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(71) **Applicant: SOLVAY SA** [BE/BE]; 310, rue de Ransbeek, 1120 BRUSSELS (BE).

(72) **Inventors: LANCASTER, Mitchell**; c/o CYTEC INDUSTRIES (US), 1937 WEST MAIN STREET, STAMFORD, Connecticut 069002 (US). **JAMES, Alun Pryce**; Rue de Ransbeek 310, 1120 Brussels (BE). **VASQUEZ, Oscar**; 20-22 McPherson Street, Banksmeadow, New South Wales 2019 (AU).

(74) **Agent: KRAUS & LEDERER PARTGMBB**; Thomas-Wimmer-Ring 15, 80539 München (DE).

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(54) **Title:** SEPARATION OF CU AND NI FROM ORE USING H_2O_2

(57) **Abstract:** The present invention relates to a process for extracting Ni and Cu from an ore comprising Ni and Cu and separating Ni from Cu, the process comprising the steps of: a) conditioning the ore to provide a conditioned ore, b) subjecting the conditioned ore to froth flotation to provide a concentrate and an underflow, and c) recovering Cu in the concentrate and Ni in the underflow, wherein conditioning the ore is conducted in the presence of H_2O_2 at a pH value of above 7.0, and H_2O_2 is used in an amount of 50 g to 500 g H_2O_2 per 1t of the ore.

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Separation of Cu and Ni from ore using H_2O_2

TECHNICAL FIELD

The present relates to a process for extracting Ni and Cu from an ore comprising Ni and Cu and separating Ni from Cu.

TECHNICAL BACKGROUND

Ores containing Ni and Cu are of economic importance. Such Ni-and Cu-containing ores can be subjected to froth flotation to recover the desired mineral and discard any gangue minerals.

Usually, this process comprises aeration of a dispersion of the ground ore to form a froth on the surface. The froth contains hydrophobic particles whereas the underflow contains hydrophilic particles. The froth is then separated from the underflow to produce a purified concentrate. This process and the relevant reagents are described in Chapter 16 "*Flotation of Nickel and Nickel-Copper Ores*" in the "*Handbook of Flotation Reagents: Chemistry, Theory and Practice*", vol 2, 2007, Elsevier B.V. by S. M. Bulatovic.

Many of the flotation reagents, such as cyanides and polyamines, are, however, environmentally harmful and potentially toxic for aquatic organisms. In addition, due to the large scale of operations, even small improvements in the extraction and separation of Ni and Cu from such ores have a large overall impact. Therefore, it is an object of the present invention to provide a process for extracting Ni and Cu from an ore comprising Ni and Cu and separating Ni from Cu that is environmentally-friendly and sustainable. In addition, it is an object of the present invention to provide a process that increases the recovery of Ni and/or Cu from an ore comprising Ni and Cu. Furthermore, it is an object of the present invention to provide a process that increases the separation of Ni from Cu.

SUMMARY OF THE INVENTION

These objects have been solved by a process for extracting Ni and Cu from an ore comprising Ni and Cu and separating Ni from Cu, the process comprising the steps of:

a) conditioning the ore to provide a conditioned ore,

b) subjecting the conditioned ore to froth flotation to provide a concentrate and an underflow, and

c) recovering Cu in the concentrate and Ni in the underflow,

wherein

conditioning the ore is conducted in the presence of H_2O_2 at a pH value of above 7.0, and

H_2O_2 is used in an amount of 50 g to 500 g H_2O_2 per 1t of the ore.

It has been found that the above process using H_2O_2 is more environmentally-friendly than the processes of the prior art. It has been found that the process of the present invention results in improved extraction of Ni and Cu and improved separation of Ni from Cu.

The present invention further relates to the use of H_2O_2 in a conditioning step for extracting Ni and Cu from an ore and separating Ni from Cu by froth flotation.

DETAILED DESCRIPTION OF THE INVENTION

The following definitions are relevant in connection with the embodiments of the present invention.

The meaning of the term “comprising” is to be interpreted as encompassing all the specifically mentioned features as well optional, additional, unspecified ones, whereas the term “consisting of” only includes those features as specified. Therefore, “comprising” includes as a limiting case the composition specified by “consisting of”.

The term “wt.-%” refers to the amount of the respective component by weight based on the total amount of the composition, unless noted otherwise.

As used herein, the singular forms “a”, “an”, and “the” include both singular and plural referents unless the context clearly dictates otherwise. By way of example, “a depressant” means one depressant or more than one depressant.

The term “ore” refers to a naturally occurring mineral from which a metal and other elements can be extracted. Metals are commonly present as oxides, aresenides, sulfides, sulfates or silicates.

The term “conditioning” refers to treating a ground ore with reagents, such as collectors, frothers, froth phase modifiers, dispersants, depressants, suppressants, pH regulators, and activators for a certain time period before subjecting the conditioned ore to froth flotation. Conditioning is applied to increase yield and/or ease of separation during froth flotation.

The term “froth flotation” refers to a method for separating minerals from a dispersion of ground ore by applying a gas, usually air, through the dispersion to form a froth or float on the surface. The float contains hydrophobic particles whereas the underflow contains hydrophilic particles. The float can also synonymously be called “froth” or “concentrate” and the underflow can also synonymously be called “tails” or “tailings”. The concentrate refers to material produced after removal of gangue minerals, leaving minerals of value in the concentrate. Froth flotation can be carried out using equipment and procedures known in the art. Froth flotation can be carried out using equipment and procedures known in the art, such as a Denver, Galigher, Wemco, Outokumpo, Sala, or a Jameson cell. When the application of gas is started, the conditioning step ends and the froth flotation step starts. In other words, the conditioning step takes place before the gas is starting to be applied or until the gas is started to be applied. And the froth flotation step starts as soon as the gas is started to be applied.

The expression “ore comprising Ni and Cu” refers to an ore comprising minerals containing Ni and Cu ions, e.g., nickel sulfides and copper sulfides. Such an ore may comprise as nickel containing mineral violarite, millerite, lattice substituted/doped pyrrhotite and/or pentlandite. Such an ore may comprise as copper containing mineral chalcopyrite, chalcocite, and/or bornite.

The term “collector” refers to a compound that increases the amount of desired mineral that is formed in the froth, i.e., a compound that renders the minerals water-repellents and increase attachment of the desired mineral to the air bubbles during froth flotation. Alkyl xanthate salts and dialkyl xanthogen formate are widely used collectors, in particular for Cu.

The term “frother” as used herein refers to a compound that increases the amount and/or stability of the froth that is formed during froth flotation. Frothers are surface-active compounds that commonly contain a polar group and a hydrocarbon chain. Non-limiting examples of frothers include phenols, alkylaryl sulfonates, aliphatic alcohols, alkoxy paraffins, , and polyglycol ethers. Different flotation reagents are disclosed in the “*Handbook of Flotation Reagents: Chemistry, Theory and Practice*”, vol 2, 2007, Elsevier B.V. by S. M. Bulatovic.

The term “froth phase modifier” as used herein refers to a compound that controls the interaction of collectors between specific minerals. These compounds show an activating or depressing action in flotation. Thus, the term “froth phase modifier” is a generic term that may encompass depressants, pH regulators and activators.

The term “dispersant” refers to compounds, usually polymers, that either increase the stability of the ore in the slurry or that prevent the formation of slimes during flotation.

The term “depressant” refers to a compound that reduces the amount of specific minerals that attach to air bubbles during froth flotation. In particular, depressants selectively inhibit the interaction of a specific mineral with the collector. As the depressant is selective for certain minerals and not others, it is preferred that the depressant is a depressant for gangue minerals and/or a Ni depressant.

The term “pH regulator” refers to a compound that regulates the pH value. Nonlimiting examples are inorganic acids, such as aqueous HCl and inorganic bases, such as lime. It is to be understood that a reagent may have more than one function, i.e. lime can be a pH regulator and a froth phase modifier.

Preferred embodiments according to the invention are defined hereinafter. It is to be understood that the following embodiments refer to all aspects of the present invention, which includes the process for extracting and separating as well as the use of H_2O_2 in a conditioning step.

In an embodiment, the present invention relates to a process for extracting Ni and Cu from an ore comprising Ni and Cu and separating Ni from Cu, the process comprising the steps of:

a) conditioning the ore to provide a conditioned ore,

b) subjecting the conditioned ore to froth flotation to provide a concentrate and an underflow, and

c) recovering Cu in the concentrate and Ni in the underflow,

wherein

conditioning the ore is conducted in the presence of H_2O_2 at a pH value of above 7.0, and

H_2O_2 is used in an amount of 50 g to 500 g H_2O_2 per 1t of the ore.

It is to be understood that the term “for extracting Ni and Cu” encompasses partial extraction of Ni and Cu. Likewise the term “for separating Ni from Cu” encompasses partial separation of Ni from Cu. Put differently, Ni and Cu do not need to be separated completely from each other and the Ni to Cu wt.-ratio in the concentrate is different than in the ore. In an embodiment, the Ni to Cu wt.-ratio in the concentrate is lower than the Ni to Cu wt.-ratio in the ore. Thus, the concentrate can be enriched with Cu. In an embodiment, the Ni to Cu wt.-ratio is larger in the underflow than the Ni to Cu wt.-ratio in the ore.

The term “recovering” can comprise further process steps, such as drying and/or a further froth flotation. In particular for recovering Ni from the underflow, it is preferred to subject the underflow to a second froth flotation step, preferably in the presence of a Ni collector, to provide a second concentrate and a second underflow and recovering the Ni from the second concentrate.

The pH value during the froth flotation process is important, yet plays a complex role. In an embodiment, the conditioning is conducted at a pH value of 8.0 to 13.0, preferably at a pH value of 9.0 to 11.0. It is preferred that the conditioning is conducted at a uniform pH value. It is further preferred that the pH value is adjusted before the ore is conditioned. Without being bound by theory, it is believed under basic pH, the H_2O_2 will alter the surface structure of the Ni-containing minerals while not altering the surface structure of Cu-containing minerals, thus making the Ni-containing minerals more hydrophilic and increasing the amount of Ni-containing minerals in the underflow.

In the process of the invention, the pH is measured by a glass pH electrode placed into the slurry present during conditioning, preferably following the grind of the ore. The probe is calibrated with commercially available, certified buffer solutions.

In an embodiment, a flotation reagent selected from collectors, frothers, froth phase modifiers, dispersants, depressants, suppressants, pH regulators, activators, and two or more thereof is added during the process. It is preferred that at least a collector, a frother, and a depressant are added during the process. In particular, said reagents are added during the conditioning of the ore. In an embodiment, during the conditioning step of the ore, first a collector is added, followed by frother, followed by a depressant.

In a preferred embodiment, a Cu collector selected from metal alkyl xanthates, preferably potassium amyl xanthate, phosphonic acids, phosphoric acid esters, carboxylic acids and salts thereof, metal alkyl sulfates, metal alkyl sulfonates, dialkyl or diaryl dithiophosphoric acids and salts thereof, mercaptobenzothiazole, xanthogen formates, alkyl ethoxycarbonyl thioureas, thionocarbamates, tertiary dodecyl mercaptans, and mixtures of two or more thereof is added during conditioning of the ore. It is preferred that the Cu collector comprises a metal alkyl xanthate. The term metal preferably refers to alkali metals in this context, such as sodium and potassium. Non-limiting examples of metal alkyl xanthates are sodium isopropyl xanthate, potassium isopropyl xanthate, sodium ethyl xanthate, potassium ethyl xanthate, sodium isobutyl xanthate, potassium isobutyl xanthate, sodium amyl xanthate and potassium amyl xanthate. It is particularly preferred that potassium amyl xanthate is added as a Cu collector during the process.

In an embodiment, the concentrate contains at least 84 % Cu based on the amount of Cu in the ore. As a non-limiting example, if a certain amount of ore contains 10 g Cu, the concentrate contains at least 8.4 g Cu. The amount of Cu can preferably be determined by wavelength-dispersive XRF (x-ray fluorescence) measurements, for example measured by an Epsilon 1 benchtop XRF analyzer from Malvern Panalytical. Alternatively, the amount of Cu can be determined by ICP-OES (inductively coupled plasma optical emission spectrometry) or SIMS (secondary ion mass spectrometry) analysis.

In an embodiment, the concentrate contains not more than 60 %, not more than 50 % or not more than 45 % Ni based on the amount of Ni in the ore. In an embodiment, the concentrate has a Cu to Ni selectivity of at least 30 % at least 35 % or at least 40 %. The selectivity “S” of the concentrate is defined as follows:

S = (amount of Cu in the concentrate based on the amount of Cu in the ore) – (amount of Ni in the concentrate based on the amount of Ni in the ore) = (Cu recovery in the concentrate) – (Ni recovery in the concentrate).

In an embodiment, the ore comprises 0.01 to 10.0 wt.-%, 0.05 to 8.0 wt.-% or 0.1 to 5.0 wt.-% Cu. In an embodiment, the ore comprises 0.01 to 10.0 wt.-%, 0.05 to 8.0 wt.-% or 0.1 to 5.0 wt.-% Ni.

In a preferred embodiment, the ore comprises 0.10 to 2.0 wt.-% Cu and 0.10 to 2.0 wt.-% Ni. It has surprisingly been found that for ores having such a low Cu and Ni content, the Cu and Ni can efficiently be extracted and the minerals containing Cu and Ni can be separated.

The type of ore is not particularly limited as long as it contains Ni and Cu. In an embodiment, the ore comprises nickel sulfides, iron sulfides and/or copper sulfides, preferably pyrrhotite, chalcopyrite and/or pentlandite, more preferably the ore comprises pentlandite as Ni-containing mineral and chalcopyrite as Cu-containing mineral. Thus, in an embodiment, the invention relates to a process for extracting a nickel sulfide and a copper sulfide from an ore comprising a nickel sulfide and a copper sulfide and separating the nickel sulfide from the copper sulfide, the process comprising the steps of:

a) conditioning the ore to provide a conditioned ore,

b) subjecting the conditioned ore to froth flotation to provide a concentrate and an underflow, and

c) recovering copper sulfide in the concentrate and nickel sulfide in the underflow,

wherein

conditioning the ore is conducted in the presence of H_2O_2 at a pH value of above 7.0, and

H_2O_2 is used in an amount of 50 g to 500 g H_2O_2 per 1t of the ore.

In an embodiment, a depressant selected from calcium carbonate, sodium chloride, sodium cyanide, sodium silicate, sodium sulfite, sodium metabisulfite, triethylenetetramine, diethylenetriamine, starch, guar, dextrins, carboxymethyl cellulose, and mixtures of two or more thereof is added during conditioning of the ore. In a preferred embodiment, carboxymethyl cellulose is added as a depressant.

In an embodiment, the H_2O_2 is added as a 10 wt.-% to 80 wt.-% H_2O_2 aqueous solution. It is preferred to use the H_2O_2 in concentrated aqueous solution, e.g. as 50 wt.-% aqueous solution. Such concentrates may comprise stabilizers.

In an embodiment, H_2O_2 is used in an amount of 60 g to 400 g H_2O_2 per 1t of the ore, preferably in an amount of 70 g to 300 g H_2O_2 per 1t of the ore, more preferably in an amount of 80 g to 200 g H_2O_2 per 1t of the ore. In a particularly preferred embodiment, the H_2O_2 is added in an amount of 90 to 150 g H_2O_2 per 1t of the ore.

In an embodiment, the ore is provided with a particle size P_{80} of 10 µm to 300 µm, preferably by grinding the ore before conditioning the ore. A ground dispersion of the ore is also referred to as pulp. In a preferred embodiment, the ore is provided with a particle size P_{80} of 50 µm to 200 µm. The particle size may be determined by sieving, using a set of screens or meshes, e.g., a vibratory sieve shaker, to separate the various particles by their respective size. The P_{80} particle size refers to a weight-based particle size distribution. In an embodiment, the particle size is determined by sieve analysis, e.g., according to ASTM C136/C136M-19. Grinding can be performed by any method known in the art, such as ball or rod milling. Providing the ore with the above-mentioned particle size increases the surfaces area of the ore, thus facilitating the conditioning process. In addition, this particle size range facilitates the froth flotation process.

In an embodiment, the conditioned ore is subjected to froth flotation for at least 5 minutes, at least 10 minutes, or at least 25 minutes. During froth flotation, the concentrate may be separated continuously or in a batch-wise fashion from the underflow.

In a preferred embodiment, no H_2O_2 is added during the froth flotation. Thus, it is preferred the H_2O_2 is added during the conditioning step only and the ore is then conditioned in the presence of H_2O_2.

The invention further relates to the use of H_2O_2 in a conditioning step for extracting Ni and Cu from an ore and separating Ni from Cu by froth flotation.

Examples

In the examples, pH measurements were taken on a Sensorex S200C pH probe interfaced to a ThermoFisher Orion DualStar pH meter. The pH meter was calibrated daily using pH 4,7,and 10 buffers purchased from ThermoFisher. Calibration was completed daily prior to any flotation testing.

Ni and Cu containing ore was provided comprising the minerals chalcopyrite, pentlandite and pyrrhotite. The ore contained 0.31 wt.-% Cu, 0.23 wt.-% Ni, 8.89 wt.-% Fe, 1.76 wt.-% S and 19.4 wt.-% MgO.

1 kg of the dry ore was added to a rod mill with mild steel grinding rods and ground at 67% solids. Lime was used to adjust the pH to 10. The ore was ground for 45 min to obtain ground ore with a P_{80} of 74 µm, before being transferred to a Denver cell and filled with tap water to produce a slurry containing 30-40 wt.-% ground ore.

5 g/t ore of a Cu collector (potassium amyl xanthate) was added to the slurry being agitated at 1100 rpm and the mixture was stirred for 1 min. Subsequently 20 g/t ore of a frother (alcohol and glycol-based frother; OREPREP®) was added and the slurry was agitated for an additional 1 min at 1100 rpm. Subsequently, a depressant (carboxymethyl cellulose) was added and the slurry was agitated for an additional 3 min at 1100 rpm.

Subsequently, H_2O_2 was added (Example 1: 100 g / 1t ore; Comparative Example 1: 0 g / 1t ore) as a 50 wt.-% aqueous solution and the slurry was stirred for 5 min to obtain a conditioned ore. The H_2O_2 addition amount was controlled through injecting a known volume of solution into the slurry through a syringe.

pH and ORP could also be used to monitor H_2O_2 addition. H_2O_2 addition only occurred prior to flotation.

Air was used as the flotation gas and concentrates were collected at intervals of 5, 10, 17.5, and 25 min (cumulative). Additional frother was added following each concentrate collection with the air turned off and conditioned for 1 min. The froth was scraped from the top of the cell every 15 sec. All concentrates (i.e. float) and tailings (i.e. underflow) were filtered dried and assayed. Additionally, to determine the Ni recovery from the underflow, the underflow was subjected to an additional froth flotation using a 50 g/ 1t of Ni collector (sodium isobutyl xanthate), additional frother (alcohol and glycol-based frother; OREPREP®) and additional depressant (carboxymethyl cellulose) and the Ni recovery from said second froth flotation was determined. The Cu and Ni content were measured using an ICP-OESanalyzer. For the ICP-OES analysis, the metals were dissolved by acid digestion and reference samples with a predetermined concentration of metal ions were used for calibration. The results (recovery) are based on the total amount of Ni (or Cu respectively) in the ore and are summarized in the Table below.

Table 1: Cu and Ni recovery

	Example 1 (100 g H_2O_2/ 1 t ore)	Comparative Example 1 (no H_2O_2)
Cu Recovery from float	86.0 %	84.1 %
Ni recovery from float	41.6 %	69.2 %
Ni recovery from underflow#	27.6 %	9.8 %

Ni recovery from the underflow was obtained by performing a second froth flotation of the underflow with a Ni collector and measuring Ni content in the froth.

As can be seen, the amount of copper recovered from the float is increased by the addition of H_2O_2 and the amount of Ni that can be recovered from the underflow is also increased, thus indicating that the separation of Cu and Ni during the second froth flotation is facilitated by the addition of H_2O_2.

CLAIMS

1. A process for extracting Ni and Cu from an ore comprising Ni and Cu and separating Ni from Cu, the process comprising the steps of:

a) conditioning the ore to provide a conditioned ore,

b) subjecting the conditioned ore to froth flotation to provide a concentrate and an underflow, and

c) recovering Cu in the float and Ni in the underflow,

wherein

conditioning the ore is conducted in the presence of H_2O_2 at a pH value of above 7.0, and

H_2O_2 is used in an amount of 50 g to 500 g H_2O_2 per 1t of the ore.

2. The process according to claim 1, wherein the conditioning is conducted at a pH value of 8.0 to 13.0, preferably at a pH value of 9.0 to 11.0.

3. The process according to claim 1 or 2, wherein a flotation reagent selected from collectors, frothers, froth phase modifiers, dispersants, depressants, suppressants, pH regulators, activators, and two or more thereof is added during the process.

4. The process according to any one of claims 1 to 3, wherein a Cu collector selected from metal alkyl xanthates, preferably potassium amyl xanthate, phosphonic acids, phosphoric acid esters, carboxylic acids and salts thereof, metal alkyl sulfates, metal alkyl sulfonates, dialkyl or diaryl dithiophosphoric acids and salts thereof, mercaptobenzothiazole, xanthogen formates, alkyl ethoxycarbonyl thioureas, thionocarbamates, tertiary dodecyl mercaptans, and mixtures of two or more thereof is added during conditioning of the ore.

5. The process according to any one of claims 1 to 4, wherein the concentrate contains at least 84 % Cu based on the amount of Cu in the ore.

6. The process according to any one of claims 1 to 5, wherein the ore comprises 0.10 to 2.0 wt.-% Cu and 0.10 to 2.0 wt.-% Ni.

7. The process according to any one of claims 1 to 6, wherein the ore comprises nickel sulfides, iron sulfides and/or copper sulfides, preferably pyrrhotite, chalcopyrite and/or pentlandite.

8. The process according to any one of claims 1 to 7, wherein a depressant selected from calcium carbonate, sodium chloride, sodium cyanide, sodium silicate, sodium sulfite, sodium metabisulfite, triethylenetetramine, diethylenetriamine, starch, guar, dextrins, carboxymethyl cellulose, and mixtures of two or more thereof is added during conditioning of the ore.

9. The process according to any one of claims 1 to 8, wherein the H_2O_2 is added as a 10 wt.-% to 80 wt.-% H_2O_2 aqueous solution.

10. The process according to any one of claims 1 to 9, wherein H_2O_2 is used in an amount of 60 g to 400 g H_2O_2 per 1t of the ore, preferably in an amount of 70 g to 300 g H_2O_2 per 1t of the ore, more preferably in an amount of 80 g to 200 g H_2O_2 per 1t of the ore.

11. The process according to any one of claims 1 to 10, wherein the ore is provided with a particle size P_{80} of 10 µm to 300 µm, preferably by grinding the ore before conditioning the ore.

12. The process according to any one of claims 1 to 11, wherein the conditioned ore is subjected to froth flotation for at least 5 minutes, at least 10 minutes, or at least 25 minutes.

13. The process according to any one of claims 1 to 12, wherein the concentrate is separated continuously or in a batch-wise fashion from the underflow.

14. The process according to any one of claims 1 to 13, wherein no H_2O_2 is added during the froth flotation.

15. Use of H_2O_2 in a conditioning step for extracting Ni and Cu from an ore and separating Ni from Cu by froth flotation.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2024/059456

A. CLASSIFICATION OF SUBJECT MATTER
INV. B03D1/008 B03D1/02 B03D1/002 B03D1/018 B03D1/01
B03D1/012 B03D1/014 B03D1/016
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
B03D B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CA 1 104 274 A (INCO LTD) 30 June 1981 (1981-06-30) Figures: 1 Claims: 1 Tables: 5 page 2, lines 4-9 page 2, steps i) and ii) page 2, step iii page 6, lines 25-27 page 13, lines 9-10 page 4, lines 17-21 page 1, line 21 page 7, line 14 page 2, lines 13-14 page 6, lines 3-4 page 12 ----- -/--	1-15

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
16 June 2024	10/07/2024

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Accettola, Francesca

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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