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DECISION
of 26 June 2002

Case Number: T 0112/00 - 3.4.2
Application Number: 87870199.4
Publication Number: 0273026
IPC: G03F 7/004, G03F 7/022
Language of the proceedings: EN
Title of invention: Solvents for photoresist compositions
Patentee: SHIPLEY COMPANY INC.
Opponent: Clariant GmbH
Japan Synthetic Rubber Co. Ltd.
Headword:

Relevant legal provisions:
EPC Art. 54, 56
EPC R. 64(b)

Keyword:
"Main request, first and second auxiliary request: Novelty - yes, inventive step - no"
"Third auxiliary request: Not covered by the appeal"
"Request of the appellant/opponent to amend the description: rejected"

Decisions cited:
T 0990/96, T 0728/98, T 0786/00, T 0279/89, T 0666/89

EPA Form 3030 10.93
Headnote:
A composition including a solvent having a purity of greater than 99% is new over a prior art composition with such a solvent the purity of which is not specified.
Case Number: T 0112/00 - 3.4.2

DECISION
of the Technical Board of Appeal 3.4.2
of 26 June 2002

Appellant: Clariant GmbH
(Opponent I)
Am Unisys-Park 1
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Representative: -

Appellant: Japan Synthetic Rubber Co. Ltd.
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Composition of the Board:
Chairman: E. Turrini
Members: M. P. Stock
V. Di Cerbo
Summary of Facts and Submissions

I. The appellant (opponent II) has lodged an appeal against the interlocutory decision of the opposition division ruling that account being taken of the amendments made by the patent proprietor during the opposition proceedings, the European patent No. 0 273 026 (application No. 87 870 199.4 claiming priorities of 23 December 1986 and 13 October 1987) can be maintained.

Oppositions had been filed by opponent I and opponent II. However, while opponent I attacked the patent as a whole, opponent II attacked claims 6 to 11 only. Both oppositions were based on Article 100(a) together with Articles 52(1), 54(1) and 56 EPC. Grounds for opposition under Article 100(b) EPC were mentioned by opponent I after expiry of the opposition period.

In its decision the opposition division made reference to documents D1 to D31.

In the following, the board will use the same document numbering as in the proceedings to date and reference will be made to the following documents:

D1: EP-A-0 211 667
D2: EP-A-0 239 423
D12: "LSI Process Engineering", Ohm Sha, 25 October 1982 and partial English translation thereof (D12a); to D12a a declaration of Dr. P. Trefonas
dated 16 June 1994 was attached

D14: Product Examination Certificate for ELK-413 dated 2 October 1985 including English translation

D17: Affidavit of Dr. P. Trefonas dated 20 January 1998

D20: Declaration of Yoji Ishino dated 15 July 1999

D21: Declaration of Toshiyuki Ota dated 16 July 1999

D31: Solid State Technology, vol. 25, no. 9, September 1982, pages 82 to 86

II. After requesting with the notice of appeal, setting aside the decision of the opposition division and revocation of the patent as a whole, the appellant requested with the statement of grounds revocation of the patent in the range of valid claims 4 to 9. The arguments of the appellant set out in the above quoted statement of grounds can be summarised as follows:

D1 discloses a radiation-sensitive resin composition according to claim 4, wherein ethyl lactate, which is identical to ethyl 2-oxypropionate, is used as a sole solvent. D1 teaches that precipitation of fine particles during storage of the resin composition must be avoided. It is general knowledge of a person skilled in the art that chemical impurities may cause precipitation during long-term storage. This general knowledge is also reflected by D31 indicating that quality control in VLSI fabrication is performed from the chemical purity aspect. D14, D20 and D21 prove that ethyl lactate distilled to a purity of greater than 99% was on the market before the priority date of the
contested patent. With this knowledge in mind, the skilled person would choose for ethyl lactate disclosed in D1, only a well-known sample having a purity greater than 99%. Therefore, for the skilled reader, this feature is disclosed in D1 thus taking away the novelty of the subject-matter of claim 4.

If claim 4 were found to be novel, its subject-matter would lack an inventive step. The only hint to particle formation is given in Example 31 of the contested patent according to which a solvent mixture comprising an essentially pure ethyl lactate is used. However, it is not shown in the contested patent that the purity (greater than 99%) of ethyl lactate as the sole solvent is the feature which provides superior effects of the composition disclosed in D1. Therefore, this feature cannot be taken into account when assessing inventive step.

According to the appealed decision, a skilled person would not derive from D1 that it is the chemical purity which is important. However, whereas D1 and D2 address the number of fine particles in solvents, it is clearly mentioned in D31 that it is the chemical purity which has to be taken into account. Distillation not mentioned in D31 is considered by the skilled person, when a liquid is to be purified. Hence, the skilled person is clearly directed to an ethyl lactate sample which has the highest possible purity on the market, as the sample ELK-413 having a purity of 99.7% (see D20 and D14), and would not use any less pure solvent, as was alleged in D17.

D21 shows that the replacement of a photoresist solvent mixture including ethyl lactate in a proportion of
98 wt% by a solvent consisting of ethyl lactate as the sole solvent of 99.7 wt% does not give any improvements or effects with respect to the number of fine particles formed in the final photoresist composition. The storage conditions of one month at 40°C used in D21 are even more severe than storage for a longer time under refrigerated conditions used in actual manufacturing.

It is expected that a non-obvious invention solves an objective problem which is intimately connected with technical consequences generated by the subject-matter of the invention. However, the problem of particle formation has already been solved by D1, by means of an ethyl lactate solvent having a purity which the skilled person would obviously adjust to greater than 99%. The further properties of the disclosed photoresist composition such as photospeed, coating uniformity, pattern resolution etc. are equivalent to those shown in D1.

III. The respondent (patent proprietor) has contradicted the appellant. His arguments are summarised as follows:

In their decisions, the EPO opposition division, the USPTO and JPO found that D1 or cases corresponding thereto provided no suggestion of a purity level for ethyl lactate of greater than 99%.

Assuming that ethyl 2-oxypropionate means ethyl lactate, D1 in any case teaches that a mixture of solvents has to be used. Since D1 considers that the problem of particle formation can effectively be solved by a huge number of solvent mixtures, the skilled person would not derive from D1 that just one solvent, namely ethyl lactate, has to be used in a specific
purity, namely greater than 99%. There was no basic knowledge of the skilled person, teaching that such a high purity had to be selected. D31 is concerned with processing chemicals and contaminants present in liquids, however, does not mention any solvents, let alone ethyl lactate, or a purity of 99%.

Document D20 related to D14, in itself is hearsay and no evidence that ethyl lactate of any particular purity was publicly available.

For a number of reasons D21 reporting certain isolated tests can be disregarded. The storage conditions of 40°C used in D21 are not relevant to commercial use, where photoresists are refrigerated. The particle detection apparatus used provides variable results, particularly depending on resist film thickness. The purity of ethyl lactate that was mixed with water or diethyl succinate is not specified in D21. Such mixtures do not correspond to ethyl lactate commercially available at the priority date.

It is quite surprising, as demonstrated by the comparative Example 31 of the contested patent, that ethyl lactate at a purity of greater than 99% would have to be used to avoid particle formation.

IV. Oral proceedings before the board took place on 26.06.02 in presence of the appellant and the respondent. Opponent I as a party to the appeal proceedings as of right pursuant to Article 107 EPC did not attend the oral proceedings.

In the oral proceedings the parties reiterated their arguments and put forward the following requests.
The respondent requested the appeal be dismissed and the patent be maintained in amended form on the basis of claims 1 to 9 according to a main request filed at the oral proceedings before the opposition division, of claims 1 to 12 according to a first auxiliary request filed during the oral proceedings before the board, of claims 1 to 9 according to a second auxiliary request filed as an auxiliary request during the oral proceedings before the opposition division and of claims 1 to 8 according to a third auxiliary request filed during the oral proceedings before the board.

Claim 1 according to any of the main, first, second and third auxiliary requests reads as follows:

"1. A composition responsive to activating radiation to form a latent image developable with an aqueous alkali solution comprising at least one alkali soluble resin and at least one photoactive compound that is an ester or polyester derived from the reaction of an oxodiazonaphthalene sulfonyl or carboxylic acid halide with a hydroxy or polyhydroxy ballast compound dissolved in a solvent mixture comprising anisole, ethyl lactate and amyl acetate."

Claim 4 according to the main request reads as follows

"4. A composition responsive to activating radiation to form a latent image developable with an aqueous alkali solution comprising at least one alkali soluble resin and at least one photoactive compound that is an ester or polyester derived from the reaction of an oxodiazonaphthalene sulfonyl or carboxylic acid halide with a hydroxy or polyhydroxy ballast compound dissolved in a solvent consisting of ethyl lactate
distilled to a purity of greater than 99 percent."

Claim 9 according to the first auxiliary request reads as follows:

"9. Use of a photoresist composition for coating a solid support, said composition being responsive to activating radiation to form a latent image developable with an aqueous alkali solution and comprising at least one alkali soluble resin and at least one photoactive compound that is an ester or polyester derived from the reaction of an oxodiazonaphthalene sulfonyl or carboxylic acid halide with a hydroxy or polyhydroxy ballast compound dissolved in a solvent consisting of ethyl lactate, characterized in that said ethyl lactate is distilled to a purity of greater than 99 percent."

Claim 9 according to the second auxiliary request reads as follows:

"9. Use of a photoresist composition in the manufacture of integrated electronic circuits, the photoresist composition being responsive to activating radiation to form a latent image developable with an aqueous alkali solution and comprising at least one alkali soluble resin and at least one photoactive compound that is an ester or polyester derived from the reaction of an oxodiazonaphthalene sulfonyl or carboxylic acid halide with a hydroxy or polyhydroxy ballast compound dissolved in ethyl lactate, wherein the ethyl lactate is distilled to a purity of greater than 99 percent."

The appellant requested the patent be revoked in so far as it is based on the main, first and second auxiliary request.
Moreover, the appellant requested that the description filed by the respondent at the oral proceedings together with the third auxiliary request be amended as follows:

Page 2, first sentence: The word "mixture" should be added to "solvent" which is the last word of the sentence.

The respondent maintained that since no grounds of appeal have been filed against the independent claims of the third auxiliary request, the appellant has no right to request an amendment of the description related thereto. Moreover the amendment would not be justified.

At the end of the oral proceedings the decision of the board was given.

**Reasons for the Decision**

1. **Admissibility of the appeal**

   The appeal complies with the provisions of Articles 106 to 108 and Rule 64 EPC and is therefore admissible.

2. **Novelty (Main Request)**

2.1 Since the subject-matter of claim 4 according to the main request only enjoys the second one of the two priorities claimed, D1 is prior art under Article 54(2) in connection with Article 89 EPC.

2.2 It was no more contested that ethyl 2-oxypropionate
disclosed in D1 designates the same compound as ethyl lactate disclosed in the contested patent. A solvent consisting of ethyl lactate is used in the Examples 1, 7 to 13, 14, 15, and 21 to 25 of D1 (see pages 20, 25, 28, 31, and 35, respectively).

2.3 Therefore it follows that D1 explicitly discloses all features indicated in claim 4 except the feature that ethyl lactate has been distilled to a purity of greater than 99 percent. Since claim 4 is directed to a composition, which is a product, the feature in question is a product-by-process feature. According to common interpretation such a feature does not limit the product to the particular process. Its meaning in the present case would be that the purity is greater than 99%, as obtainable by distillation.

2.4 If the skilled person follows the teaching of D1 for the preparation of a photoresist, she or he would have to decide on the purity of ethyl lactate used, since D1 does not give any indication on the purity which is necessary. This decision would be based on general knowledge of the skilled person. Therefore it is decisive for the question of novelty whether it was general knowledge of the skilled person in any case to choose ethyl lactate having a purity greater than 99%. However, there is no indication in the cited prior art that it was obligatory to make this choice in order to obtain a composition which has superior effects as a photoresist.

2.5 In this connection the appellant has argued as follows:

D20 in combination with D14 proves that ethyl lactate having a purity greater than 99% had been available on
the market for years before the priority date of the patent in suit. Therefore in accordance with the decision of the boards of appeal T 990/96, it would have to be concluded that D1 discloses a photoresist composition including ethyl lactate in any degree of purity, since the exceptional situation mentioned in this decision, that a particular degree of purity level could not by achieved by conventional purification processes, was not present. Moreover it is relevant whether ethyl lactate having a purity of greater than 99% provides any new effect. Only then a selection of the purity level from an unspecified range would be new. However, all examples presented in the patent are related to compositions employing mixtures of solvents and not to ethyl lactate as the sole solvent. Therefore a new effect has not been shown in the patent in suit.

2.6 These arguments are not accepted by the board.

2.6.1 The fact that ethyl lactate having a purity greater than 99% was available on the market is irrelevant since the decision T 990/96 (OJ 1998, 489) is related to a chemical compound and not to a composition of compounds. The same is true for T 728/98 (OJ 2001, 319) related to the situation where the degree of purity is defined in terms of a process of preparation, ie by product-by-process feature, like in claim 4 of the contested patent, however, again for a chemical compound and not for a composition.

2.6.2 More applicable to the present situation is T 786/00 (unpublished) related to the manufacture of a final product (polymer) by the use of starting materials (organic compounds) having a required purity. It is stated in T 786/00, see point 3.8.2 of the Reasons,
last sentence, that "the general statement in T 990/96 concerning the purity of final products cannot be applied directly to starting materials". For the present case the claimed composition could be considered as a final product and the solvent ethyl lactate would be a starting material. Therefore like in T 786/00 novelty would be established by the defined purity of the starting material, i.e. the solvent ethyl lactate.

2.6.3 As to the argument that "purity of greater than 99%" cannot be considered a new selection due to lack of a new effect, the board is of the opinion that novelty based on the definition of a value or range of a parameter which was never mentioned in the closest prior art cannot be assessed in terms of the classical selection invention. Therefore it does not make sense to verify whether the criteria for a new selection outlined in T 279/89 (unpublished, cited in Case Law, 4th edition, page 80) are met, since "purity of greater than 99%" cannot be considered as a sub-range of a broader range because the parameter "purity" is not mentioned in D1.

2.6.4 This view is also confirmed by the above-cited decision T 786/00. In point 3.8.3 of the Reasons the question of novelty of a specified minimum purity over a prior art document not disclosing a specific purity range is discussed under the aspect of overlapping ranges. It is stated that in such a case there are no overlapping ranges and, thus, the concept referred to in T 666/89 (OJ 1993, 495), of whether the skilled person would seriously contemplate applying the teaching of the prior art document in the range of overlap, is not relevant.
2.7 Therefore it is concluded that the subject-matter of claim 4 according to the main request is new (Article 54(1) and (2) EPC).

3. **Inventive step (main request)**

3.1 As was evaluated under point 2.3 above, the photoresist composition defined in claim 4 differs from what is disclosed in D1, which is the closest prior art, in that ethyl lactate has been distilled to a purity of greater than 99%. This means, as was also discussed above, that the purity of ethyl lactate is greater than 99%, as obtainable by distillation.

3.2 The objective problem solved by using ethyl lactate with such a relatively high purity addresses reduced particle formation by applying the composition as a photoresist to a surface, see the patent in suit, page 17, lines 45 to 47.

3.3 However, the problem with contamination of processing chemicals in the manufacture of VLSI integrated circuits is generally known and is discussed in D31, see the abstract. It is indicated in D31, see page 83, second paragraph, that the contaminants present in liquids can be classified in dissolved impurities and in particulates. Several methods for the detection of particulates are discussed under "Particulates" at pages 83 and 84. Photoresists, mentioned in the last paragraph of page 83, can contain transparent particles which are difficult to detect by an automated method based on light-blocking. For such transparent particles, a membrane filter technique for retaining the particles and scanning electron microscopy to identify their nature and contamination level is proposed. Hence, it
was known to the person skilled in the art that particulates in photoresist compositions were a major source for lower yield in the manufacture of VLSI devices, see "Results and Discussion" at page 85. Since the skilled person had means to detect contaminating particles in photoresists, she or he would always carry out appropriate tests and, if necessary, improve the purity of the starting materials for the photoresist composition including that of the solvent ethyl lactate the purity of which could be increased by distillation. If a higher purity of ethyl lactate, namely greater than 99%, were able to reduce particle precipitation in a photoresist, the skilled person would have found out this fact and obviously used such a relatively high pure ethyl lactate.

3.4 It is to be noted in this connection that in the description of the patent in suit, Example 31 at page 17 (corresponding to Example 195 in the application as originally filed) is the only location where the purity level of ethyl lactate is discussed. According to this example, "two resist compositions were prepared as described in Example 30A". However, Example 30A (see page 16) discloses a solvent mixture, which does not fall under claim 4 defining ethyl lactate as the sole solvent. Hence, there exists no real embodiment for the composition of claim 4.

3.5 In contrast to that in document D21 there are presented results from a comparison regarding the formation of particles between resists using 99.7% pure ethyl lactate and resists wherein ethyl lactate has a purity of 98%, the balance being water or diethyl succinate (see point 6). From this it follows that the 98% sample is obtained by mixing ethyl lactate of high purity
(greater than 99.7%) with water or diethyl succinate (DeSu) in order to provide a weight ratio of 98:2 (see the table in D21, 5th column). No significant difference is seen in the number of fine particles between runs employing ethyl lactate only (EL: 99.7%) on one side and corresponding runs employing diluted, i.e. less pure ethyl lactate (EL/DeSu = 98/2 and EL/water = 98/2 weight ratio) on the other side.

3.6 Therefore, it is not evident from the description of the contested patent that ethyl lactate having a purity of greater than 99% has an effect on particle precipitation. Since such an effect is not proven, it cannot justify an inventive step in the claimed subject-matter and would have to be considered as an arbitrary selection of features which does not solve a particular problem.

4. First and second auxiliary requests

4.1 The versions of claim 9 according to the first and second auxiliary requests are directed to the use of a photoresist composition as defined in claim 4 of the main request, for coating a solid support and in the manufacture of integrated electronic circuits, respectively.

4.2 Since it was obvious for the skilled person starting from the closest prior art according to D1 to obtain such a composition, it was also obvious to use such a composition as a photoresist composition for coating solid supports, in particular in the manufacture of integrated circuits, as is also disclosed in D1, see page 1, first paragraph to page 2, first paragraph.
5. **Arguments of the respondent**

5.1 The Respondent has argued as follows:

The invention is based on the recognition that purity of the solvent ethyl lactate is critical. This issue was not recognised in D1, otherwise it would have been discussed. According to D31 purity is an important issue. However, patent applications D1, D2 and D3 filed after the publication of D31 do not discuss the purity of solvents used in the photoresist compositions described in these applications. Furthermore, D31, see page 83, third paragraph, concentrates on metallic impurities which are a different type of purity than addressed in the patent in suit. Whereas in Table I of D31 metallic impurities in the ppb (parts per billion) range are listed, the teaching according to the patent in suit is concerned with impurities in the percent range. For this type of impurity D12 is more relevant, discussing cleaning of chemicals in LSI process engineering. Again the amount of metal impurities is given in ppb for electronic grade chemicals, which are used in LSI manufacturing. According to data sheets attached to D17, electronic grade and reagent grade ethyl lactate which were commercially available had a lower limit (98%) of a purity level lying below the limit (99%) claimed in the patent in suit. There was no reason why the skilled person would choose ethyl lactate having a higher purity, which would be more expensive, if no effect could be expected.

5.2 These arguments cannot be accepted by the board.

The problem of particle precipitation in photoresists was known from D1 and D31. From D31 the skilled person
would derive that not only particulates are to be observed but any contaminants must be controlled including dissolved contaminants. This evidently includes solvents which are diluted in an undefined manner. Moreover D31 teaches how to measure particle precipitation in photoresists. If particles were detected, the skilled person would change the preparation parameters in the sense of providing more reproducible conditions. Such a working procedure is also mentioned in the declaration attached to D12a, see page 3, penultimate paragraph. The use of a solvent having a higher purity would be a rather simple measure. D20 in connection with D14 seem to show that highly pure ethyl lactate was commercially available before the priority date of the contested patent. Apart from that, distillation of less pure, e.g. electronic grade, ethyl lactate (see D17) would be a straightforward method for purification. Costs would be of secondary importance at least at an experimental stage.

5.3 According to a further argument of the respondent the effect obtained with higher purity of ethyl lactate is disclosed in the patent in suit. The comparison made between a resist compositions employing commercially available ethyl lactate (purity 97%) and freshly distilled ethyl lactate (purity greater than 99%) according to Example 31 (original Example 195) is related to a solvent mixture containing ethyl lactate with anisole and amyl acetate according to Example 30A. The effect is obtained with this mixture comprising ethyl lactate having a purity of greater than 99%. Therefore an even better effect could be expected if this relatively high pure ethyl lactate is used as the sole solvent. In D21 not the same conditions as in the patent in suit are used for a comparison of resists
formed with ethyl lactate of different purity. The author did not look for visible particles but only for invisible particles. Moreover, the preparation of the ethyl lactate solvent samples was such that in fact two embodiments of the invention according to the patent in suit were compared to one another, namely a solvent mixture comprising ethyl lactate of a purity greater than 99% was compared with a solvent consisting of this highly pure ethyl lactate only.

5.4 This argument is also not convincing.

Example 31 shows reduced particle formation only for a mixture of solvents. It could well be that the impurities included in ethyl lactate react with the co-solvents anisole or amyl acetate to cause particle precipitation, whereas they have no effect on ethyl lactate. As far as the experiments presented in D21 are concerned, it is mentioned in the appellant's letter dated 28 March 2000, see page 12/13, first paragraph, that "at the relevant date of the opposed patent, it was well accepted by those skilled in the art that possible impurities of ethyl lactate as a photoresist solvent were water and diethyl succinate". Taking this as a fact which was not contradicted by the respondent, ethyl lactate having a purity of 99.7% diluted by water or ethyl succinate in a ratio of 98:2 could be considered as a sample of ethyl lactate having a purity of less than 99% and not as mixture of solvents. As to the argument that the author of D21 did not look for visible particles, it is indicated in D21, see page 2, first paragraph: "No visible particles were observed for the compositions of Runs No. 1 to 6 at times both after preparation and after storage at 40°C for 1 month". This means that it was looked for visible
particles, but none were detected. Therefore the comparison made according to D21 is relevant to the question whether any effect is obtained by using highly pure ethyl lactate as the sole solvent.

5.5 The respondent summarised his arguments by stating that there was no reason why the skilled person would use highly pure ethyl lactate in a photoresist composition. Starting from D1, several steps were necessary to arrive at the invention. The skilled person would have to realise

(1) that there was need for improvement;

(2) that particle formation is correlated to ethyl lactate, and

(3) that the solution would be to employ a purity greater than 99%.

Therefore the invention would not be obvious.

5.6 However, the board is convinced that in view of the generally known problem with contamination, e.g. in the manufacture of integrated circuits, the skilled person would routinely inspect photoresist layers for defects and improve the preparation of photoresist compositions, if necessary. The board is further convinced that, if there were a problem related to ethyl lactate, the skilled person would have realised this and increased the purity of ethyl lactate. However, no evidence is available that the higher purity of ethyl lactate as the sole solvent would reduce particle formation in a photoresist layer.
6. Therefore taking into due account the essential arguments of the respondent it is concluded that the subject-matter of claim 4 according to the main request, of claim 9 according to the first auxiliary request and of claim 9 according to the second auxiliary request does not involve an inventive step in the meaning of Article 56 EPC. Accordingly, the patent cannot be maintained in amended form on the basis of any of these requests.

7. Third auxiliary request

7.1 The third auxiliary request is based on independent claim 1 as granted. Dependent claims and claims related to a use based on claims as granted have been appended to claim 1. The description has been adapted to this set of claims. Claim 1 was not within the extent to which cancellation of the appealed decision is requested, see Rule 64(b) EPC. In his final requests the appellant did not request the revocation of the patent based on the third auxiliary request. For these reasons the third auxiliary request is not subject of these appeal proceedings.

7.2 It follows further that the maintenance of those parts of the description which are related to claim 1 cannot be questioned. Therefore the first paragraph of the description (page 2, lines 3 to 5) which defines the technical field to which the invention relates, see Rule 27(1) EPC, may not be amended. There is also no
contradiction between "a solvent mixture" defined in claim 1 and "a solvent" recited in this paragraph, since in the context "a solvent" is a generic term including also solvent mixtures.

**Order**

*For these reasons it is decided that:*

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent on the basis of the third auxiliary request filed during the oral proceedings before the board of appeal, i.e. with the following documents:

   - claims 1 to 8

   - description pages 2 to 17

The Registrar: The Chairman:

P. Martorana E. Turrini