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**D E C I S I O N**  
**of 12 July 2004**

**Case Number:** T 1183/01 - 3.3.6

**Application Number:** 91200661.6

**Publication Number:** 0451893

**IPC:** C11D 3/39

**Language of the proceedings:** EN

**Title of invention:**

Particulate bleaching detergent composition

**Patentee:**

UNILEVER N.V., et al

**Opponents:**

The Procter & Gamble Company

Eka Chemicals AB

Henkel KGaA Patente (TTP)

Degussa AG Intellectual Property Management, Standort Wolfgang

Solvay Interlox Ltd.

Kemira Kemi AB

**Headword:**

Bleaching Composition/UNILEVER

**Relevant legal provisions:**

EPC Art. 83, 56

**Keyword:**

"Inventive step - no"

**Decisions cited:**

-

**Catchword:**

-



Case Number: T 1183/01 - 3.3.6

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.6  
of 12 July 2004

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**Decision under appeal:** **Decision of the Opposition Division of the  
European Patent Office posted 28 September 2001  
revoking European patent No. 0451893 pursuant  
to Article 102(1) EPC.**

**Composition of the Board:**

**Chairman:** P. Krasa  
**Members:** G. Dischinger-Höppler  
V. Di Cerbo

## Summary of Facts and Submissions

I. This appeal is from the decision of the Opposition Division to revoke the European patent No. 0 451 893. The decision was based on amended sets of claims according to a main request and an auxiliary request. Independent Claim 1 of the main request reads:

"1. A particulate bleaching detergent composition comprising a zeolite built base powder containing one or more anionic and/or nonionic surfactants and alkalimetal percarbonate particles, characterised in that said alkalimetal percarbonate particles having a morphology index (MI) of less than 0.04, said morphology index being defined as:

$$MI = 0.0448 \times CV + 3.61 \times 10^6/d^3$$

where "CV" is the coefficient of variation of the weight average particle size distribution, and "d" is the weight mean average particle size (in microns), as defined by the following equations:

$$CV = \sigma/d$$

wherein:

$$\sigma^2 = \Sigma(d_i - d)^2 \times w_i/100$$

and

$$d = \Sigma d_i \times w_i/100$$

where  $d_i$  is the average particle size of the  $i^{\text{th}}$  size fraction of a complete distribution of particles, and  $w_i$  is the weight percentage of that fraction; further characterised by the presence of from 20 to 80% by weight of the composition of crystalline aluminosilicate detergency builder and in that the base powder contains more than 20 ppm iron."

Claim 1 of the auxiliary request differs from that only by the lower value of the morphology index (MI) of less than 0.03.

II. Six notices of opposition had been filed against the granted patent, wherein the Opponents sought revocation of the patent on the grounds of Article 100(b) EPC for insufficient disclosure and on the grounds of Article 100(a) EPC for lack of novelty and lack of inventive step (Articles 52(1), 54(2) and 56 EPC) and for the reason that the claims related to a mathematical method (Article 52(2)(a) EPC). The oppositions were based *inter alia* on the following documents

D1 JP-A-64/006097 (English translation) and

D11 JP-A-50-91596 (English translation).

During the opposition proceedings, the Patent Proprietor filed experimental evidence under cover of the letters dated 27 July 1998 and 18 May 2001 and the Opponents filed the following further document

D39 G. Hägg, "Allmän och Oorganisk Kemi", 8<sup>th</sup> Ed.  
Uppsala 1984, pages 376 and 378 including English  
translations of paragraphs 2 and 4 of page 376 and  
of a statement in lines 18 to 20 of page 378.

III. In its decision, the Opposition Division held that the subject-matter of Claim 1 was not based on an inventive step in view of D1 as the closest prior art since it was common general knowledge of those skilled in the art that the stability of particles increases as their diameter increases. This common general knowledge was represented by D39 and further specifically confirmed for sodium percarbonate particles by, *inter alia*, D1 and D11. Therefore, a skilled person, by applying this knowledge would use percarbonate in the form of particles which are as large as possible and avoid any small particles, i.e. select a narrow particle size distribution. The MI index as defined in Claim 1 was merely a restatement of this common general knowledge by a mathematical formula. D1 was not limited with respect to any particular contents of zeolite or metal impurities but taught that the amount of water should not exceed 15 wt% in order to avoid decomposition of the percarbonate. However, the skilled person would apply the above common general knowledge also to compositions containing 20 wt% or more of zeolite, and in the presence of high amounts of iron impurities, which may be contained in the zeolite, since the mechanism behind the decomposition of percarbonate was dependent on the surface area of the percarbonate particles and the same for any particular composition irrespective of the specific amounts of water and iron contained. It was therefore apparent for someone skilled in the art that the reactivity of percarbonate

was reduced if its surface area was reduced even in the presence of high amounts of water and iron.

IV. This decision was appealed by the Patent Proprietor (hereinafter Appellant) who filed clean copies of its main and auxiliary requests upon which the appealed decision is based. Four Opponents (hereinafter Respondents) filed submissions in reply.

V. The Appellant submitted in writing the following arguments:

- The subject-matter of Claim 1 differed from the composition disclosed in D1 in that the zeolite was present in the composition in an amount of 20 to 80 wt%, in that iron was present in the base powder in an amount of more than 20 ppm and in the feature concerning the morphology index (MI).
- D1 was also concerned with the stability of sodium percarbonate. However, it was known in the art that high amounts of zeolite and iron would lead to instability problems. Taking D1 as the closest prior art, the technical problem to be solved consisted, therefore, in providing stable particulate bleaching detergent compositions comprising surfactant and percarbonate in the presence of large amounts of 20 to 80% of zeolite and substantial levels of impurity in the form of more than 20 ppm iron.
- This problem was solved by the subject-matter of Claim 1, in particular by the general rule given therein as MI by which one could balance the

particle size distribution in conjunction with the average particle size as the important factors necessary for providing stable percarbonate formulations.

- D1 did not make any reference to the particle size distribution but simply indicated that the stability of the percarbonate in the composition increased with increasing particle size, i.e. with decreasing surface area of the percarbonate particles.
  
- It was well known in the art that percarbonate particles were unstable in the presence of moisture and that this effect could be minimised by reducing the surface area of the percarbonate in the composition.
  
- However, the inventors of the patent in suit had found that the surface area was not sufficient to explain the well known phenomenon that percarbonate particles decomposed more readily in the presence of zeolite or iron. In particular, it was found that the parameter which was critical with respect to percarbonate decomposition was the number of points of contact of the percarbonate particles with particles of the detergent composition which was not simply a function of the particle size or surface area but a function of the distribution of the particles within the system as described by the mathematical equation for the MI given in Claim 1.



- It had been demonstrated in the Appellant's experimental evidence that there was an unexpected step change in percarbonate stability when the MI of the percarbonate was less than 0.04, preferably less than 0.03.
- The prior art did not contain any hint that the problem of percarbonate instability in the presence of high amounts of zeolite and, in particular, iron could be overcome by using particles having a MI as required by Claim 1.

VI. The Respondents submitted the following arguments:

- The claimed subject-matter was insufficiently disclosed with respect to the feature concerning the MI since the value obtained for the MI was dependent on the fractions of particles and, in particular on the number of fractions to be considered, for which the patent in suit did not provide any reasonable disclosure.
- The subject-matter claimed in the main request was not novel in view of a public prior use for which evidence was submitted during opposition proceedings.
- The claimed subject-matter was not inventive in view of D1 as the closest prior art, in particular since the presence of particular amounts of zeolite and iron did not provide any unexpected effect and since the parameter MI did not provide a reliable basis for estimating the number of points of contact between the percarbonate

particles with other components of the composition. Instead, the requirement in the claims of both requests that the MI has to be low simply meant that both terms contained in the formula had to be low which implied that the size of the percarbonate particles had to be large and the particle size distribution small.

- This was, however, known from the prior art according to which the stability of percarbonate particles was increased with increasing particle size and by the elimination of fines.
- For the same reasons, the claimed process was not inventive in view of D11.

VII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request, or alternatively on the basis of the auxiliary request.

The Respondents requested that the appeal be dismissed.

VIII. Upon requests made by the Appellant and three Respondents, oral proceedings before the Board of Appeal were scheduled for 7 and 8 April 2004. Under cover of its letter dated 18 February 2004, the Appellant informed the Board that it would not be represented at these oral proceedings. The Board informed the parties by telefax sent on 31 March 2004 that the oral proceedings were cancelled.

## Reasons for the Decision

### *Main Request*

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

The Board is satisfied that no problems under Article 84 EPC have been introduced by the amendments made and that the claims comply with the requirements of Article 123(2) and (3) EPC. No objections have been raised by the Respondents in this respect.

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

2.1 The Respondents argued that the invention was insufficiently disclosed since the skilled person was unable to determine the percarbonate particles which fulfil the requirement of a MI of less than 0.04 since the MI was a new parameter, unknown in the art, but dependent on factors which were undefined in the patent in suit.

2.2 According to the formula " $MI = 0.0448 \times CV + 3.61 \times 10^6/d^3$ ", the value for MI corresponds to the sum of two mathematical terms (see Claim 1). The first one " $0.0448 \times CV$ " represents the coefficient of variation "CV" of the weight average particle size distribution of the sample considered, wherein  $CV = \sigma/d$ ,  $\sigma^2 = \Sigma(d_i - d)^2 \times w_i/100$  and  $d = \Sigma d_i \times w_i/100$  with "d" being the weight mean average particle size (in microns),  $d_i$  being the average particle size of the  $i^{th}$  size fraction of a complete distribution of particles, and  $w_i$  being the weight percentage of that fraction. The second terminus in the formula " $3.61 \times 10^6/d^3$ " simply indicates how MI

is influenced by the average particle size  $d$  of the sample.

- 2.3 Specific values for  $d$  and CV of the percarbonate used are given in the examples of the patent in suit. However, the patent in suit does not contain any information on how these values for the CV were obtained. In particular, it is not indicated which values for the parameters  $i$ ,  $d_i$  and  $w_i$  are chosen to obtain those coefficients of variations CV or the respective MI. Particular values for these parameters are not disclosed in the patent in suit.

Nor is the claimed subject-matter restricted to a particular CV, let alone to any particular number of fractions  $i$ , average particle size  $d_i$  within such fraction or weight percentage  $w_i$  thereof.

The only information in respect of how to identify the MI is given on page 4 of the patent in suit where it is stated that the percarbonate having the desired MI may be obtained by preparing various sieve fractions of a material having an unknown MI, preferably 5 ranges of about 100 microns or less, and calculating thereafter the MI of each fraction (lines 29 to 32).

In the Board's opinion, this information tells those skilled in the art simply that fractions having an average particle size suitable to fulfil the requirement  $MI > 0.04$  can be found by sieving the percarbonate material and calculating for the several fractions the MI values according to the mathematical formula. Accordingly, single fractions are considered where  $d_i = d$ .

In this case the MI value corresponds to the term " $3.61 \times 10^6/d^3$ " since it is apparent from the mathematical formula, that for  $d_i = d$ , the first part of the formula, i.e. the term " $0.0448 \times CV$ " is zero. Thus, the MI can be easily calculated for samples considered as single fractions.

Insofar, the Board is convinced that, considering the disclosure of the patent in suit, those skilled in the art are able to put the invention into practice.

- 2.4 If, however, the same sample of particles (having the same average particle size  $d$ ) is calculated as two or more fractions, the MI obtained from the term " $3.61 \times 10^6/d^3$ " is increased by a value obtained for the term " $0.0448 \times CV = 0.0448 \times \sigma/d$ ", wherein  $\sigma$  is defined via the equation  $\sigma^2 = \Sigma(d_i - d)^2 \times w_i/100$  and  $d$  is defined via the equation  $d = \Sigma d_i \times w_i/100$ . It follows that for a given average particle size, the lowest MI is obtained for  $d_i = d$  and that in this case the MI is dependent on the average particle size  $d$  only and decreases as  $d$  increases. It further follows that for more than one fraction the MI depends on the value of the coefficient of variation  $CV$  which is dependent on the number  $i$  of fractions, mean particle size  $d_i$  and weight percentage  $w_i$  of the fractions.

- 2.5 The dependency of the MI from the number  $i$  of fractions considered and from the particle size limits within these fractions has convincingly been shown by the Respondents. In particular, it has been shown that one and the same sample of percarbonate particles may fulfil the requirement of  $MI < 0.04$  if calculated as

one fraction ( $d_i = d$ ) or not fulfil that requirement if calculated as two fractions. The Board, therefore, agrees with the Respondents that the mathematical formula given in Claim 1 implies that the value obtained for the MI depends on the number and sizes of sieves used for fractionating or respectively on the method of calculation, but not necessarily on the actual particle size distribution. Nevertheless, a skilled person, by choosing the number and sizes of sieves would be able to carry out the invention.

2.6 However, the Board concludes from the above considerations, that it is apparent for those skilled in the art that the first term in the mathematical formula " $0.0448 \times \sigma/d$ " is meaningless without any limitation in respect of the parameters  $i$ ,  $d_i$  and  $w_i$  (see above 2.4) and that the only meaningful embodiment covered by Claim 1 and disclosed in the patent in suit is given for  $d_i = d$ , where MI corresponds to " $3.61 \times 106/d^3$ " (see above 2.3). Consequently, the absence of such limitations renders the scope of the claims unclear. This is, however, not a ground of opposition.

### 3. *Novelty*

Lack of novelty has been objected to in view of evidence in relation to a public prior use submitted during opposition proceedings. This ground of opposition has not been considered in the decision under appeal and is not linked to the reasons given with respect to inventive step, the only issue dealt with in the decision. However, as will be seen below, the appeal also fails for lack of inventive step. Hence, there is no reason to pursue the novelty issue

irrespective of any remaining doubts as to whether or not the subject-matter was anticipated by a prior use.

4. *Inventive Step*

4.1 The patent in suit relates to particulate bleaching detergent compositions containing sodium percarbonate as the bleaching agent (page 2, lines 5 to 7). Such compositions are said to have been known in the art. However, it is stated that there still remains the problem of providing stable compositions on the basis of zeolite built detergent powder due to the large amounts of water contained in the zeolite which decompose the percarbonate (page 2, lines 17 to 18 and lines 33 to 41). According to the patent in suit, it was surprising that this problem was solved by using a percarbonate material of specified morphology, in spite of the high amounts of iron in the zeolite built base powders (page 2, lines 40 to 41, page 4, lines 33 to 36).

4.2 For the assessment of inventive step, the Appellant and most of the Respondents use D1 as the starting point. This document is also concerned with a particulate bleaching detergent composition containing anionic and nonionic surfactants, percarbonate as the bleaching agent and zeolite as a builder material. D1 is further concerned with the same technical problem as the patent in suit, namely improving the stability of the percarbonate upon storage within the detergent composition (page 2, lines 3 to 20, page 8, lines 13 to 20, examples and claim).

Alternatively, D11 was chosen by Respondent V (Opponent V). This document relates to a method of granulation of sodium percarbonate in the presence of a peroxides containing binder at low temperature to a particle size of 700  $\mu\text{m}$  or more in order to obtain a product of high stability upon storage at low loss of effective oxygen during granulation (page 2, lines 8 to 14 and claim).

D11 is not concerned with detergent compositions or zeolite containing compositions and, therefore, less qualified than D1 as a starting point for the assessment of inventive step.

4.3 In order to improve the stability of the percarbonate within the detergent compositions, D1 suggests adding alcohol ethoxylate (nonionic surfactant) in an amount of 2 to 15 wt%. It is said that this prevents decomposition of the percarbonate in the presence of water (page 5, last two lines to page 6, line 19). However, the claimed subject-matter does not differ in this respect since, according to the patent in suit, it is preferred that the compositions contain the same nonionics in the same amounts (page 3, lines 26 to 28 and page 5, table).

4.4 According to the Appellant, D1 did not disclose the morphology index MI.

However, D1 teaches that it is important to control the average particle size of the percarbonate within the range of 250 to 2000  $\mu\text{m}$  since below that range the stability would be worsened whereas the solubility would suffer if the particle size was above 2000  $\mu\text{m}$ . It is shown in Examples 1 to 4 and comparative Example 7



of Table 1 (page 12) that within a range of between 150 to 2000  $\mu\text{m}$  average particle size of the percarbonate the stability increases as the particle size increases.

From the particular average particle sizes given in D1 for Examples 1 to 4 (i.e. 500  $\mu\text{m}$ , 1000  $\mu\text{m}$ , 1500  $\mu\text{m}$  and 2000  $\mu\text{m}$ , respectively) an MI of 0.0289, 0.0036, 0.0011 and 0.0005, i.e. less than 0.04, can be calculated according to the formula in Claim 1 of the patent in suit for  $i = 1$ . In contrast, the MI in comparative Example 7 (150  $\mu\text{m}$ ) is 1.070. Therefore, the requirement of an MI of less than 0.04 is implicitly disclosed in D1 and cannot be considered as a distinguishing feature over Examples 1 to 4 of D1.

- 4.5 The Board agrees with the Appellant insofar as D1 does not disclose the amounts of zeolite (20 to 80 wt% of the composition) and iron (> 20 ppm in the base powder) required in Claim 1 of the patent in suit. Whilst iron is not mentioned at all in D1, a particular amount of 15 wt% of zeolite is used in the compositions of all examples (page 12, Table 1).
- 4.6 The Appellant argued that it was well-known in the art that percarbonate particles decompose more easily if zeolite and iron were present. The problem to be solved in view of D1 was, therefore, to provide a stable composition even in the presence of high amounts of zeolite and iron.
- 4.7 Examples are given in the patent in suit where it is shown that in a composition containing 44.8 wt% of zeolite in the base powder of the composition, wherein the base powder constitutes 87.5 wt% of the composition,

decomposition of the sodium percarbonate decreases as the MI decreases (page 5, lines 4 to 57 and page 6, Table 1).

The Appellant filed further experimental evidence during the opposition proceedings (see II. above) where the same is shown for a composition containing 42.6 wt% zeolite in the base powder.

Neither the examples nor the experiments indicate the amount of iron contained in the compositions. Nor do they show any effects resulting from the presence of iron or from the higher amounts of zeolite as compared with D1 since they are not comparable with those of D1 (Table 1) as far as the compositions are concerned. They are, further, not comparable with respect to the decomposition rate since the measuring conditions for determining that rate are different or respectively not even precisely defined (see examples of the patent in suit, where the temperature and relative humidity during storage is not given).

Therefore, the above effects of improved percarbonate stability at decreased MI only apply for the particular compositions and for those amounts of iron brought along by the specific kind and amounts of zeolite used.

- 4.8 However, the Board accepts the Appellant's argument that zeolite, in particular due to its water content (patent in suit, page 2, lines 33 to 34), and iron contribute to percarbonate destabilisation and that iron is an impurity in natural zeolites. Assuming further, in the Appellant's favour, that the zeolite used in the examples and experiments contains in fact

high amounts of iron impurities, the technical problem actually solved in view of D1 may be seen in an improvement of the stability of percarbonate, or respectively in a minimized decomposition, even in the presence of large amounts, e.g. 44.8 or 42.6 wt%, of the iron-containing zeolite.

4.9 It remains to be decided whether, in view of the available prior art documents, it was obvious for someone skilled in the art to solve this technical problem by the means claimed, namely by using the same percarbonate particles as in D1, i.e. those having a MI of less than 0.04.

4.10 From the mathematical formula in Claim 1 of the patent in suit it follows that in the case of  $i = 1$ , MI and the average particle size  $d$  are interrelated via the equation  $MI = 3.61 \times 10^6 / d^3$  (see above 2.3). Therefore, the following correlation between average particle size, decomposition rate and MI for  $i = 1$  exists for the particular compositions of Examples 1 to 4 and comparative Example 7 of D1:

Av. Part.					
size ( $\mu\text{m}$ )	500	1000	1500	2000	150
Decompos.					
Rate (%)	30	25	20	17	85
MI					
( $i = 1$ )	0.0289	0.0036	0.0011	0.0005	1.070

D1 shows, therefore, that the percarbonate stability is sufficient if the average particle size of the percarbonate is 500  $\mu\text{m}$  and increases as the average particle size increases, or respectively if the MI is

0.0289 and increases as the MI decreases (see also 4.5 above).

- 4.11 The Appellant has not denied that reduction of the surface area of the percarbonate in a composition was known as one way of minimising its instability in the presence of water. However, the Appellant argued that the concept of the invention was based on the finding that decomposition of the percarbonate can be reduced in the presence of zeolite and iron by controlling the MI and that this concept was hitherto unknown in the prior art. The MI was not merely a restatement by a mathematical formula of the principles known in the art, but a means for describing the distribution of the percarbonate particles within the composition and hence suitable for controlling the number of points of contact of the percarbonate particles with particles of the detergent composition.
- 4.12 This argument is not acceptable since, as convincingly put forward by the Respondents and not disputed by the Appellant, it obviously does not take into account that the number of points of contact of the percarbonate particles with particles of the detergent composition not only depends on the size and size distribution of the percarbonate particles but also on their morphology (form, kind of surface and porosity) and, in particular, on the nature of all the other particles present in the composition for which no information is given in the patent in suit.
- 4.13 The Board agrees with the Opposition Division that the required low value for MI simply means for those skilled in the art selecting large particles and

avoiding or removing small particles. It is, however, evident from the examples of D1 that large particles of percarbonate improve the stability even in the presence of 15 wt% of zeolite. It is further agreed that this finding is in line with the common general knowledge of a skilled person as reflected in D39 where it reads in the second paragraph of page 376:

"The increase in surface energy per unit area as the particle size decreases makes a smaller particle react more easily than a larger one. Starting from a certain mass of a solid phase, its total contact surface with other phases can, in fact, be increased significantly as the particle size decreases".

It is appreciated that the presence of water is usually an important factor where chemical reactivity is concerned. However, the above statement is not restricted to reactions with water but extends to reactivity in general, e.g. in the presence of catalysts. This is evident from the second paragraph on page 378 of D39, where it reads:

"... it is evident that a large contact surface and, thus, high dispersing of the catalyst are important to this capability of reacting large amounts of substance per unit of time ...".

According to common general knowledge it is, therefore, a principle that the particle size influences the reactivity and that particle stability increases with its size or, in other words, as the surface area per unit volume of the particles decreases, irrespective of the specific reactants contained in a reaction mixture.

Thus, it is irrelevant in respect of this principle whether a given composition contains more or less amounts of a particular reactant.

- 4.14 The Appellant argued that the experimental evidence filed during opposition proceedings demonstrated an unexpected step change in percarbonate stability when the percarbonate has a MI of less than 0.04, in particular less than 0.03. It contested the statement of the Opposition Division, that D1 also showed a step change in the same sense as in the patent in suit.
- 4.15 However, the Respondents have convincingly demonstrated that no step change in percarbonate stability in relation to the MI exists for the examples in the patent in suit. They have further shown by calculations on the basis of the examples of D1 that the particle stability is directly correlated with the surface area but not with the MI (see letter of Respondent IV dated 19 February 2004, point 2.2). Since the Appellant has not provided any counter-evidence, the presence of an unexpected step change cannot be taken into account here.
- 4.16 From the above considerations, the Board concludes that - in view of D1 - it was obvious for a person skilled in the art to use percarbonate of large average particle size, in particular of 500  $\mu\text{m}$  or more, as suggested in the examples of D1 and which corresponds to an MI of less than 0.04, in order to minimize percarbonate decomposition even in the presence of large amounts of zeolite and iron.

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

5. *Auxiliary request*

The only distinguishing feature in Claim 1 with respect to Claim 1 of the main request consists in that the value of MI has to be less than 0.03 instead of 0.04. Since this value is also covered by D1, the above reasons also apply to Claim 1 of the auxiliary request.

Therefore, the Appellant's auxiliary request must also fail since it too does not meet the requirements of Articles 52(1) and 56 EPC.

6. *Procedural matters*

Oral proceedings have been cancelled by the Board following notification that the Appellant would not be represented. The Board has not overlooked the unconditioned request for oral proceedings filed by one of the Respondents (Opponent IV). Since, however, the present decision is in favour of this party and meets its request for dismissal of the appeal, its right to be heard (Article 113(1) EPC) is not violated by cancelling the oral proceedings.

**Order**

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

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P. Krasa