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D E C I S I O N
of 29 September 2003

Case Number: T 0495/00 - 3.3.5

Application Number: 93924600.5

Publication Number: 0660746

IPC: B01D 53/44

Language of the proceedings: EN

Title of invention:

Process for the purification of inert gases

Patentee:

SINCO ENGINEERING S.p.A.

Opponents:

- 01) Bühler AG
02) Zimmer Aktiengesellschaft

Headword:

Gas purification/SINCO

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step: yes - non-obvious alternative process"

Decisions cited:

-

Catchword:

-



Case Number: T 0495/00 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 29 September 2003

Appellant: SINCO ENGINEERING S.p.A.
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Respondent 01: Bühler AG
(Opponent 01) CH-9240 Uzwil (CH)

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Respondent 02: Zimmer Aktiengesellschaft
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 28 March 2000
revoking European patent No. 0660746 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: M. M. Eberhard
Members: B. P. Czech
H. Preglau

Summary of Facts and Submissions

- I. The appeal is from the decision of the opposition division dated 28 March 2000 revoking European patent No. 0 660 746.

The independent claim 1 of the granted patent reads as follows:

"1. Process for the purification from impurities formed of organic compounds of a recycle inert gas stream leaving a solid-state polycondensation reactor for aromatic polyester resins, comprising adding the stream with oxygen or gas containing oxygen, and circulating the gas stream on a catalytic bed containing Pt or mixtures of Pt and Pd supported on an inert porous support at temperatures from 250° to 600°C, characterized in that the quantity of oxygen used is stoichiometric with respect to the organic impurities or in such an excess that the gas at the outlet of the oxydation reactor contains up to 10 ppm of oxygen and in that the gaseous stream leaving the oxydation reactor is recycled to the solid-state polycondensation reactor previous a drying treatment to remove the water formed in the oxydation reactor."

- II. During the opposition proceedings the opponents inter alia cited the following documents:

D1: DD-A-242 181

D4: JP-A-46 020885 (translation into English)

D5: DE-B-25 59 290

D6: Leaflet labelled "Gas Purification by catalytic Oxidation, Chemisorption and Adsorption",
© Copyright 1992, Silica Verfahrenstechnik GmbH,
Berlin, Germany

D9: EP-A-0 222 714

D14: Kuznetsova, E. V. et al, "Purification of industrial vapor-gas discharges and wastewaters by vapor-phase catalytic oxidation", Khimicheskaya Promyshlennost, Vol. 19, No. 10, 1987, pages 16-18 (translation into English)

D16: DD-A-240 672.

III. In the contested decision, the opposition division came to the conclusion that, starting from the disclosure of D4 as closest prior art, the subject-matter of claim 1 was not based on an inventive step in view of the contents of D6 and D14.

IV. In its written statement setting out the grounds of appeal, the appellant (proprietor of the patent) contested the conclusions drawn by the opposition division.

V. In their respective replies, respondent 01 (opponent 01) and respondent 02 (opponent 02) maintained their objection, i.e. lack of inventive step of the claimed process, against the patent in suit.

VI. The parties were summoned to oral proceedings. In the annex to the summons, the board indicated that D9 or the prior art mentioned in the introductory part of the patent may, rather than D4, be considered to represent the closest prior art.

VII. With a telefax dated 4 September 2003, the appellant filed a declaration of Professor Bond and the further document

D15: Mars, P. and van Krevelen, D. W., "Oxidations carried out by means of vanadium oxide catalysts"; Spec. Suppl. Chem. Eng. Sci., 1954, **3**, pages 41-57.

VIII. Oral proceedings took place on 29 September 2003.

IX. The written and oral submissions of the parties, as far as they are relevant for the present decision, can be summarised as follows:

During the oral proceedings, the appellant confirmed that the process referred to in the introductory part of the description belonged to the prior art. Starting from this prior art or from D9 as the closest prior art, the claimed process was to be considered as a novel and different solution of a known problem. The proposed solution was a new approach which left the known path, and which relied on the dosing of the oxygen gas rather than on a subsequent hydrogenation step to control the amount of residual oxygen in the recycled gas.

Documents D1, D9 and D14 showed that up to 6 years before the invention was made, the skilled person considered that a substantial excess of gaseous oxygen was required to achieve low residual concentrations of

organic impurities in inert gases by means of catalytic oxidation. D6 was a commercial publication of a somewhat ambiguous teaching with respect to the removal of carbohydrate impurities. The method of D4 was fundamentally different from the one claimed. It did not involve a catalytic reaction in the sense of the patent since it was the lattice oxygen of the metal oxide that was consumed in the oxidation of the impurities. Hence, none of these documents could have incited the skilled person not knowing the invention to reduce the amount of gaseous oxygen supplied to the inert gas stream undergoing oxidation on a noble metal catalyst to a near stoichiometric amount, to thereby still obtain a satisfactory removal of the impurities present and very low residual oxygen concentrations, whilst omitting a subsequent hydrogenation step.

According to respondent 01, D4 was to be considered as the closest prior art. In view of e.g. D15, the process of D4 was to be considered as a catalytic process. D4 identified the need to avoid the presence of excess oxygen in the gas recycled to the solid-state polycondensation ("SSP") reactor. In order to avoid the catalyst regeneration necessary according to D4, the skilled person would consider the replacement of the metal oxide catalysts of D4 by other available catalysts not requiring regeneration, such as the known Pt oxidation catalysts. Since the addition of excess oxygen leads to extra costs and to disadvantageous levels of residual oxygen in the recycled stream, a more precise dosing of the oxygen required was an obvious measure, in particular in view of the common general knowledge as illustrated by D6 and D14. Carrying out near stoichiometric catalytic oxidations

was known, e.g. in the field of automobile exhaust gas purification. Reducing the amount of added oxygen to the lowest amount actually required, i.e. to a near stoichiometric amount not requiring subsequent hydrogenation, was thus a matter of routine experimentation and did not involve any inventive step.

Respondent 02 considered the prior art process referred to in the introductory part of the patent as the closest prior art. It argued however that the claimed process lacked inventive step irrespective of whether the said process or the processes according to D4 or D9 were considered as the closest prior art. Pointing to D5, it submitted that the skilled person knew that the residual oxygen concentration of the recycled gas stream needed to be less than 10 ppm. Hence, lowering the amount of added oxygen to near stoichiometric amounts was a mandatory and obvious measure for the skilled person. A further clear suggestion to operate in this way could e.g. be found in the table on page 4 of D6. It referred to its calculations submitted during the opposition proceedings and argued that applying the information contained in D6 to the purification of the SSP recycle gas would lead to the claimed process. Concerning D4, it pointed out that the overall oxidation reaction of the impurities was the same as according to claim 1 of the patent in suit, although the catalyst used was different and more difficult to handle. The skilled person would thus look for a more convenient catalyst. In D14, he would find the information that the performance of Pt catalysts was comparable to the one of metal oxide catalysts. The replacement, in the method of D4, of the metal oxide catalyst by a Pt catalyst was thus an obvious measure.

Keeping in mind that the residual oxygen concentration in the recycled gas was to be kept below 10 ppm, the skilled person would inevitably arrive at the claimed process.

- X. The appellant requested that the decision under appeal be set aside and that the patent be maintained with the claims as granted and the description as amended during the oral proceedings.

The respondents both requested that the appeal be dismissed.

Reasons for the Decision

1. The board is satisfied, and it was not contested by the respondents, that the amendment consisting in the deletion of lines 51 to 53 of the patent as granted is in compliance with the requirements of Articles 84, 123(2) and 123(3) EPC. This amendment removes possible doubts concerning the nature, origin and destination of the gas to be treated in the process of claim 1.

2. The board is convinced that the claimed process is novel with respect to the documents cited by the respondents. Since this was not in dispute, further considerations are not necessary. The differences in the claimed process over the prior art will appear from the following discussion of inventive step.

Inventive step

3. *Closest prior art*

3.1 In its introductory part, the contested patent (and the application as filed) refer to a process for the purification of inert gases coming from SSP reactors which includes an oxidation stage of the impurities, a deoxidation stage with hydrogen in order to eliminate the oxygen used in the first stage and a drying-process stage of the gaseous stream in order to eliminate the water formed in the previous stages. In connection with said prior art process, the patent in suit further indicates the following: "The oxidation stage is carried out with oxygen or with gas containing oxygen (generally air) by using an oxygen excess on the stoichiometric quantity as regards the impurities. The oxidation stage performance is controlled so that the gaseous stream at the outlet contains an oxygen excess of 50-500 ppm. The reaction is generally carried out at a temperature between 500°C and 600°C by circulating the gaseous stream on a catalyst bed formed of a support coated with platinum or platinum/palladium. The high oxygen content present in the gaseous stream coming out of the oxidation section does not allow to recycle the same to the SSP reactor, previous drying-process, due to the possible oxidation reactions and/or polymer degradation. It is necessary a deoxidation treatment (sic) with hydrogen in order to eliminate the present oxygen. The performance of the deoxidation section is monitored by controlling the stream hydrogen excess at the outlet and the oxygen content. The last stage is a drying-treatment carried out by circulating the gas on a silica gel, molecular sieves or other beds

of drying materials. In this stage the water stripped from the polymer chips and generated in the oxidation and deoxidation stages, is eliminated. ... After this stage the gas is recycled to the SSP reactor". See page 2, lines 8 to 28 and line 35 of the patent.

- 3.2 As confirmed by the appellant during the oral proceedings, this process indeed belongs to the prior art to be taken into consideration. This was not disputed by the respondents. D9 discloses a process of this kind involving a catalytic oxidation of the impurities with air and the removal of the oxygen excess by catalytic hydrogenation, but does not indicate the specific type of oxidation catalyst to be used, see claims 1, 6, 10; page 4, line 11 to page 4, line 4; and Figure 1.
- 3.3 Considering its similarity to the process claimed in terms of the type of oxidation process (catalyst used, addition of gaseous oxygen to the inert gas stream), the board takes the view that the process referred to in the introductory part of the patent in suit represents the closest prior art for the assessment of inventive step.
4. Starting from the said prior art process mentioned in the patent in suit, the technical problem to be solved by the claimed process can in any case be seen in providing a further process for the purification of the inert gas stream to be recycled in a SSP process for the production of aromatic polyester resins, by which impurities are reduced to an acceptable level without causing oxidation reactions or polymer degradation in

- the polymerisation reactor. See page 2, lines 21 to 23 and 42 to 44 of the patent in suit.
5. The board considers it to be plausible and the respondents have not contested that the measures proposed by claim 1 of the patent in suit solve the stated technical problem. Hence, it remains to be seen whether the claimed solution was suggested by the cited prior art in view of the general knowledge of the person skilled in the art.
 6. It emanates from prior art on file that it was known at the priority date of the patent in suit that the residual concentrations of both impurities and oxygen in the recycled inert gas stream should be sufficiently low so as to avoid the disturbance of the polymerisation reaction. See e.g. D5 (column 4, lines 43 to 59) and D9 (page 5, lines 2 to 4), which respectively refer to residual oxygen concentrations in the recycled gas of less than 10 ppm and less than 2 ppm, and D4 (page 3, last full sentence).
 7. The solution foreseen by the stated closest prior art and D9 comprises the removal of unreacted oxygen previously added in excess by means of a catalytic hydrogenation step. Hence, this prior art, taken alone, cannot possibly be considered to suggest the omission of the said catalytic hydrogenation step.
 8. Generally speaking, a skilled person will usually carry out a chemical reaction using those amounts of reagents, and not more, that are considered necessary to achieve the required degree of conversion of the starting products, bearing in mind the kinetic and equilibrium

characteristics of the reaction. However, documents D1, D9, D14 and D16 show that before the filing of the patent in suit, a skilled person had good reasons to believe that a substantial excess of gaseous oxygen was necessary for an almost complete oxidation of organic impurities contained in inert gases at relatively low concentrations in the presence of oxidation catalysts, which excess of gaseous oxygen could be expected to lead to residual oxygen concentrations of more than 10 ppm when applied to the purification of the recycle gas from an aromatic polyester SSP reactor. D14 and D16 specifically refer to noble metal catalysts. See D1, page 1, section entitled "Charakteristik der bekannten technischen Lösungen", second paragraph, D9, page 4, lines 18 to 23, D14, page 29, last paragraph, page 30, third and fourth full paragraphs, and D16, claim 1 and page 1, second paragraph from the bottom. Hence, in view of the evidence on file, it cannot be concluded that the skilled person starting from the closest prior art and confronted with the stated technical problem, aware of the contents of the said documents and relying solely on his general common knowledge, would have considered lowering the amount of oxygen added to the extent that the subsequent hydrogenation step could be disposed of as an obvious, self-imposing measure.

9. D6 is a publication of advertising/commercial character describing different technologies offered for various gas purification problems. D6 refers to the removal of oxygen, carbon monoxide, hydrogen and hydrocarbon compounds from gases. The impurities to be removed are normally present in concentrations of less than 2 to 3 vol%, and their "reachable" residual content is less than 1 ppmv. One of the suggested methods is the

catalytic combustion of the impurities with oxygen added to the gas to be purified on noble metal catalysts (Pd and Pt). See page 3, right-hand column and page 4, right-hand column. Having regard to the removal of hydrocarbons from gases, it is indicated in the table on page 4 labelled "Characteristic data of this process" that the temperatures to be applied are in the range of 250 to 500°C. Concerning the inlet concentration of the hydrocarbons, the reader is referred to the manufacturer of the system (see "on request"). A residual content of impurities of less than 1 ppmv is indicated in a general manner, i.e. for all the purification reactions summarised in the said table. Similarly, the consumption of reaction gas is generally specified to be "stoichiometric ratio + 0.1% surplus" without, however, indicating the basis for the said percentage.

9.1 D6 addresses several different possibilities for the purification of gases of various compositions in a rather general and condensed manner. D6 does not explicitly disclose the catalytic combustion of specific hydrocarbons present at specific concentrations in a gas to be purified of specific origin. Consequently, D6 does not disclose any corresponding specific (absolute) initial and residual oxygen concentrations. D6 also does not address the oxidation of hydrocarbon **derivatives**, let alone of the glycol and aldehyde type.

9.1.1 Respondent 02 argued that the "surplus" of 0.1% referred to on page 4 of D6 was to be based on the stoichiometric amount of the reaction gas added. However, as pointed out by the appellant, the table on

page 4 of D6, which refers to the said "0.1% surplus", i.e. to an excess of the reaction gas, also refers to the catalytic combustion of oxygen contained in a gas as an impurity by means of an excess of hydrogen. Concerning this particular type of purification, the table on page 3 of D6 mentions that the purified gas comprises residual hydrogen (reaction gas) in a concentration of 0.1%, which is much higher than the 10 ppm residual reaction gas (oxygen) concentration referred to in claim 1 of the contested patent. Assuming an almost complete reaction (" < 1 ppmv" residual impurities), this would mean that the 1000 ppm residual hydrogen are to be considered as the "surplus" amount provided in excess of the stoichiometric amount. In accordance with the calculations submitted by the appellant during the opposition proceedings, the validity of which has not been contested as such, equating these 1000 ppm to the said "surplus of 0.1%" (based on the required stoichiometric amount) would mean that the stoichiometric amount of hydrogen used was 1,000,000 ppm.

9.1.2 The latter amount does not appear to be consistent with the relatively low impurity contents of up to 3 vol% (30,000 ppm) to be dealt with according to D6. Moreover, the board notes that the operating conditions for purifying gases with higher impurity concentrations are not disclosed in D6, which teaches without indicating anything more that "special measures" are necessary for treating such gases. See page 3, right-hand column, 2nd paragraph.

9.1.3 In view of the inconsistencies between the information presented in the tables on pages 3 and 4, D6 is

considered to be ambiguous with respect to the meaning of the indication "+ 0.1% surplus".

9.2 Respondent 02 argued that D6 would generally disclose the catalytic oxidation of hydrocarbon impurities down to a residual level of less than 1 ppmv by means of oxygen added in a stoichiometric amount plus a 0.1% excess (based on the latter amount). However, considering

- the ambiguity addressed under 9.1.3 above,
- that D6 refers the reader to the manufacturer with respect to the treatment of hydrocarbon containing gases (see "on request"), and
- that the only residual reaction gas concentration explicitly mentioned in D6 in connection with a specific purification being the 0.1% (1000 ppm) hydrogen mentioned in the table on page 3,

the board cannot accept this interpretation of the disclosure of D6.

9.3 Consequently, the board does not accept the argument of respondent 02 that applying the teaching of D6 to the recycle gas stream of an SSP reactor meant, according to its calculations submitted during the opposition procedure, that, the stoichiometric amount of oxygen required to oxidise impurities present in the recycle gas stream at concentrations of about 2000 to 3000 ppm (defined as methane equivalents, see patent in suit, page 2, lines 8 to 9) being about 4000 to 6000 ppm, a "surplus" of 0.1%, based on the latter amount, would

thus only correspond to about 4 to 6 ppm, i.e. a concentration within the limits of present claim 1.

- 9.4 For the preceding reasons, the board is not convinced that the information presented in D6 clearly suggests that the specific impurities occurring in the recycle inert gas streams of the SSP for producing aromatic polyesters could be removed by reaction with gaseous oxygen on noble metal catalysts at an acceptable rate and to the extent required for their recycling, and without leading to a residual oxygen concentration of more than 10 ppm. Hence, D6 does not provide a clear and strong incentive to deviate from the prior art method and consider replacing the use of an excess amount of oxygen with subsequent hydrogenation by the use of either a stoichiometric amount of oxygen or an amount as defined in claim 1 in a manner permitting the omission of a subsequent hydrogenation.
- 9.5 Document D14 is a document investigating the catalytic "deep" oxidation of organic compounds with air in a medium of water vapour. Platinum and copper oxide catalysts on alumina carriers were tested at elevated temperatures. The compounds investigated include ethylene glycol and aldehydes. See the translation into English, page 28, Table 1, page 29, Table 2 and the last two paragraphs, page 30, second, third and fourth full paragraphs. In order to obtain a high degree of purification with the said catalysts, the oxidising air is present in the vapour-air mixture at "excess air coefficients" of at least 1.3 (see all the examples in Table 2 and the fourth full paragraph on page 30).

9.5.1 The sole purpose of the process disclosed in D14 is the purification of waste water or vapour-gas industrial discharges. The recycling of the treated gas to a specific process is not envisaged. Hence, the residual oxygen concentration of the treated gases, or any risks associated therewith, are not addressed as an important issue, let alone the risk of oxidation reactions and polymer degradation associated with the residual oxygen concentration in the recycled gas of a SSP process for producing aromatic polyester resins.

9.5.2 Considering this remote field of application, the person skilled in the art confronted with the stated technical problem would not even take D14 into consideration. Moreover, D14 suggests the use of excess air coefficients which, when applied to the catalytic purification of the water vapour containing SSP recycle gas (see Table 1, column 1 of the patent in suit) could not be expected to lead to residual oxygen concentrations of less than 10 ppm. Hence, even assuming for the sake of argument, that the skilled person would consider D14, he would find there even less incentive than in D6 for modifying the known oxygenation/hydrogenation technique in a way leading to the claimed process.

10. Document D4 also discloses a method for purifying a recycle inert gas stream from a solid phase polymerisation for producing aromatic polyesters. In order to purify it, the said stream is contacted with at least one metal oxide selected from vanadium oxide, molybdenum oxide, silver oxide and phosphorus oxide kept at a temperature of from 150 to 300°C. Organic reaction products comprised in the gas stream, i.e.

glycol and aldehyde, are oxidised to CO₂ and water by the oxygen of the metal oxides, whereby the inert gas stream is purified from compounds interfering with the polymerisation reaction. The water in the gas stream is removed in a subsequent process step. In the course of the process, the metal oxide decreases in oxygen content and therefore needs to be regenerated, although "seldom", by re-oxidation with air at higher temperatures. See D4, page 1, 1st paragraph, the paragraph bridging pages 2 and 3, page 4, 2nd, 3rd and 4th paragraphs, page 5, 1st and 2nd paragraphs, page 6, 2nd paragraph. According to example 2, a nitrogen gas stream containing ethylene glycol vapour at 0.1 mmHg was passed through a column charged with vanadium pentoxide at varying temperatures and contact times. Analysis of the outlet gases "indicated that they were composed almost exclusively of water and carbon dioxide gas". According to comparative example 3, referring to example 2, a nitrogen gas stream containing ethylene glycol at the same concentration was passed through the column charged with vanadium pentoxide (contact time about 1 second), but "oxygen necessary for the decomposition of ethylene glycol (partial pressure of 0.35 mmHg) was contained in the nitrogen gas". See D4, page 6, last line to page 7, 3rd paragraph). It was found that "there was little or no difference from the decomposition using the oxygen free gas". See D4, page 7, last paragraph to page 8, last paragraph.

- 10.1 The board accepts that the method of D4 can be considered as a "catalytic" method in the broadest sense of the term. This view is, for instance, confirmed by document D15 cited by the appellant, see e.g. the expression "vanadium oxide catalysts" used in

its title. However, the method of D4 differs from the one of the closest prior art in that it is based on a different reaction mechanism wherein the oxygen required for the oxidation of the impurities is provided by the metal oxide itself. Hence, the method of D4 does not require the addition of controlled amounts of gaseous (molecular) oxygen to the recycle inert gas stream. Consequently, it is plausible that according to D4 the impurities are oxidised without the occurrence of undesirable excess concentrations of residual oxygen in the gas leaving the purification. Comparative example 3, which describes the treatment of an ethylene glycol (0.1 mmHg) containing nitrogen stream additionally containing oxygen (0.35 mmHg) focuses on the degree of decomposition of the impurities and does not report the residual O₂ concentration in the outlet gas.

- 10.2 D4 also deals with the problem of providing a process for the purification of the recycle inert gas stream in a SSP process, by which impurities are reduced to an acceptable level and undesirable effects on the polymerisation reaction are to be avoided, see D4, page 2, last paragraph to page 3, line 1 and page 3, last full sentence. Confronted with the stated technical problem, the author(s) of D4 have envisaged *inter alia* the possibility of removing the impurities by reacting them with oxygen introduced into the inert gas stream. However, they apparently considered such a method to be disadvantageous in terms of the reaction rate and the resulting residual oxygen content, and therefore opted for the method based on the use of metal oxide as oxidant. See page 3, last full paragraph. In the board's view, comparative example 3 merely shows

that when vanadium oxide is used as source of oxygen, the addition of gaseous oxygen to the inert gas stream is not necessary for achieving the required purification, and does not suggest an operating mode with a continuous addition of oxygen gas in the presence of a Pt or a Pd/Pt catalyst, wherein the residual concentration of said added oxygen is less than 10 ppm.

10.3 In view of the different oxidation techniques respectively relied upon by the closest prior art and by the method of D4, and also in view of the discouraging character of the quoted passage of D4 relating to the concept of reacting gaseous oxygen with the impurities, a skilled person confronted with the stated technical problem would not have envisaged a combination of the closest prior art and D4 at all. Even assuming for the sake of argument that the skilled person would have taken D4 into consideration at all, the latter cannot be considered to suggest those modifications of the method according to the closest prior art which are necessary to arrive at the claimed process, i.e. a close control of the addition of oxygen gas to the stream to be purified making it possible to omit the hydrogenation step.

10.4 Even assuming, for the sake of argument and in the favour of the respondents, that D4 could be considered to represent the closest prior art, the board, for analogous reasons, would still come to the same conclusion. None of D6, D14 or the prior art identified in the introductory part of the patent can be considered to suggest the replacement of the oxide catalyst of D4 by a Pt or Pd/Pt catalyst, while at the

same time carrying out a close control of the oxygen addition in a manner permitting the omission of the subsequent hydrogenation.

11. The remaining documents cited by the parties are less relevant and contain no additional information which, in combination with the preceding documents, could render the subject-matter of claim 1 obvious.
12. The subject-matter of claim 1 as granted and, consequently, of dependent claims 2 to 8 as granted, is thus based on an inventive step.

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 with
 - the claims as granted,
 - description pages 2 and 4 as granted,
 - amended description page 3 as filed during the oral proceedings.

The registrar

The Chairman

A. Wallrodt

M. Eberhard