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DECISION
of 18 March 2004

Case Number: T 1055/00 - 3.3.6

Application Number: 93202468.0

Publication Number: 0584879

IPC: C10G 69/06

Language of the proceedings: EN

Title of invention:

Process for the preparation of lower olefins

Patentee:

SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

Opponent:

Exxon Chemical Patent Inc.
Sasol Technology Ltd.

Headword:

Lower Olefin/SHELL

Relevant legal provisions:

EPC Art. 123(2)(3), 100(b), 84, 83, 54, 56

Keyword:

"All requests: Extension of the protection conferred by the patent (no), sufficiency of disclosure (yes), novelty (yes)"
"Main request, first and second auxiliary request: inventive step (no)"
"Third and fourth auxiliary request: Extension beyond the content of the application as filed (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 1055/00 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 18 March 2004

Appellant: SHELL INTERNATIONALE RESEARCH
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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted 18 August 2000
revoking European patent No. 0584879 pursuant
to Article 102(1) EPC.**

Composition of the Board:

Chairman: P. Krasa
Members: G. Dischinger-Höppler
J. H. Van Moer

Summary of Facts and Submissions

- I. This appeal is from the decision of the Opposition Division to revoke the European patent No. 0 584 879 relating to a process for the preparation of lower olefins. The decision was based on an amended set of 8 claims.
- II. In their notices of opposition filed against the patent, the Respondents (Opponents) sought revocation of the patent on the grounds of Article 100(b) EPC for insufficient disclosure and on the grounds of Article 100(a) EPC for lack of novelty and lack of inventive step (Articles 52(1), 54(2) and 56 EPC). The opposition was based inter alia on the following documents
- D6 EP-A-0 161 705,
- D8 C.D. Frohning et al "Chemical feedstocks from coal" in Hydrocarbon Processing, November 1974, pages 143 to 146, and
- A9 B. Büssemeier et al "Lower olefins via Fischer-Tropsch" in Hydrocarbon Processing, 1976, vol. 55, no. 11, pages 105 to 112.
- III. In its decision, the Opposition Division dismissed the objections raised during the opposition proceedings under Article 123, 83, 84 and 54 EPC, but held that the subject-matter of Claim 1 was not based on an inventive step since a skilled person would obtain the claimed subject-matter in an obvious manner by using in the process disclosed in D8, a naphtha feed derived from a

Fischer-Tropsch (hereinafter "FT") synthesis process with cobalt as the catalyst instead of an iron catalyst.

IV. This decision was appealed by the Appellant (Proprietor) who filed under cover of the letters dated 18 February 2004 and 10 March 2004, amended sets of claims in a new main request and in four auxiliary requests. Claim 1 of the main request reads:

"1. Process for preparing lower olefins from a hydrocarbon feed having a boiling point range of from 30°C to 200°C, which process comprises thermal cracking of the hydrocarbon feed, wherein at least part of the hydrocarbon feed is a hydroprocessed synthetic oil fraction which synthetic oil fraction is prepared by a Fischer Tropsch synthesis process, comprising contacting at elevated temperature and pressure a synthesis gas with a catalyst comprising cobalt as the catalytically active component, the elevated temperature being a temperature between 175 and 250°C, and wherein the hydroprocessed synthetic oil fraction has been prepared by hydrogenation of the synthetic oil fraction at elevated temperature and pressure in the presence of hydrogen and a hydrogenation catalyst."

Claim 1 of the first auxiliary request differs therefrom in that the term "lower olefins" has been replaced by "lower olefins, that is, olefins having from 2 to 4 carbon atoms,".

Claim 1 of the second auxiliary request differs from that of the first auxiliary request by the addition of the feature ", and in which hydrogenation process any unsaturated hydrocarbons and oxygenates present in the

synthetic oil are hydrogenated" at the very end of the claim.

Claim 1 of the third auxiliary request differs from that of the first auxiliary request by the addition of the feature "at an average temperature of 840°C, an average pressure of 2.25 bar, a residence time of 0.2 seconds and an inert gaseous diluent hydrocarbon feed ratio of 0.8," between the terms "..., which process comprises thermal cracking of the hydrocarbon feed" and "wherein at least part of the hydrocarbon feed is ...".

Claim 1 of the fourth auxiliary request differs from that of the third auxiliary request in that the term "an inert gaseous diluent/hydrocarbon feed" has been replaced by "a nitrogen/hydrocarbon feed".

The Respondents in turn filed inter alia document

A13b Ullmann's Encyclopaedia of Industrial Chemistry, 5th ed, vol. A10, 1987, pages 46 to 59.

V. The Appellant submitted orally and in writing in summary the following arguments:

- D6 was the closest prior art since it also related to a cobalt-catalysed FT process from which high yields of lower olefins were obtained upon thermal cracking.
- The problem to be solved consisted in providing a process giving a maximum yield of lower olefins from a feed produced by FT synthesis.

- As was shown in the examples of the patent in suit, this problem was solved by hydrogenating the product obtained by a FT synthesis using cobalt as the catalyst. This solution was, however, not obvious since the yields of lower olefins obtained in accordance with the process of D6 from a feedstock produced by a low temperature FT process with cobalt as the catalyst and in the absence of a hydrogenation step were extremely high when compared with the yields obtained according to D8 from hydrogenated high temperature iron catalyst based FT naphtha. Therefore, it was apparent from D6 that in the case of low temperature cobalt catalyst based FT naphtha there was no need at all to perform hydrogenation. Also A9 did not give any hint to hydrogenate the naphtha in D6 before the thermal cracking since it merely indicated that it was essential to hydrogenate specifically the C₅-C₁₁-fraction obtained from a high temperature FT process, but did not inform the skilled reader about any reasons for doing so.

- Therefore, a person skilled in the art had no reason to hydrogenate the FT naphtha obtained in D6 with cobalt as a catalyst in the expectation to improve the yield of lower olefins in a thermal cracking reaction.

VI. The Respondents' arguments were in summary:

- The amendments made to the claims of all requests were not admissible under Article 123(3) EPC due to the deletion of the term "having at least a fraction boiling above the boiling point of the

lower olefins". They were further not admissible under Article 123(2) EPC as far as the second auxiliary request was concerned due to the introduction of an inadequate limiting feature and as far as the third and fourth auxiliary requests were concerned due to the inadmissible combination of general disclosure with features specifically disclosed in particular examples. Moreover, the amendments introduced problems under Article 84 EPC.

- The claimed subject-matter was insufficiently disclosed with respect to the meaning of the term "which process comprises thermal cracking".
- The subject-matter claimed in the main request was not novel in view of A9 since - for those skilled in the art - the reference concerning the additional hydrogenation step was not limited to a product of the Synthol process.
- D8 or A9 were the most appropriate starting points for the assessment of inventive step, but even if one started from D6 as proposed by the Appellant, the claimed subject-matter was not inventive for the following reasons:

It was known from A9 that the olefin content of the FT product was not only dependent on the catalyst used but also on other process conditions such as pressure and H₂/CO ratio. In this respect, the examples of D6 cannot be compared with those of the patent in suit. Consequently, it has not been shown that the technical problem of improving

the yield of lower olefins in view of D6 has been solved, the more so as the claimed process was not limited as to the olefin content of the feedstock prior to hydrogenation. However, it was obvious for the skilled person to reduce the content of olefins in the feedstock before cracking, since it was generally known in the art that highly paraffinic feedstock provides the greatest yield of lower olefins upon cracking. Evidence for that was given in D8, A9 and A13b.

VII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request, or first to fourth auxiliary request (main request, first, third and fourth request filed with the letter dated 18 February 2004, second auxiliary request filed with fax of 10 March 2004).

The Respondents requested that the appeal be dismissed.

VIII. Oral proceedings before the Board of Appeal were held on 18 March 2004.

Reasons for the Decision

All Requests

1. *Amendments (Articles 123(3) and 84 EPC)*

1.1 The Respondents objected to the amendments under Article 123(3) EPC. In their opinion, the term "lower olefins" encompassed embodiments with olefins having carbon numbers greater than 4. The removal from the

claims as granted of the limitation "having at least a fraction boiling above the boiling point range of the lower olefins" and the introduction of the lower boiling point limit of 30°C resulted in claims extending to the preparation of C₅ olefins having a boiling point (42°C) above 30°C from a feed with a boiling point below 42°C. This embodiment, so the Respondents argued, did not fulfil the requirement of the claims as granted that at least a fraction of the hydrocarbon feed must have a boiling point above that of the olefin.

The Board does not agree. The feature in question which was present in the claims as granted reads "process for preparing lower olefins from a hydrocarbon feed having **at least a fraction boiling above the boiling point range** of the **lower olefins**". Thus, the claims as granted cover the production of olefins from a hydrocarbon feed having several fractions with the requirement that at least one of them boils above the boiling point range of the lower olefins whereas all other fractions may boil below that range.

According to the new feature reading "process for preparing lower olefins from a **hydrocarbon feed** having a **boiling point range** of from 30°C to 200°C" the hydrocarbon feed has a boiling point range, not only a particular single boiling point of 30°C. If, therefore, the term "lower olefins" covers C₅ olefins having a boiling point above 30°C, the hydrocarbon feed nevertheless contains one or more fractions boiling above the boiling point of any such C₅ olefins and up to 200°C.

The Board, therefore, concludes that the amendments made to the claims actually provide a limitation of their subject-matter rather than an extension in that the hydrocarbon feed is now limited to one boiling from 30°C to 200°C.

Consequently, the amendments made to the claims comply with the requirements of Article 123(3) EPC.

- 1.2 Objections under Article 84 EPC were made with respect to the terms "lower olefins" and "wherein the hydroprocessed synthetic oil fraction has been prepared by hydrogenation". The Board agrees that both terms might be vague and unsuitable to define clearly the subject-matter either with regard to the actual chain length of the olefins to be prepared or with regard to any distinction between the terms "hydroprocessed" and "hydrogenated".

However, the term "lower olefins" was already contained in the claims as granted and does not result from the amendments. In this respect, no problem under Article 84 EPC was created by the amendment.

In contrast, the second term has been introduced during the appeal proceedings and might be unclear insofar as it does not precisely indicate whether or not the claims encompass further hydroprocessing steps such as hydrocracking or hydroconversion (see column 7, lines 2 to 7 of the patent in suit). According to the description of the patent in suit, the hydroprocessed synthetic oil fraction may, nevertheless, be prepared by hydrogenation only (*loc. cit.*). Since the appeal fails for other reasons (see under 4. below), it is,

for the purpose of this decision, sufficient to discuss the present case on the basis of this definition.

2. *Sufficiency of disclosure (Article 100(b) EPC)*

The Respondents argued that due to the wording "comprises thermal cracking", the claimed subject-matter included further undisclosed process steps. It, therefore, encompassed completely different processes, for example a cracking process with 90% of the cracking being catalytic cracking and only 10% thermal cracking.

However, the Respondents have not provided any evidence to show that the process disclosed in the patent in suit covers embodiments which a skilled person would be unable to carry out. On the contrary, the Board is convinced by the Appellant's argument that it is apparent from the description of the patent in suit which further steps are included in the claimed process, namely feeding and withdrawing chemicals into and from reactors, heating and cooling of material within a reactor and, in particular, fractionating the product obtained by the FT synthesis before its hydrogenation in order to obtain a hydrocarbon feed having a boiling point range of from 30°C to 200°C (see examples).

The Board is, therefore, satisfied that the patent in suit meets the requirements of Article 100(b) EPC.

Main Request

- 3.1 The Board is satisfied that the amendments made to the claims are allowable within the requirements set out in

Article 123(2) EPC. No objections have been made by the parties in this respect.

3.2 Novelty

The Respondents objected to the novelty of the claimed process with respect to the prior art disclosed in A9.

This document is a scientific article concerning the production of lower olefins via FT synthesis. It considers, *inter alia*, hydrogenation and subsequent cracking of FT produced naphtha for ethylene and propylene production.

However, its main concern is to directly produce ethylene, propylene and butenes by FT synthesis which is held to be more promising under economic aspects. The basic research problem was, therefore, to develop a suitable FT synthesis catalyst for that purpose (page 105, left column, first paragraph to right column, second paragraph).

Cobalt and iron catalysts are said to be the classic FT synthesis catalysts (page 105, right-hand column, last three lines) and compared with each other throughout the whole article with the finding that using iron in the FT synthesis is preferable since it produces the higher amount of unsaturated aliphatic hydrocarbons whereas cobalt has a strong hydrogenating activity and yields mainly saturated products (page 108, right-hand column, first full paragraph). The final conclusion in A9 is, therefore, that FT synthesis with particularly modified iron catalysts and using CO rich syngas makes it possible to directly produce up to 50% of short

chain hydrocarbons with 50 to 80% thereof being olefinic. Nevertheless it is suggested to continue research with a view to possibly further increase the olefin selectivity of syngas conversion (page 112, last two paragraphs).

A9 refers in particular to those modifications of the FT process which are known as "ARGE" and "Synthol" processes (page 105, left-hand column, first two paragraphs). Those differ from each other insofar as the ARGE process is run in a fixed bed and at the same low temperature conditions (e.g. 220 to 240°C) as the claimed process (175 to 250°C) while the Synthol process is carried out in a fluid bed at high temperature (e.g. 320 to 340°C) (see also page 108, Table 2).

Additional hydrogenation of naphtha as being essential for using the material as cracker feedstock is mentioned on page 105 only (left-hand column, last paragraph) and specifically with respect to the product obtained by the Synthol process. No particular type of catalyst is referred to in this regard.

The Respondents argued that those skilled in the art knew that the higher a feedstock was saturated the more it was suitable as a cracker feedstock. Therefore, a skilled person would understand from the above-mentioned paragraph in A9 that hydrogenation is essential in any case, irrespective of the particular FT synthesis conditions.

This argument is not convincing since it is apparent from page 108 of A9 (right-hand column, lines 13 to

21), that the olefin content in the FT product, in particular in the naphtha fraction (see also Table 2), and correspondingly, the need for hydrogenation, is dependent from the particular process conditions. It has been found that the conditions of the Synthol process, inter alia the high reaction temperature, are responsible for the high content of olefins in the products as compared with products of the ARGE process. In addition, A9 indicates that - for the purpose of producing lower olefins - naphtha production via FT synthesis only appeared to be economic under special conditions (page 105, sentence bridging the columns). Therefore, given the economic aspects of A9, there is no clear disclosure that the more saturated ARGE products should be hydrogenated.

A similar reasoning applies to the question whether A9 discloses hydrogenation of a product obtained by a cobalt-catalysed FT synthesis since A9 indicates that - unlike iron catalysts - the using of cobalt catalysts gives mainly saturated products (page 108, right-hand column, first full paragraph).

The Board, therefore, concludes that A9 does not contain any clear and unambiguous teaching of an additional hydrogenation of the FT product obtained at low reaction temperature, e.g. in the ARGE process, let alone when cobalt is used as the catalyst. It follows that the process of Claim 1 is not anticipated by A9 and, consequently, is deemed to be novel.

4. *Inventive step*

4.1 The patent in suit relates to a process for preparing lower olefins, i.e. olefins having from 2 to 4 carbon atoms, from a hydrocarbon feed by thermal cracking. It is said that such a process is known in the art, for example from D6, which discloses that a fraction of a FT synthesis process may be used as the hydrocarbon feed in the thermal cracking process (column 1, lines 3 to 15 and 33 to 52).

According to the patent in suit, the problem to be solved in view of this prior art is to improve the selectivity of the thermal cracking process (column 1, lines 30 to 32 and lines 53 to 55).

4.2 In the Respondents' opinion either D8 or A9 represented the most promising starting point for investigating inventive step since they both related to the same technical problem as the patent in suit, namely the production of lower olefins from a synthetic hydrocarbon feedstock. However, the Board shares the opinion of the Appellant that D6 is also concerned with that problem. Whilst realizing that D6 primarily refers to the preparation of linear C₁₀ to C₂₀ olefins (page 1, lines 1 to 3, page 2, lines 34 to 35), one of its objects is, nevertheless, to produce a mixture of lower olefins by subjecting the lighter C₉-fractions to a steam cracking. The purpose of this measurement is to oligomerize the obtained lower olefins into C₁₀ to C₂₀ olefins to improve their yield (page 5, lines 4 to 32).

4.3 The Board, therefore agrees with the Appellant that D6 qualifies as a suitable starting point for the assessment of inventive step.

4.4 In order to produce the lower olefins, D6 suggests to carry out a cobalt-catalyzed FT synthesis at elevated temperature (220°C or 204°C) and elevated pressure and to subject the gasoline fraction, i.e. the naphtha fraction, obtained to a thermal cracking step. The product obtained contains between 54 and 62% wt of lower olefins having from 2 to 4 carbon atoms (page 9, lines 14 to 22, page 10, lines 6 to 11 and Tables I and II).

This process differs from the claimed one only in that no hydrogenation is carried out before the cracking step.

4.5 The Appellant argued that the technical problem in view of D6 was to improve the yield in lower olefins.

It was evident from Experiment 7 of D6 when compared with example 3 of the patent in suit that this problem has been solved since this experiment corresponded to comparative example B in the patent in suit which was also run in the absence of any hydrogenation to produce the same yield (63.2% wt) of lower olefins in the same composition upon thermal cracking of the C₅₋₉ fraction obtained by the FT synthesis. Example 3 of the patent in suit showed, therefore, that the C₂₋₄ olefins yield, which amounted to 71.3% wt, was increased over comparative example 3 of the patent in suit as well as over Experiment 7 in D6 if the C₅₋₉ fraction was hydrogenated before subjecting it to thermal cracking. It was, therefore, apparent that in view of D6 the above technical problem was actually solved by the claimed subject-matter, i.e. by the distinguishing

feature of hydrogenation of the synthetic oil fraction before cracking.

Experiment 7 of D6 has been made with the C₅₋₉ fraction of the FT synthesis product according to Experiment 4 of this citation and example 3 as well as comparative example B of the patent in suit has been made with the C₅₋₉ fraction of the FT product obtained in example 1. However, the process parameters used in Experiment 4 differ from those used in example 1 of the patent in suit, in particular with respect to the pressure (20 instead of 36 bar), the H₂/CO ratio (2.0 instead of 1.1) and the space velocity (500 instead of 1125 Nl.l⁻¹.h⁻¹). Therefore, neither are example 1 of the patent in suit and Experiment 4 of D6 comparable nor the products obtained therein.

- 4.6 The Appellant alleged that the differences in the process parameters were irrelevant for the product obtained since the composition of the naphtha was always the same.

This allegation fails for the simple reason that - as eventually conceded by the Appellant - the process parameters influence at least the olefin content of the naphtha as well as its yield. This is corroborated by Experiments 4 and 5 in D6 where different process parameters give products with different olefin content and different yields in naphtha.

- 4.7 Therefore, the Board does not accept the Appellant's arguments that there was evidence showing an improvement of the yield of lower olefins in view of D6. In the absence of such evidence, the technical problem

credibly solved by the claimed subject-matter, therefore, boils down to the provision of a further process for the manufacture of lower olefins.

- 4.8 It remains to be decided whether, in view of the available prior art documents, it was obvious for someone skilled in the art to solve this technical problem by the means claimed, namely by subjecting in the process of D6 the naphtha fraction obtained from the FT product to hydrogenation before the thermal cracking.
- 4.9 In the Appellant's opinion, there was no guidance in the art to perform hydrogenation in the case where the gasoline was obtained by low temperature FT synthesis since it was known from D6 that in this case the olefin content in the fraction used for thermal cracking was very low, only 8% mole at most, and very high yields of lower olefins were obtained without hydrogenation. In addition it was known from A9 and D8 that hydrogenation was only essential if the gasoline was produced by the high temperature Synthol process and in cases where the olefin content was more than 10 to 15%.

These arguments are not convincing since both A9 and D8 do not consider hydrogenation prior to any cracking step disadvantageous from a technical point of view. Only under the economic aspects of the overall process for the manufacture of C₂-C₄ olefins, respectively of ethylene and propylene by FT synthesis, a hydrogenation was said to be unfavourable (A9, page 105, paragraph bridging the columns; D8, page 145, right-hand column, lines 3 to 26). In contrast, process economy is not an objective of the patent in suit and the claimed process

is not limited with respect to the degree of saturation in the gasoline fraction produced by the FT synthesis nor is any such degree even disclosed in the patent in suit. The fact that the product of Experiment 4 of D6 contains relatively little amounts of olefins neither proves that gasoline from low temperature FT synthesis generally contains low amounts of olefins, nor prevents those skilled in the art from applying their general technical knowledge in those cases where economy of a hydrogenation step is not an issue.

A13b is representative for such general knowledge relevant in the field of production of ethylene and other lower olefins from naphtha by thermal cracking (page 47, right-hand column, first full paragraph and page 48, Table 2). According to this knowledge, the preferred feedstock for producing high yields of ethylene and, depending on the chain length, propene by thermal cracking are alkanes, i.e. saturated hydrocarbons. It is emphasised that one of the conditions required to maximize the yields of olefins, in particular the ethylene production, is a highly saturated feedstock (see A13b, page 51, right-hand column, second full paragraph to page 52, left-hand column, second full paragraph). This is confirmed by the statement in D8 that the high content of olefins of the Synthol gasoline fraction reduces its versatility as a cracking feedstock unless part of its unsaturation is removed by hydrogenation (page 145, sentence bridging the columns).

- 4.10 The Board, therefore, concludes that for the purpose of providing an alternative to the process disclosed in D6, a skilled person would, with a reasonable expectation

of success, have applied the principles disclosed in A13b for also obtaining high yields of lower olefins by hydrogenating the naphtha before thermal cracking.

The Board notes, for the sake of completeness, that the patent in suit contains no indication that any possible problems with the economy of the hydrogenation step were overcome by the claimed process, but were simply accepted.

Consequently the Appellant's main request must fail since the subject-matter of Claim 1 is not based on an inventive step (Articles 52(1) and 56 EPC).

First Auxiliary Request

5. The above reasoning also applies to Claim 1 of the first auxiliary request since the olefins produced by thermal cracking of gasoline according to D6, D8, A9 and A13b are also "olefins having from 2 to 4 carbon atoms" and, therefore the same as in the prior art (D6, Table II, D3, Table III, A9, page 105, left-hand column, last paragraph, and A13b, Table 2 and page 51, right-hand column, first and second full paragraph).

Second Auxiliary Request

6. According to the Appellant, the feature "and in which hydrogenation process any unsaturated hydrocarbons and oxygenates present in the synthetic oil are hydrogenated" introduced into Claim 1 of the second auxiliary request was not obvious in view of the prior art since more severe conditions were required for hydrogenation of oxygenates than for hydrogenation of

olefins. According to D8, it was not even necessary to hydrogenate under conditions which transform all the olefins into saturated hydrocarbons.

The Board concedes that hydrogenation of oxygenates is not mentioned in the cited prior art. However, no unexpected advantages of this feature in view of the process of D6 have been shown. Therefore, the Board agrees with the Respondents that - in the light of the common general knowledge as represented by A13b and if economy of the process is not at stake - no inventive merit can be attributed to the performance of hydrogenation under conditions which result in the highest yield of alkanes in order to provide an alternative to the process of D6.

Since the second auxiliary request fails, therefore, also under Article 56 EPC, there is no need to consider the Respondents' objection under Article 123(2) EPC.

Third and Fourth Auxiliary Requests

7. The application as filed is based on the finding that the selectivity of the thermal cracking process towards lower olefins can be significantly increased when use is made of a synthetic oil fraction - such as a FT product - as a hydrocarbon feed in the thermal cracking process, which synthetic oil fraction has been hydroprocessed (page 2, lines 12 to 17).

This finding is demonstrated in the examples, where it is shown that high yields of lower olefins can be achieved by applying the particular cracking conditions of Example 3 to the C₅₊ product obtained under the

particular hydrogenation conditions described in Example 2.

However, Claims 1 of the third and fourth auxiliary request have been modified by introducing only the particular cracking conditions disclosed in Example 3 of the patent in suit, independent of the cracker feed. This implies that the particular cracking conditions of Example 3 should be favourable and valid for any cracker feed, a concept for which the application as filed does not provide a basis.

The Board, therefore, concludes that the amendments made to the claims do not meet the requirements of Article 123(2) EPC.

8. Therefore, none of the Appellant's requests meets the requirements of the EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P. Krasa