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**Datasheet for the decision  
of 26 February 2018**

**Case Number:** T 1266/16 - 3.3.10  
**Application Number:** 07737056.7  
**Publication Number:** 2008998  
**IPC:** C07C319/14, C07C335/32,  
C08G75/04, G02B1/04, C08G18/38  
**Language of the proceedings:** EN

**Title of invention:**

PROCESS FOR PRODUCTION OF (POLY)THIOL COMPOUND FOR USE AS  
OPTICAL MATERIAL

**Patent Proprietor:**

Mitsui Chemicals, Inc.

**Opponents:**

Sommer, Johanna  
Covestro Deutschland AG  
Bruno Bock Chemische Fabrik GmbH & Co. KG

**Headword:**

**Relevant legal provisions:**

EPC Art. 100(c), 54(2)

**Keyword:**

Amendments - added subject-matter (yes) - main request, second and fourth auxiliary requests

Novelty - (no) - first, third and fifth auxiliary requests

**Decisions cited:**

T 0242/88

**Catchword:**



**Beschwerdekammern**  
**Boards of Appeal**  
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Case Number: T 1266/16 - 3.3.10

**D E C I S I O N**  
**of Technical Board of Appeal 3.3.10**  
**of 26 February 2018**

**Appellant:** Mitsui Chemicals, Inc.  
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**Decision under appeal:**      **Decision of the Opposition Division of the  
European Patent Office posted on 15 March 2016  
revoking European patent No. 2008998 pursuant to  
Article 101(3) (b) EPC.**

**Composition of the Board:**

**Chairman**                    P. Gryczka  
**Members:**                 R. Pérez Carlón  
                                  C. Schmidt

## Summary of Facts and Submissions

- I. The appellant (patent proprietor) lodged an appeal against the decision of the opposition division to revoke European patent No. 2 008 998.
- II. Three notices of opposition had been filed on the grounds of added subject-matter (Article 100(c) EPC), insufficiency of disclosure (Article 100(b) EPC) and lack of novelty and inventive step (Article 100(a) EPC).
- III. The documents filed during the opposition proceedings include the following:
- D1 JP 2001-39944 (including two translations into English)
  - D3 EP 0 378 895 A1
  - D4 Thiourea (Reagent) JIS K 8635, established in 1953, 1992 revised edition (including a partial translation into English)
  - D5 Hazardousness Assessment Report Ver. 1.1 No. 49 Thiourea, prepared in March 2003 (including a partial translation into English)
  - D6 Safety (Hazard) Assessment Sheet of Existing Chemical Substances, prepared in June 1997 (including a partial translation into English)
  - D10 Thiourea, various Lot-Numbers, Analytical Result, 2005
  - D14 Mertschenk *et al.* Thiourea and Thiourea Derivatives, Ullmann's Encyclopedia of Industrial Chemistry, Wiley-VCH Verlag GmbH&Co. KGaA, Weinheim 2005

The following declarations were filed during the opposition proceedings:

- D29 Declaration from Shigetoshi Kuma
- D30 Declaration from Seiichi Kobayashi

The experimental evidence filed during the opposition proceedings includes the following:

- D34 Experimental Report re D3 and D18
- D35 Experimental Report re D3 Example 3
- D36 Thiourea Preparation
- D42 Repeat Preparations

- IV. The opposition division concluded that claim 1 of the patent as granted, which is also the main request in these appeal proceedings, contained added subject-matter, and that the process of claim 1 of the first auxiliary request then pending, which corresponds to claim 1 of the first auxiliary request in appeal, was not novel.
- V. With the statement setting out the grounds of appeal dated 25 July 2016, the appellant requested maintenance of the patent as granted and filed a main request and a first auxiliary request. Under cover of a letter dated 9 January 2018, the appellant filed its second to fifth auxiliary requests.
- VI. The board informed the parties in a communication dated 30 October 2017 in preparation for the oral proceedings before it, which took place on 26 February 2018, that there were some differences between claim 5 as granted and claim 5 of the main request filed with the statement of grounds of appeal. The board understood that the appellant requested maintenance of the patent

as granted, in its legally binding version as reflected in the "Druckexemplar".

Claim 1 of the patent as granted reads as follows:

*"A process for producing a (poly)thiol compound for an optical material comprising:*

*reacting an organic (poly)halogen compound or a (poly)alcohol compound with thiourea to produce an isothiuronium salt, and*

*hydrolyzing the obtained isothiuronium salt in the presence of aqueous ammonia to produce a (poly)thiol compound,*

*in which the calcium content in the thiourea is not more than 1.0 wt%."*

Claim 1 of the second and forth auxiliary requests contains, like claim 1 of the main request, the feature

*"hydrolyzing the obtained isothiuronium salt in the presence of aqueous ammonia to produce a (poly)thiol compound"*.

In claim 1 of the first auxiliary request, this feature is worded as follows:

*"hydrolyzing the obtained isothiuronium salt using aqueous ammonia to produce a (poly)thiol compound"*

Claim 1 of the third and fifth auxiliary requests differs from claim 1 of the first auxiliary request only in that it requires a calcium content in the thiourea of no more than 0.5 wt% and 0.2 wt%, respectively.

VII. The arguments of the appellant which are relevant for the present decision are the following:

Claim 1 of the patent as granted found a basis in the combination of claim 1 as originally filed and paragraph [0025] of the description. The skilled person would recognise the role of aqueous ammonia in the claimed process, which thus did not need to be explicitly stated in claim 1. Furthermore, ammonia was a product of the hydrolysis of thiuronium salts, so that its presence in the claimed process was implicitly disclosed for the skilled reader. For these reasons, claim 1 of the patent as granted did not contain added subject-matter. The same arguments applied to claim 1 of the second and fourth auxiliary requests.

The subject-matter of claim 1 of the first auxiliary request was novel, as D1 did not disclose a process inevitably carried out with thiourea having less than 1.0 wt% calcium. Firstly, it was plausible that thiourea having more than 1.0 wt% calcium was available before the filing of D1. Secondly, the evidence provided as D34, D35 and D42 proved that polythiol synthesis had already been carried out using thiourea with a high content of calcium. Lastly, the authors of D1 could have synthesised thiourea which contained more than 1.0 wt% of calcium, as is shown in D36.

The arguments with respect to novelty applied in the same manner to claim 1 of the third and fifth auxiliary requests.

VIII. The arguments of the respondents (opponents) which are relevant for the present decision are the following:

The passage in paragraph [0025] cited by the appellant



could not provide a basis for the feature "in the presence of aqueous ammonia", as it disclosed aqueous ammonia as hydrolysis reagent. This role introduced further limitations in the claimed subject-matter, such as with respect to the required amount, which were not features of claim 1. For this reason, claim 1 of the main request and the second and fourth auxiliary requests contained subject-matter going beyond that of the application as originally filed.

Regarding novelty, the respondents argued that the appellant bore the burden of proof with respect to the presence of calcium above the threshold of 1.0 wt% required by claim 1 from the process of D1, as it was not feasible for them to prove its absence beyond any reasonable doubt. The evidence on file was, however, not sufficient to discharge such burden.

Document D1 related to the preparation of polythiols for plastic lenses. The skilled reader, even in the absence of information on the relative amount of calcium used in the examples of D1, would inevitably consider thiourea of standard purity (D4-D6, D10, D14) as the starting material for such process. Thiourea of standard purity has a relative amount of calcium below 0.05 wt%, as shown by the amount of sulfates/ash disclosed in that documents. For this reason, the process of claim 1 of the first auxiliary request was not novel. The same reasons applied to the third and fifth auxiliary requests.

- IX. Respondent 3 informed the board that it would not be attending the oral proceedings.
  
- X. The final requests of the parties were the following:

- The appellant requests that the decision under appeal be set aside and the patent be maintained as granted, i.e. in the form of the text of the "Druckexemplar" (main request) or, subsidiarily, in the form of the first auxiliary request, filed with the statement setting out the grounds of appeal dated 25 July 2016, or of one of the "Replacement second auxiliary claim request" to "Replacement fifth auxiliary claim request" requests filed under cover of a letter dated 9 January 2018.
  
- The respondents request that the appeal be dismissed.

XI. At the end of the oral proceedings, the decision was announced.

### **Reasons for the Decision**

1. The appeal is admissible.

Amendments, main request, second and fourth auxiliary requests

2. Claim 1 of these requests contains the feature

*"hydrolyzing the obtained isothiuronium salt in the presence of aqueous ammonia to produce a (poly)thiol compound".*

3. The appellant argued that paragraph [0025] of the application as originally filed provided the required basis for the wording *"in the presence of aqueous ammonia"*. This passages discloses

*"Hydrolysis which subsequently carried out after producing an isothiuronium salt is conducted by using*

*usual base water, similar to a conventional method. Examples of the kind of base water in use include [...] ammonia water[...]"*.

4. However, paragraph [0025] of the application as originally filed only discloses aqueous ammonia in a specific role, namely for hydrolysing the corresponding thiuronium salt. This role implicitly introduces limitations to the process, for example with respect to the amount of ammonia required, which are not inherent to the feature "in the presence of aqueous ammonia".

For this reason alone, claim 1 contains subject-matter going beyond that disclosed in the application as originally filed, with the consequence that the ground of opposition defined in Article 100(c) EPC precludes the maintenance of the patent as granted.

5. The appellant argued that the skilled person would have read claim 1 of the patent as granted with a mind willing to understand. The standard preparation of thiols using thiourea requires an aqueous base hydrolysis. Considering that aqueous ammonia would not have taken part in the process of claim 1 was not a reasonable reading of claim 1.

However, the skilled reader would consider that claim 1 as granted, by mentioning "in the presence of aqueous ammonia", also relates to a process in which the amount of ammonia is too low for hydrolysing a thiuronium salt. This embodiment is, however, not envisaged by the wording of paragraph [0025] of the application as originally filed. This argument of the appellant is thus not convincing.

6. In a different line of argument, the appellant concluded that, since ammonia is a product of the basic hydrolysis of thiuronium salts, its presence was an inherent feature of the claimed process and could not represent added subject-matter.

However, the description of the application as originally filed, including the examples, discloses aqueous ammonia as a reagent. The interpretation of the appellant that aqueous ammonia in claim 1 could refer to ammonia formed in the reaction is not in agreement with the disclosure of the patent application as a whole. For this reason, this argument of the appellant fails to convince the board.

7. As claim 1 of the second and fourth auxiliary requests also contains the feature "in the presence of aqueous ammonia", in the same context as claim 1 of the patent as granted, these requests contain added subject-matter for the same reasons as the latter, with the consequence that they are not allowable.

Novelty: first, third and fifth auxiliary requests

8. Claim 1 is directed to a process for producing a (poly)thiol compound for an optical material using thiourea with a calcium content of not more than 1.0 wt%.

It is not disputed that example 1 of document D1 explicitly discloses all the features of claim 1 with the exception of the amount of calcium in thiourea. The parties were, however, divided on whether or not this example implicitly disclosed a process carried out with thiourea having a calcium content below the threshold

required by claim 1.

9. According to respondent 1, citing decision T 242/88 (not published in OJ EPO), the appellant arguing that the calcium content in the thiourea used in D1 is above the threshold indicated in claim 1 bears the burden of proof of showing the fact it alleges.
10. The appellant has not disputed that the burden of proof was on its side in this respect, and argued that the evidence submitted credibly showed that the authors of D1 could have used thiourea having more than 1.0 wt% calcium.

In this respect, the appellant relied on three lines of argumentation, namely that

- it was at least plausible, if not likely, that thiourea available from commercial sources had a calcium content greater than 1.0 wt% at the filing date of D1,
- having regard to APHA values in the art, the authors of documents such as D3 relating to polythiol synthesis must have used thiourea having more than 1.0 wt% calcium and, lastly, that
- the authors of D1 could have synthesised thiourea instead of purchasing it from a commercial source; D36 proved that it was highly possible that such urea would have contained more than 1.0 wt% of calcium.

The appellant concluded for these reasons that it was not proven beyond reasonable doubt that the process of D1 had been carried out with thiourea containing less

than 1.0 wt% calcium, as required by claim 1, whose subject-matter was for this reason novel.

On the three lines of argumentation, the board concludes as follows:

10.1 Commercially available thiourea

There is no evidence on file which could show that thiourea with a higher content of calcium than required by claim 1 was commercially available at the filing date of D1.

The appellant submitted two declarations (D29, D30) by the inventors of the patent in suit stating that "during the development of the invention, thiourea was obtained from a number of different commercial suppliers. The Ca content in the commercially available industrial thiourea from some of these suppliers was found to be more than 1.0 wt%".

Notwithstanding that both declarations are word-for-word identical regarding thiourea purity, they do not contain any specific detail with respect to the suppliers they refer to, let alone any data regarding purchase or analysis performed. As such, these statements, devoid of any corroborating evidence, cannot be considered as proof of the appellant's argument.

In contrast, the respondents have submitted ample evidence that thiourea having a very low calcium content was not only available but standard before the filing date of D1. D4 refers to 98.0% pure thiourea having an ignition residue (sulfate) of 0.01% or lower, which includes not only calcium, but also sulfate

conterions and other cationic impurities; D5 and D6 also refer to 99% pure thiourea (see partial translations); D10 relates to 99.7% pure thiourea having less than 0.1% sulfate-ash; D14 discloses 99.0% pure thiourea having less than 0.10% ash.

This argument of the appellant is thus not convincing.

#### 10.2 Relationship between APHA value and Yellow Index and calcium content

The appellant acknowledged that none of the examples of document D3 were relevant for the novelty of claim 1, as the hydrolysis step was carried out with NaOH instead of with aqueous ammonia.

Document D3 disclosed the APHA of the polythiol obtained, and these values reflected its colour: the smaller the value, the less coloured the product.

The appellant relied on the experimental evidence D34 and D35 for showing that some processes for producing polythiols of the prior art, such as that of D3, used thiourea having a calcium content above the threshold defined in claim 1, in particular having regard to the APHA values obtained.

D34 and D35 disclose the results of carrying out various examples of D3 with thiourea having different relative amounts of calcium. The appellant concluded from them that D3 must have been carried out with thiourea having more than 1.0 wt% of calcium.

##### 10.2.1 Experimental evidence D34

The polythiol obtained according to example 1 of D3 has

an APHA value of 15. D34 relates to experiments carried out according to examples 1 (synthesis of polythiol) and 9 (preparation of lenses from the polythiol of example 1) of document D3, using thiourea containing different amounts of calcium.

The data in D34 shows a higher APHA value of those polythiols obtained with thiourea having a higher calcium content (1.2 wt% vs. 0.05 wt%). Thiourea having 1.2 wt% calcium lead to polythiols having the same APHA value as in example 1 of D3.

D34 merely proves that the APHA value disclosed in D3 could have been due to the presence of calcium in the thiourea, i.e. that its presence is *sufficient* for the colouring, notwithstanding that this conclusion is rebutted by the results of D35 (see following point). It fails to prove, however, that such presence is *necessary* for achieving it. For this reason alone, the appellant's argument is not convincing.

#### 10.2.2 Experimental evidence D35

D35 relates to experiments carried out in accordance with example 3 of document D3, according to which an APHA value of 10 was obtained.

As in the previous case, D35 shows a link between the colouring of polythiols and the amount of calcium present in thiourea, but fails to show that the authors of D3 had used thiourea with an unusually high calcium content.

In addition, D35 proves that the conclusion drawn from D34 by the appellant (see previous point), i.e. that the presence of a high relative amount of calcium is a



sufficient condition for the colouring is not correct, as it is possible to obtain a polyol with an APHA value of 10 by using thiourea containing more than 1.0 wt% of calcium.

#### 10.2.3 Experimental evidence D42

The appellant further argued that the yellow index (YI) of a sample was a more accurate colour indication than the APHA value. D42 showed a direct relationship between the yellow index and the calcium content of the thiourea used.

As in the previous cases, even acknowledging that the alleged relationship existed, i.e. that the presence of a high relative amount of calcium were *sufficient* for the colouring, this experimental evidence fail to prove its presence above the threshold defined in claim 1.

#### 10.3 Alleged synthesis of thiourea by the authors of D1

The appellant argued that the inventors of D1 could have prepared thiourea, instead of purchasing it from a provider. Document D36 proved that such a thiourea could contain more than 1.0 wt% calcium.

Contrary to the arguments of the appellant, experimental evidence D36 does not disclose the preparation of urea using the standard process described for example in D14, section 2.3, as it does not include a CO<sub>2</sub> treatment step to form (water insoluble) calcium carbonate.

In addition, thiourea having 98.70% purity cannot contain 1.2 wt% calcium, even if it were the sole impurity present, as it requires a counterion. Due to

the synthesis process, calcium in the product of D36 should be in the form of calcium hydroxyde, which implies that, at most, 54% of the impurities can be calcium  $M_r(\text{Ca}(\text{OH})_2)=74$ ;  $A_r(\text{Ca})=40$ . Thus, the amount of calcium in a 98.70% pure urea cannot be higher than 0.65 wt%.

The appellant argued in this respect that purity could have been determined using a technique which would not take into account the amount of calcium, such as  $^1\text{H}$  NMR, and the purity obtained by this technique would be compatible with the relative amount of calcium in D36.

However, section 2.6 of D14 discloses that thiourea's purity is analysed by titration. The board sees no reason why a product which is produced in large amounts (D10 refers to 5 tons; D14, section 2.7 discloses that it can be packed in folding containers holding up to 1500 kg) and whose synthesis has been known for more than 50 years (statement of grounds of appeal, page 18, lines 28-33) should be analysed using a non-accurate method such as  $^1\text{H}$  NMR, which would not detect inorganic impurities, as long as an accurate protocol is available. This argument of the appellant is thus dismissed.

In conclusion, the board is of the view that it is not plausible that the skilled person would have either carried out the preparation of thiourea as in D36, or analysed its purity, as argued by the appellant, let alone have used such a starting material in the process of D1.

10.4 The appellant has thus also not proven that thiourea having calcium above the required threshold was

available to the skilled person from usual sources, such as providers, nor that such thiourea must have been used by the authors of documents in the field of the patent in suit, nor that if the authors of document D1 had synthesised thiourea following generally acceptable preparation processes such as that of D14, a product having more than 1.0 wt% of calcium would have been obtained.

For these reasons, it is concluded that the appellant has not proved that the process disclosed in document D1 could have been carried out using thiourea having a calcium content over the threshold defined in claim 1.

11. Considering the above, with respect to the novelty of the claimed process over that of document D1, the skilled reader's understanding of the disclosure of the latter remains to be examined.

It is not disputed that document D1 neither discloses the purity of thiourea used, let alone its calcium content, nor refers to any commercial source or discloses any preparation method.

It is further not disputed that thiourea having a very low calcium content was not only available but standard before the filing of D1.

The appellant acknowledged that the preparation of thiourea had not substantially changed for more than 50 years (see for example its statement of grounds of appeal, page 18, lines 28-33). Document D14 (section 2.6) discloses that commercial thiourea is of relatively high quality; every data on file refers to calcium contents well below 0.1 wt % (see for example D4, point 2.5, calcium is quantified as "ash"; D5, D6,

D10).

12. Taking into consideration that

- document D1 is directed to a process for producing polythiols suitable for the preparation of plastic lenses [0055], i.e. that colouring of the final product is important
- thiourea containing very few impurities (calcium included) was standard in the market before the filing date of D1, and
- it is generally known that impurities present in starting materials may have an influence on the colouring of the final product,

the board concludes that the skilled reader, despite D1 being silent on the amount of impurities of thiourea, would consider the use of thiourea at least of standard purity (D4-D6, D10, D14), i.e. with a relative amount of calcium well below the threshold defined in claim 1, to be inherently disclosed.

For these reasons, the process of claim 1 of the first auxiliary request is not novel (Article 54(2) EPC), with the consequence that this request is not allowable.

13. The appellant argued that there was no indication that thiourea with a low content of calcium could be used for obtaining thiols in a process such as that of D1 and that, in any case, the arguments in the previous point related to inventive step and not to novelty.

However, the issue is not whether the skilled reader

would have considered urea of standard purity (such as that of D14) as a suitable starting material in the process of D1, which would indeed be an issue related to inventive step.

This argument of the appellant is thus not convincing.

14. The appellant has not disputed that the conclusion on the issue of novelty would not differ with respect to claim 1 of the third and fifth auxiliary requests, as the amount of calcium defined by these requests also lies well above the limit of calcium in thiourea of standard purity. These requests are thus not allowable for the same reasons as the first auxiliary request.

## Order

### **For these reasons it is decided that:**

The appeal is dismissed.

The Registrar:

The Chairman:



C. Rodríguez Rodríguez

P. Gryczka

Decision electronically authenticated