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D E C I S I O N
of 13 November 2002

Case Number: T 0204/00 - 3.4.2

Application Number: 90314083.8

Publication Number: 0438902

IPC: H01M 8/10, C25B 5/00, B01D 71/02, C25B 3/00, C25B 1/00

Language of the proceedings: EN

Title of invention:
Electrochemical reactors and multicomponent membranes useful for oxidation reactions

Patentee:
THE STANDARD OIL COMPANY

Opponent:
AIR PRODUCTS AND CHEMICALS, INC.

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56, 107
EPC R. 65(1)

Keyword:
"Admissibility of appeal by patentee (no, not adversely affected)"
"Novelty and inventive step (yes)"

Decisions cited:
G 0009/92, G 0004/93, T 0012/81, T 0666/89, T 0506/91, T 0528/93, T 0613/97, T 0793/93, T 0396/89

Catchword:
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Case Number: T 0204/00 - 3.4.2

D E C I S I O N
of the Technical Board of Appeal 3.4.2
of 13 November 2002

Appellant: (Opponent)
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Respondent: (Proprietor of the patent)
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Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office posted 10 December 1999 concerning maintenance of European patent No. 0 438 902 in amended form.**

Composition of the Board:

Chairman: E. Turrini
Members: A. G. Klein
V. Di Cerbo

Summary of Facts and Submissions

I. Both the patent proprietor and the opponent have appealed against the interlocutory decision of the opposition division finding European patent No. 0 438 902 (application No. 90 314 083.8) as amended by the patent proprietor during the opposition proceedings to meet the requirements of the EPC.

The opposition filed by the opponent against the patent as a whole was based, among others, on the grounds of lack of novelty and lack of inventive step (Article 100(a) EPC).

In the decision under appeal the opposition division held, *inter alia*, that the subject matter of the claims of the amended patent documents according to the single final request submitted by the patent proprietor during the oral proceedings was neither anticipated nor rendered obvious by the available prior art comprising, among others, the following documents:

D1: English translation of JP-A-63-156516; and

DX: "Properties of iron-doped lanthanum chromite", P P Zhuk *et al.*, English translation, published by Plenum Publishing Corporation (1988), of Izvestiya Akademii Nauk SSSR, Neorganicheskie Materialy, Vol. 24, No. 1, January 1988, USSR; pages 88 to 91.

II. In an annex to summons to oral proceedings, the board expressed its preliminary opinion that the patent proprietor would not appear to be adversely affected by the interlocutory decision of the opposition division

within the meaning of Article 107 EPC, first sentence and that for this reason the appeal filed by the patent proprietor would not appear to be admissible. In addition, with regard to the requests submitted by the patent proprietor with his statement of grounds of appeal, the board drew the attention of the parties to the principle of prohibition of *reformatio in peius* set out in the decisions of the Enlarged Board of Appeal G 9/92 and G 4/93, OJ, EPO 1994, 875 (point 16 of the Reasons).

III. During the oral proceedings held on 13 November 2002 the opponent withdrew an objection under Article 123(2) EPC previously raised in his statement of grounds of appeal and requested that the decision under appeal be set aside and the patent be revoked in its entirety.

The patent proprietor for his part withdrew previous requests submitted with his statement of grounds of appeal and requested that the patent be maintained in the form allowed by the opposition division.

At the end of the oral proceedings, the board gave its decision.

IV. Claim 1 of the amended patent on which the contested decision is based reads as follows:

" 1. A solid multi-component membrane for use in an electrochemical reactor characterised by a mixed metal oxide material having a perovskite structure represented by the formula:

$$A_s\ A^{I}_t\ B_u\ B^{I}_v\ B^{II}_w\ O_x$$

wherein A represents 1) a lanthanide or Y, or a combination of La and Y; A^I represents 2) Sr; B represents 3) Fe; B^I represents 4) Cr or Ti, or a combination of Cr and Ti and B^{II} represents Mn, Co, V, Ni or Cu, or a mixture thereof, and wherein; s, t, u, v, w and x each represent a number such that:

the ratio s/t equals from about 0.01 to about 100,

u equals from 0.01 to about 1,

v equals from 0.01 to about 1,

w equals zero to about 1,

x is a number that satisfies the valencies of the other elements present in the above formula; and

$0.9 < (s + t) / (u + v + w) < 1.1$ "

Claims 2 to 20 are directed to membranes, elements, electrochemical reactor cells and electrochemical and electrocatalytic processes, and all these claims are directly or indirectly referred back to claim 1.

V. The arguments put forward by the opponent in support of his requests can be summarized as follows:

Document D1 is directed to an oxygen permeation apparatus comprising a membrane consisting of an oxide of Sr, La, Co and Fe mixed with $SrTiO_3$. According to the example described on page 8 of the document, the two oxides are "well mixed" (page 8, line 6) and then sintered. The "Experimental Report" annexed to the statement of grounds of appeal shows the results of experiments carried out following the procedure of the example described in the document. According to these results, the X-ray diffraction pattern of the mixed oxide material presents two distinct peaks before sintering (Figure 1 of the report) and one single peak after sintering (Figure 2), thus implying that the

process according to document D1 results inherently in the formation of a predominant single phase oxide compound having the structure and the composition of claim 1. This conclusion is confirmed by the results of an analysis carried out using scanning electron microscopy and electron dispersive spectroscopy (Figures 3 to 6 of the "Experimental Report") and showing that the sintered product includes, in addition to grains of $SrTiO_3$ (Figure 6), large grains of an oxide of La, Sr, Ti, Co and Fe (Figures 4 and 5) resulting from the reaction of both starting oxides. Although document D1 mentions mixing with a mortar, the particular mixing conditions are not important as long as the oxides are well mixed in a powder scale. Therefore, the disclosure of document D1 inevitably results in a membrane including a mixed oxide material as defined in claim 1 and, following the decisions T 12/81 and T 666/89, the document is novelty destroying for claim 1. As to the tests submitted by the patent proprietor with his statement of grounds of appeal, the shift and the enlargement of the diffraction peaks after sintering shown in Figures 1a and 2a do confirm, rather than contradict, the submissions above since the shift and the enlargement of the peaks indicate that the two oxides have mixed together and that after sintering a new compound has been formed with a separate phase.

With regard to the issue of inventive step, starting with the oxygen-ion conductor oxide material disclosed in document D1 as the closest prior art, the oxide material of claim 1 solves the problem of improving the stability of the membrane. Since improving stability requires avoiding large grains with corresponding large boundaries and document D1 already stresses that the

starting oxides should be well mixed, it is obvious to mix the starting oxides in the form of finer grains, thus improving the homogeneity of the material. This procedure leads inherently to the material of claim 1 as evidenced by the results of the tests submitted with regard to the issue of novelty. In addition, the claimed solution also results from the combination of the disclosure of document D1 with the teaching of document DX.

Alternatively, document DX can also be considered as the closest prior art. This document discloses mixed oxides having high stability. The oxide materials are formed into rods not for commercial use, but only for experimental testing, and the document mentions in its first paragraph the use of the mixed oxides as oxygen-ion conductive solid electrolytes, i.e. as membranes for electrochemical reactions. The document discloses in particular mixed oxide materials of La, Ca, Cr and Fe and reports on the electrical conductivity of the materials as measured by the four-probe method, i.e. on the total conductivity encompassing both the electron and the oxygen-ion conductivity, whereby the increased electrical conductivity reported in the document leads to an increased oxygen-ion flux across the membrane. Therefore, the membrane of claim 1 differs from the disclosure of document DX only in the replacement of Ca by Sr. Document DX, however, already gives a hint towards the claimed subject matter since the first paragraph of the document refers to expanding the usefulness of the oxides by doping with alkaline-earth metals, of which the predominant and obvious alternative at the priority date of the opposed patent was Sr as evidenced by document D1 disclosing strontium-doped oxides. Furthermore, a comparison of

the conductivity data shown in Table 1 of document D1 and in Figure 2 of document DX clearly hints at the replacement of Ca by Sr in the oxide of document DX in order to increase the oxygen-ion conductivity. In addition, document D1 discloses strontium-doped oxides with sintering temperatures of about 1300°C (page 8, second paragraph) that are lower than the temperatures between 1600 and 1950°K required for sintering the calcium-doped oxides of document DX (document DX, page 88, fifth paragraph); the use of Sr as taught in document D1 instead of Ca as in document DX therefore results in lower sintering temperatures and thus in a clear manufacturing advantage.

VI. The patent proprietor's arguments are essentially the following:

Document D1 explicitly refers to a mixed sintered body with an intergranular composition in which the two starting metal oxides are present as two distinct oxide phases and there is enlargement of the particle boundaries for the purpose of improving the oxygen-ion conductivity (sentence in the middle of page 6). In addition, in carrying out the example 1 of document D1, the authors of the document clearly obtained two phases (page 8, second and third paragraph). Therefore, document D1 clearly teaches away from the membrane material defined in claim 1. In addition, it is *prima facie* clear that an experiment as that carried out by the opponent and leading to a different result is not a faithful reproduction of the example disclosed in document D1. According to the opponent's "Experimental Report", the starting oxides were "mixed together by vibratory milling the powders for 4 hours" (page 1 of the report), a significantly harsher mixing technique

than that used in document D1 involving mixing with a mortar, and this difference may account for the different results obtained by the opponent. In addition, the results of an analysis using x-ray diffraction (Figures 1 and 2) and scanning electron microscopy (Figures 3 and 4) of samples obtained by mixing with a mortar and submitted with the own statement of grounds of appeal show that the diffraction peaks of the sintered material are shifted with respect to, but are still close to the peaks of the starting oxides, thus confirming the results reported by the authors of D1 that the two oxide phases remain separate after sintering.

With regard to the issue of inventive step, document D1 teaches separation into two different phases corresponding to the two oxides (sentence in the middle of page 6) and is silent as to any improvement of the stability of the material. Therefore, document D1 does not address the problem solved by the claimed subject matter, i.e. improving the stability without prejudicing the oxygen-ion conductivity, and is far from suggesting the claimed oxide material. Document DX discloses mixed metal oxides containing Ca, not Sr, and in addition the document is not concerned with the oxygen-ion transport properties of the material, let alone with its use as a membrane. Document DX is rather directed to the effect of iron-doping on the structural, electrical and thermo-physical properties of the oxide material, and the oxygen ion conductivity is not addressed at all in the document. In addition, the improved electrical conductivity reported in document DX does not necessarily imply a higher oxygen-ion conductivity. For these reasons, document DX does not hint at the claimed oxide material for the purpose

of improving oxygen-ion conductivity, still less is there any suggestion to replace Ca by Sr.

Reasons for the Decision

1.1 *Admissibility of the appeal filed by the opponent*

The appeal filed by the opponent is admissible.

1.2 *Admissibility of the appeal filed by the patent proprietor*

The interlocutory decision under appeal maintaining the patent in amended form is based on the amended patent documents according to the final version of the main and only request submitted by the patent proprietor during the oral proceedings held before the opposition division (point 3 of the reasons of the decision). This is confirmed by the statement in point 5, first paragraph of the minutes of the oral proceedings that "all other requests were withdrawn" by the patent proprietor. In these circumstances, since the request submitted by the patent proprietor during the first-instance opposition proceedings and forming his sole, final request has been allowed in its entirety, the patent proprietor cannot be considered to be adversely affected by the decision under appeal within the meaning of Article 107 EPC, first sentence (see decisions T 506/91, T 528/93 and T 613/97 cited in "Case Law of the Boards of Appeal", 4th edition, 2001, chapter VII.D, section 7.3.2, first paragraph). Accordingly, the appeal filed by the patent proprietor is rejected as inadmissible pursuant to Rule 65(1) EPC.

2. *Procedural matters*

In view of the inadmissibility of the appeal filed by the patent proprietor, the opponent (in the following the "appellant") and the patent proprietor (in the following the "respondent") are to be considered respectively as the sole appealing party and as a party to the appeal proceedings as of right (Article 107 EPC, second sentence) for the purpose of applying the principle of prohibition of *reformatio in peius* set out in the decisions of the Enlarged Board of Appeal G 9/92 and G 4/93, OJ EPO 1994, 875 (point 16 of the reasons). Since during the oral proceedings held before the board the respondent withdrew the previous requests filed with his statement of grounds of appeal and requested the patent to be maintained in the form allowed by the opposition division, the appellant's request complies with the principle set out in the decisions mentioned above according to which the respondent is in the present case "primarily restricted during the appeal proceedings to defending the patent in the form in which it was maintained by the opposition division in its interlocutory decision" (G 9/92, *supra*, Headnote 2).

3. *Novelty of the subject matter of the claims*

3.1 Document D1 discloses an oxygen permeable membrane for use in a selective oxygen permeation apparatus. The membrane is made of a sintered material that is prepared from two starting oxide materials, namely a conductor material of a mixed metal oxide of Sr, La, Co and optionally at least a metal selected from Fe, Mn, Cr or V, and an intergranular deposition agent of an oxide of Sr and at least a metal selected from Ti, Zr

and Hf (page 5, first and second paragraphs), it being undisputed by the parties that the conductor material has a perovskite structure. In the example disclosed on page 8, the membrane material is obtained by first mixing $Sr_{0.65}La_{0.35}Co_{0.7}Fe_{0.3}O_{3-ä}$ as the conductor material and $SrTiO_3$ as the intergranular deposition agent, and then sintering the resulting mixture.

It has been undisputed by the parties that neither one of the conductor material or the intergranular composition agent disclosed in document D1 anticipates the oxide material defined in claim 1 of the patent as amended. In particular, although the family of oxide materials according to the claimed subject matter falls within the generic formula of the conductor material disclosed in document D1, the document does neither disclose nor exemplify the simultaneous selection of Fe and Cr in the generic formula of the conductor material. In addition, the composition of the conductor material specified in the example on page 8 does not comprise Cr or Ti as required by the claimed subject matter.

The appellant, however, has submitted that the mixing and sintering process described in the example of document D1 results in a reaction mixture of the two starting oxides and that the resulting sintered body inevitably comprises a mixed metal oxide material according to the subject matter of claim 1, thus anticipating the claimed subject matter in the sense of decisions T 12/81 (OJ 1982, 296) and T 666/89 (OJ 1993, 495). The appellant has relied in support of his submissions on the results of an alleged repetition of the example of document D1 conducted by the appellant himself and shown in the "Experimental Report".

Figure 2 of this report shows in particular that the diffraction peaks associated with each of the two oxide phases before sintering are replaced after sintering by new peaks representing a new single phase. According to the appellant's submissions, this result indicates that the two mixed starting oxides have reacted during sintering with each other to form a new oxide having the composition of the material defined in claim 1 as further confirmed by the results of the analysis carried out on the sintered material and shown in Figures 3 to 6 of the report.

However, the replacement of the diffraction peaks of the starting oxide materials by new peaks in the diffraction pattern of the sintered material according to the submissions and the evidence presented by the appellant not only is in contradiction with the experimental results reported by the authors of document D1 that "new diffraction lines due to the second added substance appear near the diffraction lines of the [conductor oxide]" in the X-ray diffraction of the resulting sintered material and that from the evaluation of the relative intensity of the peaks "it was confirmed that it separated into phases" (page 8, third paragraph), but runs also counter the essential feature taught in document D1 that the improved oxygen-ion conductivity of the resulting mixed sintered material results from the "separation into 2 phases" of the two oxide materials (page 6, lines 12 to 16, and page 6, line 22 to page 7, line 3). In addition, the replacement of diffraction peaks alleged by the appellant is also at variance with the results of the corresponding tests conducted by the respondent. According to these tests, the diffraction peaks of the starting oxide phases are slightly shifted after

sintering, but no discernible new diffraction peaks indicating the formation of a new distinct oxide phase become apparent in the diffraction pattern.

According to established case law of the boards of appeal, the standard of proof to be applied in establishing the inevitable outcome of a prior art disclosure for the purpose of assessing novelty should be much stricter than the balance of probabilities (see in this respect decisions T 793/93, not published in the OJ, point 2.1 of the reasons, and T 396/89, not published in the OJ, points 4.3 to 4.7 of the reasons). This is particularly so in the circumstances of the present case where the inevitable result alleged by the appellant is in contradiction with the explicit disclosure of document D1 and the results obtained by the respondent. In addition to that, the party submitting the allegation has not only the burden of reproducing the earlier disclosure in such a way as to demonstrate the alleged inevitable outcome, but also the burden of showing convincingly that if any significative deviation from the conditions specified in the earlier disclosure has occurred, this deviation is not material to the outcome (see T 396/89 *supra*, points 4.5 and 4.7 of the reasons). In the present case, however, the experimental tests conducted by the appellant did not involve mixing of the two starting oxides with a mortar as specified in the example of document D1 and as it was also the case in the tests conducted by the respondent, but involved mixing using vibratory milling during four hours (first page of the "Experimental Report"), a mixing procedure that departs from that specified in document D1 and that according to the respondent constitutes a significantly harsher mixing technique and might well lead to a different

result. Although the appellant did not dispute this deviation from the conditions disclosed in document D1, he denied any technical significance of this deviation provided that the oxides are well mixed in the sense of the example of document D1. Nonetheless, the appellant failed to submit arguments or evidence in support of such a contention so that it cannot be excluded that the deviation might have caused a significant difference in the structure of the sintered sample obtained by the appellant.

Accordingly, in view of the fact that the inevitable result alleged by the appellant relies on the results of tests that are not only in contradiction with the explicit teaching of document D1 and the results reported by the authors of the document, but are also at variance with the results of the tests conducted by the respondent and allegedly based on an exact repetition of the example disclosed in document D1, and since the appellant has failed to discharge the burden of showing convincingly that the deviation from the conditions specified in document D1 was not material to the result of the mixing and sintering process conducted by him, the board is not convinced that the mixing and sintering process disclosed in document D1 would inevitably result in a new oxide phase having the composition defined in claim 1.

According to an alternative line of argument advanced by the appellant, the shift and the enlargement of the diffraction peaks observed after sintering in the tests conducted by the respondent, rather than proving that no new oxide material is formed in the sintered body, would in fact corroborate the contention that the starting oxides react with each other during sintering

so as to form a new oxide. However, even though it cannot be excluded, and it would even appear plausible, that in the course of the sintering process **traces** of titanium may have diffused from the titanate into the conductor oxide, the respondent's tests are not conclusive as evidence that titanium would then have migrated into the conductor oxide to an extent sufficient to be present in the resulting conductor oxide of the sintered material in a stoichiometric amount between 0.01 and 1 as required by the subject matter of claim 1.

In the absence of clear and convincing evidence to the contrary, the board concludes that the subject matter of claim 1 is not anticipated by the disclosure of document D1.

3.2 Document DX reports on the properties of mixed oxides of La having a perovskite structure, and in particular on that of an iron-doped lanthanum chromite of the formula $La_{0.8}Ca_{0.2}Cr_{1-x}Fe_xO_3$ with x = 0 to 1 (first paragraph of section "Experimental"), x = 0,2 being recommended (page 90, third paragraph). Although the introductory paragraph of the document mentions doping lanthanum rare-earth oxides with alkaline-earth metals, which family of metals include among others Ca and Sr, the document does not mention Sr nor disclose, either explicitly or implicitly, Sr as one alternative example of doping agent. For this reason alone, the disclosure of document DX fails to anticipate the subject matter of claim 1.

In addition, during the oral proceedings the appellant did not dispute anymore the novelty of the subject matter of claim 1 over the disclosure of document DX.

3.3 Having regard to the above, the subject matter of claim 1 is considered to be novel (Articles 52(1) and 54 EPC) over the disclosure of documents D1 and DX.

The same conclusion applies to the subject matter of claims 2 to 20, all of which refer to the membrane defined in claim 1 or encompass an element of a mixed metal oxide material as defined in claim 1.

4. *Inventive step of the subject matter of the claims*

4.1 Closest prior art

The invention is primarily directed to the oxygen-ion conduction characteristics of oxygen-ion conductive membranes for use in electrochemical reactors (page 3, lines 5 to 10 together with page 8, lines 32 to 36 and page 11, lines 11 to 14). Since document D1 already concerns oxygen-ion conductive membranes and discusses the oxygen-ion conduction characteristics of the membranes and none of these aspects are addressed in document DX, at least not explicitly, the board considers document D1 to represent the most appropriate starting point for the assessment of inventive step according to the problem-solution approach.

4.2 Objective problem

The distinguishing feature of the subject matter of claim 1 over the membrane disclosed on page 8 of document D1 is the presence in the mixed metal oxide of the conductor material of Ti or Cr or a mixture thereof in a stoichiometric amount between 0.01 and 1.

According to the disclosure of the patent (page 8,

lines 32 to 36 together with page 11, lines 11 to 13) and the respondent's submissions, this feature has the advantage of stability under electrocatalytic conditions without sacrificing the oxygen-ion conductivity of the membrane.

Accordingly, the objective problem solved by the subject matter of claim 1 with regard to the membrane disclosed in document D1 may be seen in improving the stability of the membrane under electrocatalytic conditions without detriment to the oxygen-ion conduction characteristics of the membrane.

4.3 Inventive step

According to the main line of argument of the appellant, document D1 teaches explicitly that the starting oxide materials are "well mixed", and since improving stability requires avoiding large grains with corresponding large boundaries, it is obvious to mix the starting oxides in the form of homogeneous, finer grains, thus leading inherently to the material of claim 1 as supported by the evidence and the arguments submitted with regard to the issue of novelty. However, document D1 does not contain any indication or suggestion towards the improvement of the degree of stability of the membrane and is in particular silent as to any effect of the grain size and the grain boundary on the stability of the material. In addition, the document mentions the positive effect of the enlargement of the particle boundaries on the oxygen-ion conductivity of the material (page 6, lines 12 to 16) and thus teaches away from the procedure suggested by the appellant, i.e. teaches away from reducing the

particle size or reducing the boundary of the oxide particles by mixing the oxides in a fine size powder degree below that achievable with a mortar (page 8, lines 6 to 8).

The appellant has advanced a second line of argument according to which the combination of the disclosure of document D1 with the teaching of document DX would result in the claimed subject matter. However, although document DX discloses mixed oxides containing Cr and refers in its introductory paragraphs to the use of mixed rare-earth oxides doped with alkaline-earth metals as oxygen-ion conductive solid electrolytes and to the stability properties of mixed oxides with a perovskite structure, none of these indications render obvious the claimed subject matter. The appellant has in particular failed to identify any suggestion or indication in document DX that would have prompted the skilled person to solve the problem formulated above by adding Cr, or alternatively Ti, to the specific conductor material disclosed in document D1 so as to arrive at the claimed subject matter.

Therefore, the subject matter of claim 1 appears to involve an inventive step with regard to the disclosure of document D1 as the closest prior art.

4.4 For the sake of completeness, it is noted that the appellant's alternative approach relying on document DX as the closest prior art and on the use of Sr instead of Ca as the sole distinguishing feature of the subject matter of claim 1 over the disclosure of document DX would not affect the conclusion in point 4.3 above. Although the introductory paragraph of the document refers generally to doping rare-earth oxides with

alkaline-earth metals - which encompass, among others, Ca and Sr -, the disclosure of the document focuses exclusively on the properties of a rare-earth chromite containing Ca on account of its optimal conductive characteristics (page 88, fourth paragraph) and fails to provide any suggestion towards the replacement of Ca by Sr in order to achieve the improvements of the invention. In particular, the document discloses that doping with iron the rare-earth chromite increases the electrical conductivity of the material while preserving its stability (page 90, first paragraph), but only the electron conductivity component of the electrical conductivity is mentioned when discussing the mechanism underlying the improved electrical conduction characteristics (page 89, last paragraph). The further submission of the appellant that the lower sintering temperatures referred to in document D1 as compared with those specified in document DX would suggest replacing Ca by Sr in the oxide material disclosed in document DX does not convince the board either because the citations contain no indication that the alkaline-earth metal might have any particular influence on the sintering temperature of the material.

4.5 Having regard to the above, the board concludes that the subject matter of claim 1 involves an inventive step over the disclosure of documents D1 and DX within the meaning of Articles 52(1) and 56 EPC.

The same conclusion applies to the subject matter of claims 2 to 20 by virtue of their reference to claim 1.

5. The board concludes that the appeal of the opponent, although admissible, is not allowable.

Order

For these reasons it is decided that:

1. The appeal filed by the patent proprietor is inadmissible.

2. The appeal filed by the opponent is dismissed.

The Registrar: The Chairman:

P. Martorana E. Turrini