

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property
Organization
International Bureau

(43) International Publication Date
07 July 2022 (07.07.2022)



(10) International Publication Number
WO 2022/144292 A1

(51) International Patent Classification:

C07C 319/20 (2006.01) A23K 20/142 (2016.01)
C07C 323/60 (2006.01) A23K 50/80 (2016.01)
C07D 241/08 (2006.01)

(21) International Application Number:

PCT/EP2021/087451

(22) International Filing Date:

23 December 2021 (23.12.2021)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

20217816.6 30 December 2020 (30.12.2020) EP

(71) Applicant: **EVONIK OPERATIONS GMBH** [DE/DE];
Rellinghauser Strasse 1-11, 45128 Essen (DE).

(72) Inventors: **BRAUNE, Sascha**; Leo Vermandellei 17, 2180
Antwerpen (BE). **VAN EESTER, Geert**; Thibautstraat
56 A6, 2100 Antwerpen (BE). **HASSELBACH, Hans-
Joachim**; Deutschordenstr. 6, 63571 Gelnhausen (DE).
DYBALLA, Claudia; Liestalerstr. 17, 4412 Nuglar (CH).

(74) Agent: **EVONIK PATENT ASSOCIATION**; c/o Evonik
Industries AG, IP Management, Bau 1042A, PB15, Paul-
Baumann-Strasse 1, 45772 Marl (DE).

(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO,
DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN,

HR, HU, ID, IL, IN, IR, IS, IT, JO, JP, KE, KG, KH, KN,
KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD,
ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO,
NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW,
SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN,
TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every
kind of regional protection available): ARIPO (BW, GH,
GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ,
UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ,
TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK,
EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV,
MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM,
TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,
KM, ML, MR, NE, SN, TD, TG).

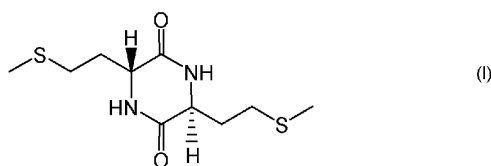
Declarations under Rule 4.17:

— as to applicant's entitlement to apply for and be granted a
patent (Rule 4.17(ii))

Published:

— with international search report (Art. 21(3))
— before the expiration of the time limit for amending the
claims and to be republished in the event of receipt of
amendments (Rule 48.2(h))

(54) Title: PROCESS FOR THE DIASTEREOMERICALLY PURE PREPARATION OF DL/LD-METHIONYLMETHIONINE



(57) Abstract: The invention relates in particular to a process for the preparation of DL/LD-methionine diketopiperazine (I) characterized in that a DD/LL-methionylmethionine-containing aqueous solution or suspension, which has a pH of 3 to 7, measured using a pH electrode at 20°C, is reacted at a temperature of 150 to 220°C, preferably at 170 to 210°C, until an aqueous solution or suspension has formed containing at least a proportion of 30 mol%, preferably 50 mol%, especially 60 mol% of DL/LD-methionine diketopiperazine, based on the total content of DL/LD- and DD/LL-methionine diketopiperazine in the reaction mixture. This process forms part of an overall process for the preparation of DL/LD-methionylmethionine, which is obtained by hydrolysis from the DL/LD-methionine diketopiperazine (I) produced initially.

WO 2022/144292 A1