover, there are numerous special cases in which initiative may play a part. Finally, there is the interpretation of the results and the conclusion, for which no procedure whatever can replace the critical judgement and the experience of the analyst.

Not until November 1929 could discussions on the subject of standardization be renewed. At the Barcelona Congress of the Société de Chimie industrielle française, Professor Veselý of the Czechoslovak Polytechnic at Brno made a plea for standardization and reproached us, and in particular the French members, for obstructing—by our inactivity—the efforts made in Italy and in Central Europe, where committees had been formed.

That decided the matter. Immediately after my return from Barcelona I made an attempt to set up a 'French Committee for the Standardization of Analytical Methods for Fat Products and Their Derivatives'. Most French chemists who specialized in fat chemistry joined this committee, with Professor Rivals as President and Mr Halphen as Vice-President. A draft set of methods was submitted to them. In January 1930, after three days of discussions, a compilation of recommended methods for the analysis of fat products and soaps was published.

This new body enabled Professor Fachini, in April 1930, to call a meeting, in Geneva, of the National Committees of the most important European countries and to invite representatives for those countries where National Committees had not yet been established.

Without any official recognition, and supported only by the acknowledged competence of the members representing Germany, France, Italy, The Netherlands, Switzerland and Czechoslovakia, the assembly proclaimed itself the International Commission for the Study of Fat Products. Professor Fachini was appointed President, Professor Steger, of Delft University, Vice-President, and Dr Sporer, assistant to Professor Fachini, Secretary.

The Commission drew up its rules, and especially those governing its composition—three

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members at most for each participating country: in addition, technical discussions were immediately started. It is not surprising that at this first meeting, at which the members were not well acquainted with each other and at which everybody tried to overcome the language barrier and the difficulties caused by imperfect translations, only the definition of acidity and the method of its determination were arrived at.

Later it was apparent that the confidence resulting from better mutual understanding improved the output of the meetings and that the slow progress was due less to the diversity of languages than to differences in mentality and different ways of thinking, and also to habits often nearly amounting to prejudices.

However, at the following meetings: Zurich 1931, Prague 1932, Rome 1933 (at which the Presidency was given to the doyen Professor Rivals, the Vice-Presidency to Professor Franck (Germany) and the Secretariat to myself) and Paris 1934, important results were obtained.

But at the meeting in London 1935 and Lucerne 1936, as a result of internal changes, little progress was made. The United Kingdom, which until then had only been represented by observers, joined the International Commission. Naturally, the procedures previously adopted were submitted to the new member for approval. In addition, the German delegation including Professor Franck and Dr Rietz which, since Geneva, had been sponsored by the 'Wizoeff', was presided over in London by Professor Täufel and later, in Lucerne, by Professor Kaufmann, the 'Wizoeff' having become the Deutsche Gesellschaft für Fettforschung.

Moreover, apart from the technical discussions, part of the sessions was occupied in working out statutes which had become necessary in view of the possible affiliation to IUPAC, which was achieved at the Lucerne meeting.

At Lucerne, the Presidency of the Commission was given to Professor Veselý, and Professor Kaufmann was appointed Vice-President. The International Commission submitted to IUPAC the internationally adopted specifications relating to the following methods:

Water and Volatile Constituents, Impurities, Ash, Acid Value, Saponification Value, Iodine Value, Density, Refractive Index, Unsaponifiable Matter.

It was not much to show after six years, but this only stresses the difficulties attached to the enterprise. However, with the support of the IUPAC and having attained a certain cohesion and a spirit of confidence and even of friendship among its members, which makes for better mutual understanding, the Commission could look forward to greater efficiency in future. A working meeting was held at Paris in 1937, and at Rome in 1938 where Professor Kaufmann was elected President. It was intended to meet again in Berlin in September 1939, but the war occasioned a long and painful break.

The resumption of the meetings took place in London in 1946. The Commission was established again under the Presidency of Dr G.L. Voerman (Netherlands), whose initiative led to Integration of the Commission into IUPAC as a sub-section of the Section of Applied Chemistry. The Secretariat was again entrusted to me. At this new start the Commission comprised members from: Denmark, the USA, France, the UK, Italy, The Netherlands, Switzerland and Czechoslovakia.

The activities of the Commission were resumed at the London meeting in 1947. The meeting envisaged for Paris in 1948 was cancelled. We then thought it would be sufficient to meet every two years, at the same time as the IUPAC, but since the Amsterdam meeting in

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1949 the annual routine has been resumed as it was considered that the efficiency would be adversely affected by long periods without contact. At Amsterdam, Spain was admitted as a new member. Dr Voerman, the President, having expressed his desire to retire from his office, was replaced by Dr K.A. Williams, with Mr Foster Dee Snell (USA) and Dr H. Sturm (Switzerland) as Vice-Presidents.

Then came Paris, 1950, and New York, 1951. In order to bring its regulations into line with the statutes of the Union, a Sub-Commission which was incorporated in the IUPAC. Foster Dee Snell became President and H. Sturm Vice-President.

At Oxford, 1952, the Commission changed its name and became the Oils and Fats Division of the Section of Applied Chemistry of the IUPAC; and the Sub-Commission became the Division Committee.

Austria, which before the war had been represented by Dr Halden as observer, and Belgium, which had meanwhile been admitted, became members of the Division at the Stockholm meetings in 1953. The present author was appointed President and Dr H. Sturm Vice-President, while the Secretariat was entrusted to Mr J.-E. Bertrand (Belgium). It was important for the Secretary to have a perfect knowledge of French, for from the beginning it had been decided that the authorized versions should be worded in this language. Since that time the correctness of the choice has been demonstrated by his activities and the efficiency with which he has dealt with the increasing amount of work which has resulted from the participation of more and more countries.

Germany, which, in the circumstances, had not taken part since 1939, again joined the Commission at Seville in 1954.

The Division Committee submitted some modifications because of the limitations on the periods of office imposed by the statutes of IUPAC Dr S.H. Bertram (Netherlands) became Vice-President.

Then came the meeting in Zurich in 1955 and in Copenhagen in 1956.

In 1957, at Paris, Dr H.K. Sturm was elected President, with Professor Jacini (Italy) Vice-President.

In 1958, at Brussels, Ireland was admitted to the Division. The Argentine, which had been represented by Professor Cattaneo for three years, again became a member. Fourteen countries are, therefore, represented. Sweden and Israel are on the waiting list for membership.

In 1959, at Munich, the Division was in a position to submit to the Bureau of IUPAC, with a view to publication, more than forty test methods relating to oil seeds, fat products, glycerides and soaps, which had been subjected to a very strict selection after long co-operative studies and comparisons. The compendium of these methods will constitute a work of reference. The door has been opened for the admittance of new trends. Thus, after five years of, at times, heated discussions, struggling against ingrained habits which could not apparently be broken, the Division has now rejected subjective colour determinations in favour of the photo-electric eye of the spectrometer. The document is not final; work is still progressing, certain methods can be modified, others are being studied.

No human task is perfect nor ever finished, but all those who have participated in this work may be proud. Towering above them all is Professor Fachini, who initiated the project and who has inspired all. The thirtieth anniversary of the Division will be celebrated in 1960, in his country.

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Some members have passed away. My thoughts go out to the memory of Professor Rivals and of Dr Voerman, both of whom presided so ably.

But a team continues to operate. The names of those who compose it are a guarantee of its quality.

November 1959

J. VIZERN

Former Vice-President of the Applied
Chemistry Section of IUPAC
Former Chairman of the Oils and
Fats Division of the IUPAC

Since 1959, nearly 20 years have passed and the administrative history of those years may be briefly summarized.

Annual meetings of the Division have been held: after meeting at Bari (1960), London (1961) and Prague (1962), the Division met again in London (1963) when H.J. Vos replaced J. Bertrand as Secretary, and the 'Oils and Fats Division' was renamed the 'Oils and Fats Section'. Later, two of the founder members retired: G. Wolff in 1964 at Rotterdam and J. Vizern at Paris (1965).

Meetings were held at Dublin (1966), Prague (1967), Vienna (1968) and at Cortina d'Ampezzo in 1969: at this meeting the 'Conseillers' became 'Titular Members' and 'Associate Members' were designated.

Stockholm was the venue in 1970 and St Gallen in 1971, when, at the behest of IUPAC, the Oils and Fats Section was divided into two Commissions: the 'Commission for Oils and Fats' and the 'Commission for Soaps and Oleochemicals', the Titular Members being the same in both Commissions.

In 1972 the meeting was held at Chester, and at Munich in 1973, H.J. Vos became President and was succeeded as Secretary by C. Paquot.

Subsequent meetings were held at Jablonna (1974) and Madrid (1975), and the 'Section' was re-entitled the 'Oils and Fats Commission'; the two former Commissions were reunited in the Commission, which has to conform progressively to the bye-laws of IUPAC: eight Associate Members (but twelve until 1979) and one National Representative per country (two until 1977) are allowed.

Following Lyngby in 1976, the 1977 meeting was held at Warsaw: the President, H.J. Vos, yielded the chair to C. Paquot and A. Hautfenne became the new Secretary. The Commission altered its name to 'Commission on Oils, Fats and Derivatives'.

Since 1959 the number of adhering countries of the Commission has increased: Hungary and India joined in 1971, as well as Nigeria (until 1976); in 1972 Canada, in 1973 Japan and New Zealand, in 1974 Bulgaria, in 1975 Norway, in 1976 Australia and in 1977 Greece and Turkey joined in the work. Argentina, however, is no longer a member of the Commission.

Over the years, the Commission has been saddened by the deaths of its last founder members: Professor Vessely in 1964, J. Vizern in 1968 and G. Wolff in 1969, but the memory of them will always remain green, because the Commission owes so much to them.

It is useful to record that, since 1959, more than thirty new methods have been added to the fifty or so previously published. The work continues: twelve methods are now under study;

likewise, the revision of Sections III (Glycerines) and IV (Soaps) proceeds, while the preparation of further Sections is in hand.

So we have good reason to claim that the Commission on Oils, Fats and Derivatives continues its efforts to be useful to all.

C. PAQUOT
President of the Commission on
Oils, Fats and Derivatives

Only eight years have elapsed since the publication of the 6th edition of our Methods. During this period, which though short, has been none the less rich in activities and results, life of the Commission on Oils, Fats and Derivatives may be summed up as follows, by the rhythm of the meetings, which have taken place yearly.

At the 1977 Warsaw meeting, C. Paquot became Chairman, assisted by E. Delvaux, Vice-Chairman, and A. Hautfenne, Secretary. After Brussels in 1978, the Commission met in Davos (Switzerland) in 1979 where E. Delvaux retired having reached the university age limit.

The 1980 Paris meeting gave the opportunity of celebrating the 50th anniversary of the Commission.

In Leuven (Belgium) in 1981, C. Paquot gave up the Chairmanship to D. Firestone; M. Naudet became Vice-Chairman, A. Hautfenne keeping the Secretariat. In order to conform to the Statutes and by-laws of the Union, the Commission was obliged to reduce to six the number of its Titular Members but was authorized to bring to twelve the number of its Associate Members.

After Zeist (The Netherlands) in 1982, came Lyngby (Denmark) in 1983, when M. Naudet succeeded D. Firestone as Chairman, A. Hautfenne became Vice-Chairman and W.D. Pocklington became responsible for the Secretariat.

After its meeting in 1984 in Turku (Finland), the Commission met in Lyon (France) in 1985.

During this period, the Commission has seen the number of represented countries increase very significantly: in 1979, Brazil, Finland and Yugoslavia (until 1981) rejoined the Commission but Norway left; in 1981 Argentina rejoined, after an absence of four years, together with Egypt, Romania, South Africa and the USSR; Israel and Malaysia joined in 1983.

During the same time, several changes arise among the Members of the Commission mainly due to modification or ceasing of activities. Unfortunately one must also mention the death of two very long-serving Members: Mrs E. Lewkowitsch-Morton and K. Williams.

Revision of methods included in Sections III and IV of the 5th edition has been completed and the revised texts have been published in *Pure and Applied Chemistry* (PAC) as Supplement to the 6th edition. Fifteen new methods have been investigated, adopted and drafted. They have also been published in PAC and they are incorporated in the present edition.

About twenty other methods are already at the final draft stage or are only investigated. After their adoption and drafting, they will be published in PAC with the statistical report relating to the collaborative study.

COMMISSION ON OILS, FATS AND DERIVATIVES

The Commission has strengthened existing bonds and has created new ones with several International Organizations by means of a constant collaboration and exchange of Observers.

The general objectives assigned to the Commission on Oils, Fats and Derivatives continue to be pursued energetically, thanks to the dedication of all its Members.

January 1985

M. NAUDET
Chairman of the Commission on
Oils, Fats and Derivatives

Warning

Some of the methods require the utilization of particularly flammable and/or poisonous solvents.

Included in these solvents are: benzene, carbon tetrachloride (Wijs solution for the determination of the iodine value), and chloroform.

In these cases, the safety instructions for the handling of these solvents should be observed.

The procedure must be carried out in a well-ventilated room, preferably in a fume cupboard, to prevent the exposure of laboratory workers to poisonous vapours.

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Section 1

Oleaginous Seeds and Fruits