European Cooperation in the field of Scientific and Technical Research
- COST -

Secretariat

Brussels, 21 January 2004

COST 226/04

DRAFT MEMORANDUM OF UNDERSTANDING

Subject :	Draft Memorandum of Understanding for the implementation of a European Concerted Research Action designated as COST Action E41 "Analytical Tools with Applications for Wood and Pulping Chemistry"

Delegations will find attached the abovementioned Memorandum of Understanding.

DRAFT

MEMORANDUM OF UNDERSTANDING

FOR THE IMPLEMENTATION OF A EUROPEAN CONCERTED RESEARCH ACTION DESIGNATED AS

COST E41

"ANALYTICAL TOOLS WITH APPLICATIONS FOR WOOD AND PULPING CHEMISTRY"

The Signatories to this Memorandum of Understanding, declaring their common intention to participate in the concerted Action referred to above and described in the Technical Annex to the Memorandum, have reached the following understanding:

1. The Action will be carried out in accordance with the provisions of document COST 400/01 "Rules and Procedures for Implementing COST Actions", the contents of which the Signatories are fully aware of.

2. The main objective of the Action is to develop and evaluate analytical methods related to wood, pulping and bleaching chemistry.

3. The economic dimension of the activities carried out under the Action has been estimated, on the basis of information available during the planning of the Action, at Euro 25 million in 2003 prices.

4. The Memorandum of Understanding will take effect on being signed by at least five Signatories.

5. The Memorandum of Understanding will remain in force for a period of four years, calculated from the date of first meeting of the Management Committee, unless the duration of the Action is modified according to the provisions of Chapter 6 of the document referred to in Point 1 above.

TECHNICAL ANNEX

COST E41:
"ANALYTICAL TOOLS WITH APPLICATIONS FOR WOOD AND PULPING CHEMISTRY"

A. BACKGROUND

A variety of analytical methods are needed in the research aiming at clarifying wood chemistry and at improving delignification and bleaching methods, as well as evaluation of the properties of the pulps for paper making.

State-of-the-art and current state of knowledge

The main principles of the chemistry involved with pulping and bleaching have been discovered during the intense research activity in the field for several decades. Many of the basic analytical tools are still relevant and widely applied.

In many cases, several parallel methods, based on different analytical techniques and principles, have been developed for the same purpose. The results obtained by the different methods are not always equivalent. The main reason for this is the complexity of the matrix: fibre and isolated fibre components. In many cases, it is not possible to define the exact structure of the analysed component; rather it is a question of more general characterisation. Thus, the different techniques including different sample pre-treatments may provide complementary information, but their correct interpretation is a challenge.

In recent research, a number of chemistry-related problems have emerged, which require development of new analytical techniques. The technical development of the analytical instruments has brought about possibilities for new applications. It is also probable that analytical techniques, already developed for other purposes, could be applied in the pulping research either as such or after modifications.

Links and complementarity with EU research programmes

There have been several EU research projects connected to the development of chemical pulping and bleaching. These projects have focused on process development and pulp properties rather than chemical aspects. However, chemical analysis methods have been part of these projects as tools, helping reach the final goals. In these applied projects, only a limited amount of actual development of analytical methods has been included, but they have provided a forum for the partners to exchange information and experience of the different methodologies. Recent projects of this type (ended or active):

- Oxidative Enzymes for the Pulp and Paper Industry, "OXEPI" DG-XII, FAIR-No CT95-0805
- Bleachability of alkaline pulps, "PulpBleach" FAIR-CT98-3460:
  Towards efficient oxygen delignification, "OXYDELIGN" Quality of Life Program QLK5-CT-1999-01277
- New environmentally-sound methods for pitch control in different paper pulp manufacturing processes", acronym Pitch, FP5 QLK5-99-1357
- Fungal metalloenzymes oxidising aromatic compounds of industrial interest, acronym Pelas, FP5 QLK3-99-590
- Wood extractives in pulp and paper manufacture: Technical and environmental implications and biological removal", acronym Web, FP4 FAIR-CT95-0560
- Concerted Action "Co-ordination network for lignin – standardisation, production and applications adapted to market requirements, EUROLIGNIN" (Promoting Competitive and Sustainable Growth -programme) focuses on lignin utilisation, and is thus related to this COST Action, as lignin is one of the main fibre components.

The COST Action

There is a strong interest and a lot of research activities in Europe in the field of chemical fibre characterisation. This was evidenced *e.g.* in the 7th European Workshop on Lignocellulosics and Pulp, held in Turku, Finland, August 2002. The subtitle chosen for the workshop was "Towards molecular-level understanding of wood, pulp and paper". Accordingly, a large number of papers from many European countries dealt with chemical analysis of fibre and fibre components.
It is clear that there are many research groups in Europe with similar interests, which would benefit

from co-operation and exchange of experience. It is also believed that research on development of analytical tools and techniques that could be applied in the area of wood and pulping chemistry, is carried out in universities without contacts with the pulping industry. These should be integrated into this COST Action. Short-Term Scientific Missions would be a good way to distribute new methodologies between laboratories.

B. OBJECTIVES AND BENEFITS

The main objective of the Action is to develop and evaluate analytical methods related to wood, pulping and bleaching chemistry.

The secondary objectives are:

- to create a platform for interaction between scientists in Europe to provide a good transfer of knowledge
- to evaluate the potential and restrictions of the presently available chemical analytical methods for wood and pulp fibre and fibre components
- to develop further the presently available chemical analytical methods for wood and pulp fibre and fibre components
- to evaluate the possibilities to apply new chemical analysis methods developed in other research fields for wood and pulp fibre and fibre components
- to evaluate the potential of the analytical methods even for non-woody lignocellulosic raw materials used by the pulp and paper industry
- to identify the topics where more research should be focused

The expected benefits to the European pulp and paper industry are:

- Promotion of the development of pulping and bleaching operations towards high and even pulp quality
- Promotion of the development of environmentally benign pulping and bleaching methods
- Promotion of the development of cost effective processes for pulping and bleaching
- Promotion of the development of tools for process control in pulping and bleaching operations

C. SCIENTIFIC PROGRAMME

The Action will include the following types of activities:

(a) workshops and seminars, that could result in publication of a series of monographs
(b) exchange of experts, scientists and graduate students for training
(c) exchange of reports, publications and experimental procedures
(d) state-of-the-art conferences and workshops

The scientific areas under consideration will be those focusing on methods for chemical characterisation of fibres and fibre components.

As wood or pulp fibre is a complex material, isolation of its components for analysis is a critical step. Comparison of isolation methods, and evaluation of their advantages and disadvantages including the representativeness of the obtained sample, will be part of the Action.

Scientific rationale of the specific aspects

Lignin and lignin-carbohydrate complexes

One challenging topic is the analysis of the molecular size of lignin. During pulping, lignin is leached out of the fibre through the porous cell wall. Also in mechanical and chemimechanical pulping part of the lignin is leached out. As the pore sizes in fibre are of the same magnitude as the diffusing components, this may be a rate-limiting step in delignification. Physicochemical characterisation of lignin, *e.g.* its hydrodynamic volume at the applied reaction conditions, is therefore crucial. In addition to depolymerisation, the structure of lignin is altered during pulping and bleaching. The formation of ionisable phenolic and acidic groups enhances the solubility of lignin, whereas the formation of condensed structures hinders delignification. Structural characterisation of lignin is essential in order to understand the chemistry behind the pulp manufacturing processes. Radicals are involved in many of the reactions of lignin especially during bleaching. Therefore, methods for analysing the radical intermediates will be included in the Action.

One of the main obstacles in delignification is linkages between lignin and pulp polysaccharides. The presence of lignin-carbohydrate complexes (LCC) hinders lignin dissolution even if the lignin itself would be hydrophilic. Therefore, characterisation of LCC is an important topic in the Action.

Examples of suitable techniques for the characterisation of lignin and LCC are listed below. Each of them is comprised of several specific methods for various purposes. Especially in the case of LCC analysis, even carbohydrate analysis methods are required:

- Isolation and fractionation methods (*e.g.* chemical and enzymatic hydrolysis techniques, preparative chromatography)
- Chromatographic techniques (*e.g*. size exclusion chromatography, typically combined with UV detection, LC/MS)
- Capillary electrophoresis (mobility, charge)
- Determination of elemental composition
- Degradative techniques (*e.g.* oxidative degradations, analytical pyrolysis)
- Wet-chemical methods for functional group analysis (*e.g*. phenolic hydroxyl groups, total hydroxyl groups, carboxylic acids and methoxyl groups)
- Spectroscopic techniques (*e.g.* FTIR, Raman, UV)
- Mass spectrometric methods (*e.g*. electron spray ionisation)
- NMR techniques (liquid and solid; pulse field gradient)
- ESR for radical analysis

Polysaccharides

Another important aspect is the analysis of the reactions of pulp polysaccharides, in particular their depolymerisation due to unspecific delignification reactions, which deteriorates pulp quality. Therefore, the degree of polymerisation of the polysaccharide components, especially cellulose, is essential to know. Polysaccharides may also react without simultaneous chain rupture leading to altered chemical structure, possibly more prone to degradation in later pulping and bleaching stages. In the case of hemicelluloses, dissolution leading to yield losses is the most critical reaction. In

mechanical pulping, hemicelluloses, together with extractives, form the colloidal material in the process waters. As in the case of lignin, the dissolution of hemicelluloses is affected by physicochemical aspects, which need to be taken into account. In addition to the main pulp polysaccharide components, recent results have indicated that some minor components, *e.g*. galactans, may have crucial roles in pulping and bleaching.

Examples of suitable techniques for the characterisation of polysaccharides are listed below:

- Isolation and fractionation methods (*e.g*. extraction sequences)
- Size exclusion chromatography (detection based on RI, MALLS, viscosity)
- Mass spectrometric methods (MALDI-TOF, electron spray ionisation)
- Compositional analysis (hydrolysis into monosaccharides and chromatographic separation, methanolysis)
- Structural analysis (NMR, enzymatic methods, degradative methods)

Extractives

Wood extractives induce problems in the pulping line, forming deposits in pulp and equipment. They have a significant effect on fibre properties, especially on those of mechanical and chemi-mechanical pulps. Many extractives are surface-active compounds and in the paper they affect the surface properties, such as the binding between fibres, the water adsorption and friction. Further, some extractives have been identified as main contributors to the toxicity of untreated effluents.

Strong interactions have been proposed to exist between extractives and other pulp components, evidenced by the presence of “unextractable extractives”. The impact of this kind of material on the pulping process and final pulp and paper properties are not known. Also in general, the determination methods for extractives, including sample pre-treatment, need to be compared and evaluated, both for pulp and paper samples and for water samples in order to find optimal analytical procedures. The variation in the composition of the extractives between different wood species and also between different types of pulps, e.g. chemical pulps, mechanical pulps and pulps from de-inked pulp, must also be considered. Examples of suitable techniques for the characterisation of extractives are listed below:

- Extraction (various equipments, solvents and sample pre-treatments)
- Chromatographic techniques
- Spectroscopic methods
- Analytical pyrolysis

Most of the methods require isolation of the sample before analysis. However, there are also methods which can be applied directly on the fibre sample. Depending on the method, these are either surface specific (e.g. ESCA, TOF-SIMS), or they detect the whole fibre (*e.g.* UV resonance Raman). These methods are interesting, since the often-tedious sample preparation can be avoided, and representativeness is not a problem.

Efficient analytical methods are required in the research laboratories in order to get new information about the chemical composition of wood and pulp components. However, an increasing amount of analysis data is nowadays needed directly from the process. Therefore, emphasis will also be put to the development of analytical on-line applications.

Hyphenated techniques (combination of several analytical techniques) and application of statistical multi-component analyses are also included in the Action.

Analytical methods used in closely related areas like biotechnology, synthetic polymers and biomass utilisation could in many cases be applied, probably with some modifications, also in the wood and pulping chemistry.

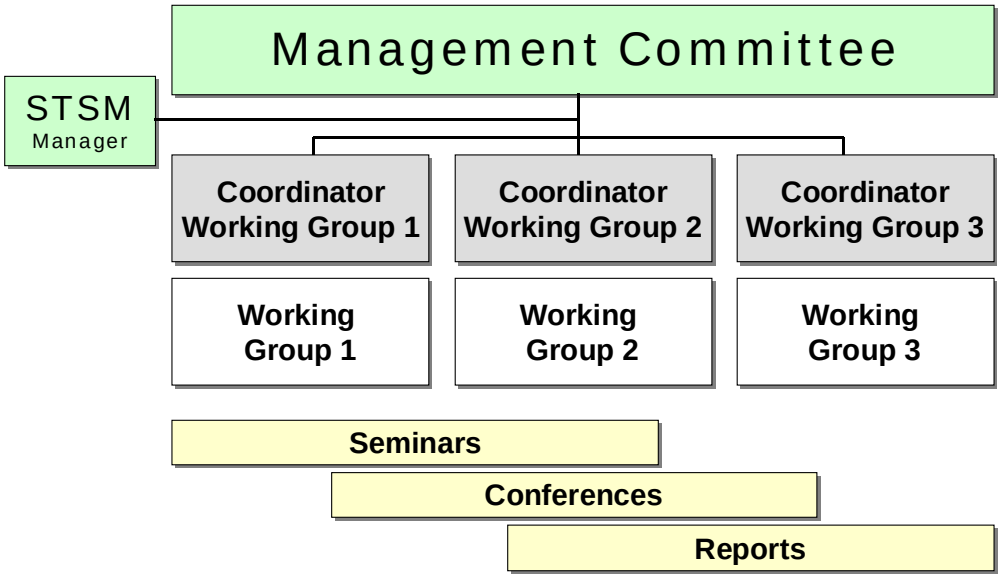
Analytical methods and techniques are needed for:

- Research purposes; to get new information on the chemical composition of wood and fibre
- Process and/or product quality control and assessment purposes. These could be realised as “routine type” analyses in mill laboratories or as on-line process applications

D. ORGANISATION

D.1 Organisation, management and responsibilities

The organisational structure of the Action is depicted in the following chart. A Management Committee organises and controls the Action. Three Working Groups (WGs) will be formed (WG 1, WG 2 and WG 3) to cover the scientific areas. All WGs will organise seminars and conferences. Dedicated reports will be published by the WGs (see also section F). A Short-Term Scientific Mission (STSM) Manager will organise and be responsible for the STSMs.



D.2 Working Groups

The Action will be organised in three WGs in accordance with the specific aspects outlined in section C. Interaction among the WGs will be essential, primary interfaces are indicated in the description of the scientific programme (see section C). Each WG will elect a Leader and Deputy Leader, and they will assist the Management Committee Chairperson and Vice-Chairperson in ensuring that the Groups' objectives as stated in the Memorandum of Understanding are achieved and of a high standard. The WGs will hold separate meetings, once or twice per year if possible. Wherever possible the Management Committee, WGs and other meetings associated with the Action will be held at the same time and venue.

Working Group 1: Characterisation of lignin.

Working Group 2: Characterisation of polysaccharides.

Working Group 3: Characterisation of extractives.

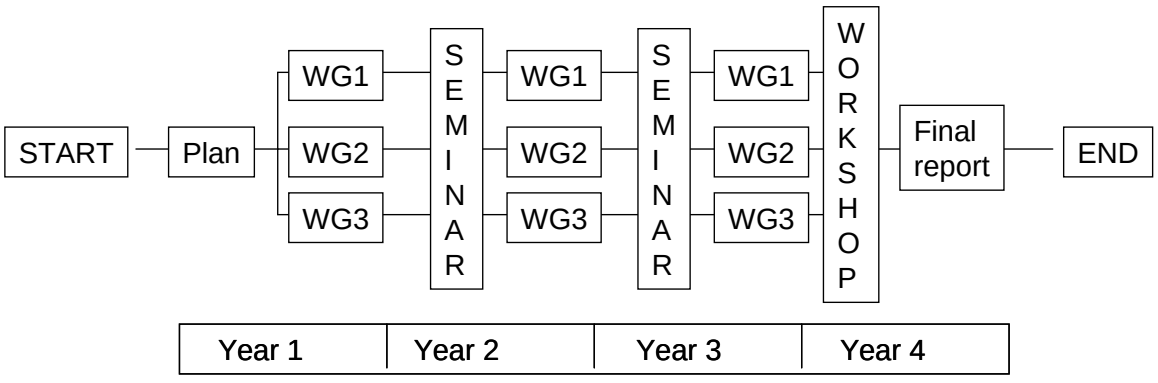
In addition to the separate WG meetings, seminars will be organised once per year. These will be joint meetings with all WGs to ensure discussion between the WGs. The final seminar will be organised in the form of an open workshop.

The Action will encourage the exchange of scientists between the participating research laboratories through STSMs.

The COST activity will be carried out in co-operation with existing national and international research programmes related to pulping and papermaking. Scientists from related fields (biotechnology, synthetic polymers) are encouraged to take part in the Action in order to provide input of new ideas. The form of co-operation will be exchange of information.

D.2. Timetable

The Action will run over a period of four years. Meetings and seminars in the WGs will be held according to the plan below. The seminars will be common for the three WGs. The MC meetings and the seminars will be held at the same time.



E. ECONOMIC DIMENSION

The following COST countries have actively participated in the preparation of the Action or otherwise indicated their interest:

Austria, Finland, France, Germany, Greece, Hungary, Italy, Latvia, Norway, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden.

On the basis of national estimates provided by the representatives of these countries, the economic dimension of the activities to be carried out under the Action has been estimated, in 2003 prices, at roughly Euro 25 million.

This estimate is valid under the assumption that all the countries mentioned above but no other countries will participate in the Action. Any departure from this will change the total costs accordingly.

F. DISSEMINATION PLAN

The most important results from the Action will be improved chemical characterisation methods for fibre components. A main target group for dissemination of such results is the pulp and paper industry. The participating partners (mainly national research institutes and universities) will distribute reports and presentations from the Action to the national industry. Industry people will be invited to the final workshop.

Some of the topics covered by the Action are also in the interests of ISO/EN standardisation groups. These groups will be informed of relevant results from the Action.

A web-site for the Action will be created and maintained to enhance communication within the Action, inform the external scientific community and disseminate the results.

The MC will also maintain an active contact to the COST Forestry and Forest Products Technical Committee by:

Establishing close contact with the Technical Committee through its appointed liaison officer and invitation of the MC chairperson or her/his representative at the meetings of the Forestry and Forest Products Technical Committee.

Submitting annual progress report to Forestry and Forest Products Technical Committee.
Contributing to the COST Forestry and Forest products Technical Committee Activity report.