Supporting Information

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Isolation of amygdalin epimer at high diastereomeric purity and its structural characterization by spectroscopic and

Q-TOF LC-MS methods

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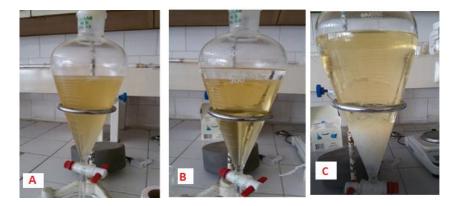


Figure S1: Liquid-Liquid Extractions (LLE); A = n-hexzane, B = petrelium ether, C = chloroform

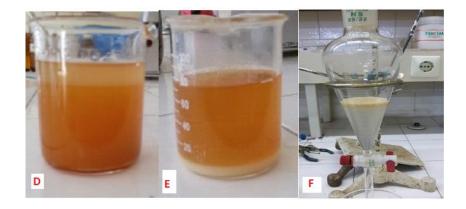


Figure S2: Decantation Methods; D = n-hexzane, E = petrelium ether, F = chloroform

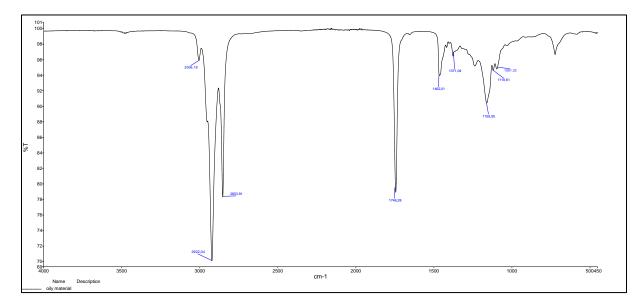


Figure S3: ATR-IR spectrum of the amygdalin-free component of the extract

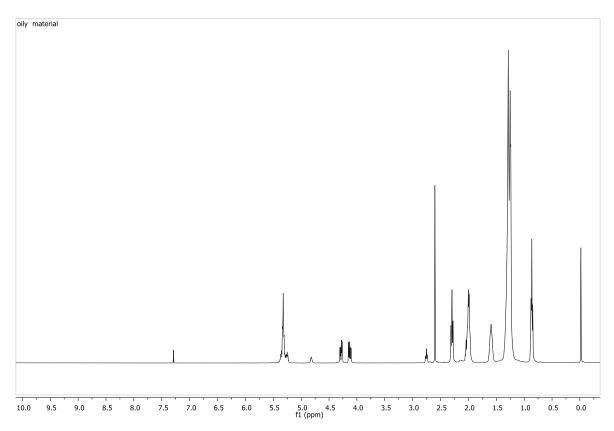


Figure S4: ¹H-NMR (400 MHz, CDCl₃) spectrum of the amygdalin-free component of the extract

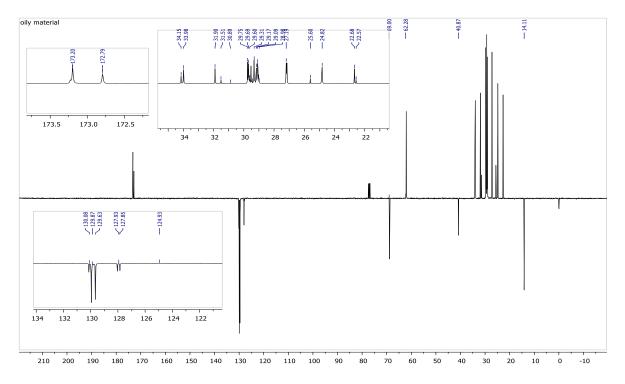


Figure S5: ¹³C-NMR (400 MHz, CDCl₃) spectrum of the amygdalin-free component of the extract

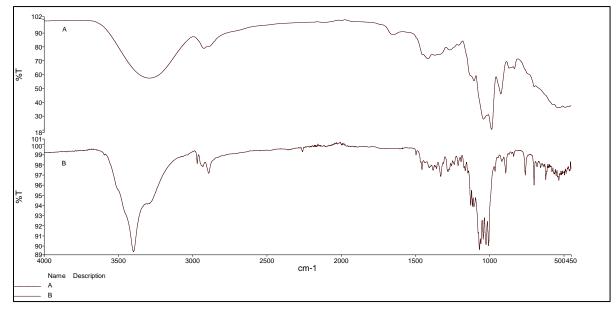


Figure S6 :(A) ATR-FTIR spectrum of the extract obtained without any purification operations; (B) ATR-FTIR spectrum of the isolated amygdalin at high diastereomeric purity

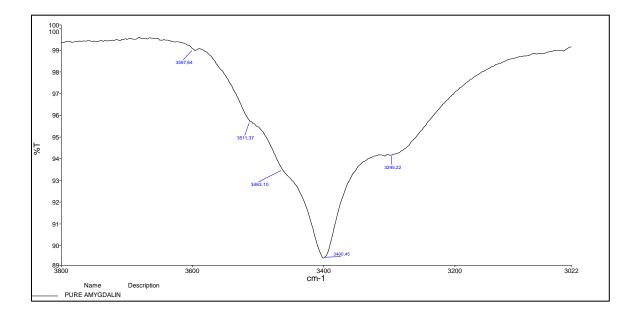


Figure S7: O-H bonds region of amygdalin

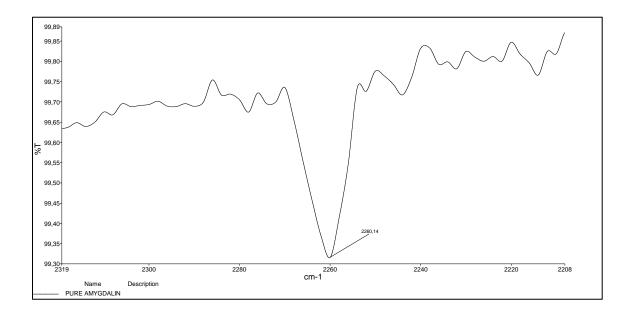


Figure S8: Nitrile region of amygdalin

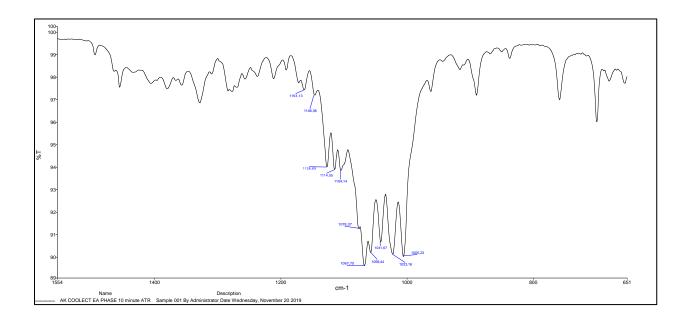


Figure S9: C-O-H and C-O-C bond region of amygdalin

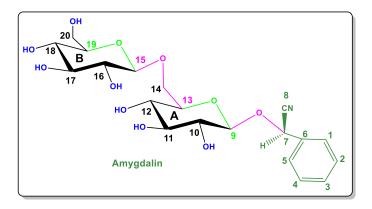
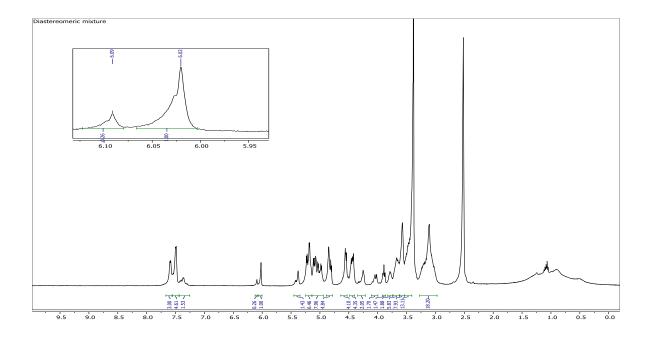


Figure S10: Numbering system used to evaluate NMR spectra of amygdalin. It is not related to the IUPAC numbering system



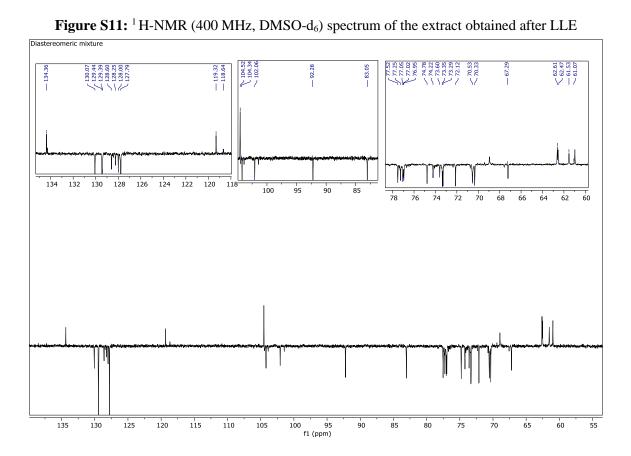


Figure S12: ¹³C-NMR (100 MHz, DMSO-d₆) spectrum of the extract obtained after LLE

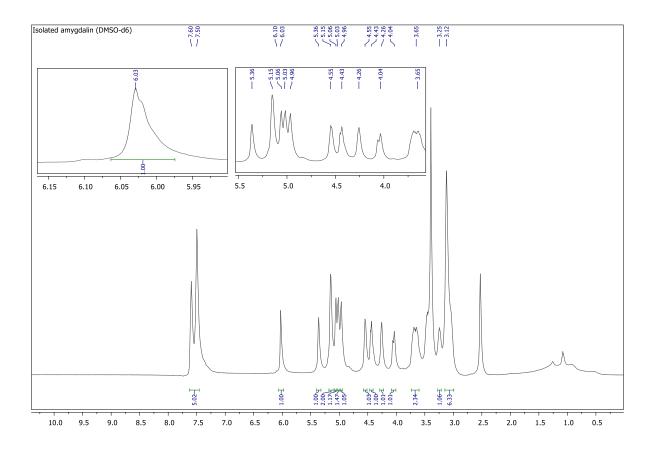


Figure S13: ¹H-NMR (400 MHz, DMSO-d₆) spectrum of the amygdalin isolated at high purity after column chromatography

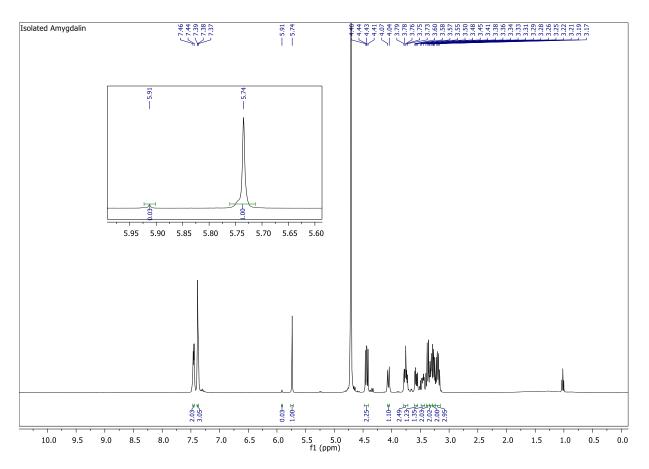


Figure S14: The ¹H-NMR (400 MHz, D₂0) spectrum of the amygdalin after column chromatography

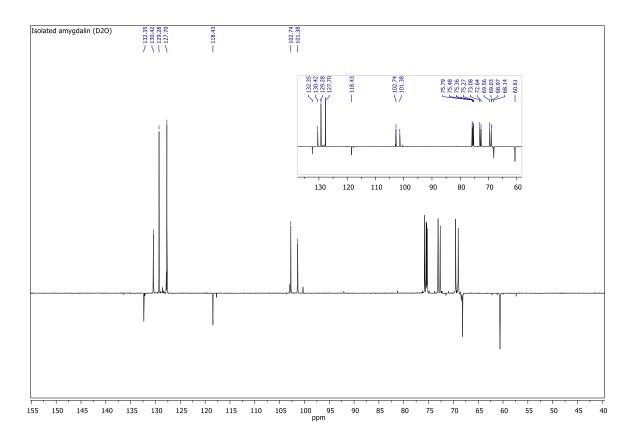
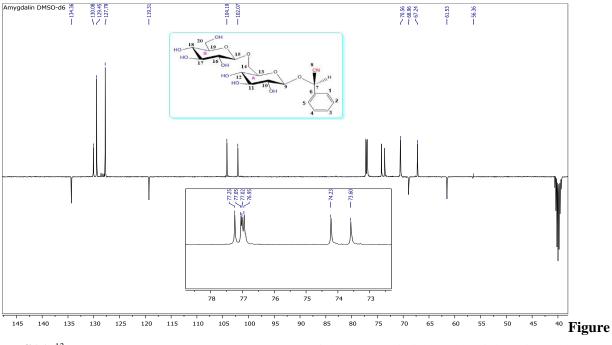


Figure S15: ¹³C-NMR (100 MHz, D₂O) spectrum of the amygdalin isolated after column chromatography



S16: ¹³C-NMR (100 MHz, DMSO-d₆) spectrum of the amygdalin isolated at high purity column chromatography

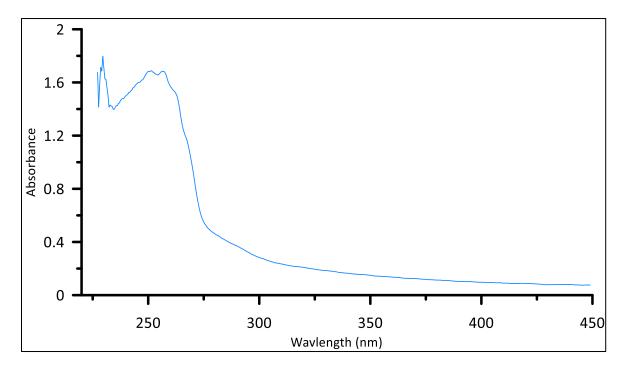


Figure S17: The UV-VIS spectrum of the isolated pure amygdalin.

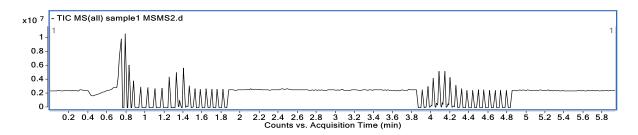


Figure S18: TIC of the sample in MS-MS mode

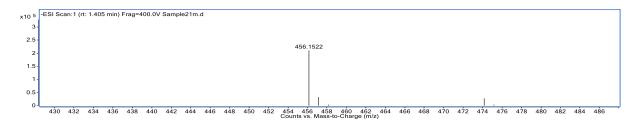


Figure S19: MS spectrum of peak at RT 1.41

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|------|-------|---------------------|--------------------|-----------|-----------------|-------------|---------------|--------------|-------|-----------|------------------|----------------|-------------|-----------------|-------|
| Bea | at T | ⊽+¤ ID Source ⊽+¤ | Name 🔽 😐 🛛 For | mula | v + Species * | ∀+ ₽ | m/z ⊽+¤ Sc | ore⊽⊽≠PDif | (ppm) | ∵r⇔ Sc | ore (MFG) 🕆 🗝 | Mass 🔽 🗝 | | | |
| • • | | MFG | C20 H2 | 7 N O 1 1 | (M-H)- | | 456.1522 87 | .71 -2. |)9 | 87 | .71 | 457.1594 | | | |
| | Sco | re (iso. abund) 🛛 🐨 | 🕈 Score (mass) 🔽 🕂 | Score (| IFG, MS/MS) | 7 - s | Score (MS) 🔽 | - Score (MFC |) 🗸 🕫 | Score (i | so. spacing) 🔽 - | P Height ⊽-P | Ion Formula | V ⊕ Species V + | m/z 💎 |
| | 65.46 | | 95.68 | | 4 | 87.71 | 87.71 | | 98.47 | | 210776.2 C | 20 H26 N O11 | (M-H)- | 456.152 | |
| | | Height (Calc) 🛛 🕆 🛱 | Height Sum% (Calc) | ₩÷ He | ight % (Cale) 5 | 7-1= n | n/z (Calc) ⊽≉ | Diff (mDa) 🔽 | + He | ight ⊽ +¤ | Height % 💎 🖶 | Height Sum % 5 | 7+ m/z - + | Diff (ppm) 🔽 🖶 | |
| | | 194524.4 | 78.5 | 10 | D | 4 | 456.1511 | -1.1 | 21 | 0776.2 | 100 | 85 | 456.1522 | -2.31 | |
| | | 44185.9 | 17.8 | 22 | 7 | 4 | 457.1544 | -0.4 | 33 | 032.2 | 15.7 | 13.3 | 457.1549 | -0.96 | |
| | | 9186.5 | 3.7 | 4 | | 4 | 458,1566 | -0.1 | 40 | 88.5 | 1.9 | 1.6 | 458,1567 | -0.23 | |

Figure S20. Isotopic distribution of 456 ion at RT 1.42 showing experimental and calculated values

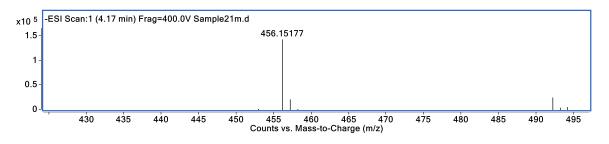


Figure S21: MS spectrum of peak at RT 4.17

| · · | | | | ation Re | | | | | | | _ | | | | | | | | | | | | | | |
|----------|---------------|-------|-----------|----------|----------------|------------|----------|--------|----------|-----------|------|--------|---------|------------------|-----------|---------|------------|------------|----------------|----------|-----------|-----------|-------------------|-------|-------------|
| <u>ا</u> | Autor | mati | ally Sho | ow Colur | nns | * 8 | 네 🔀 | 6 | R | 🤹 👧 | | | | | | | | | | | | | | | |
| В | est | 7+ | ID Sou | rce ⊽+¤ | Nan | ne ⊽+¤ | For | nula | 7₽ | Species | 7₽ | m/z | ⊽⊅ | Score | ⊽∀₽ | Score | (RT) 🖓 🕫 | RT Diff ⊽‡ | Diff (pp | m) \7 +¤ | Score (L | ib) ⊽ 中: | Score (DB) 🗸 | + Sco | ore (MFG) 7 |
| | ۲ | | MFG | | | | C20 H2 | 7 N O1 | 1 | (M-H)- | | 456.15 | 5177 | 88.09 | | | | | -1.16 | | | | | 88. | 09 |
| | Spe | ecies | 7- | m/z S | 7+Þ | Score (i | so. abur | d) ⊽⊀ | Sco | re (mass) | 7-10 | Score | (MFG | i, MS/I | /IS) ⊽+= | Score | e (MS) 🖓 🛱 | Score (MF | G)⊽ ∀ + | Score | (iso. spa | cinq) ⊽+= | Height ⊽+Þ | lon f | Formula 7 |
| | (M-H)- 456.15 | | 456.151 | 77 | 77 61.88 98.65 | | | 5 | | | | | 88.09 | | 9 | 88.09 | | 98.43 | | | 143313.3 | C20 H | C20 H26 N O11 | | |
| | | Hei | aht (Calc |) 7+ | Heid | aht Sum' | % (Calc) | \ | Height | t % (Calc |)7₽ | m/z ((| Calc) ٦ | 7 4 0 |)iff (mDa |) 7+ | Height VH | ⊨ Height % | | ight Sum | % ⊽≠ | m/z ⊽ | - ⊐ Diff (ppm) |) 🖓 🛱 | |
| | | 1: | 1868.8 | | 78.5 | | | | 100 | | | 456.1 | | | 0.6 | | 143313.3 | 100 | 85 | | | 456.1517 | | | |
| | | 2 | 953.8 | | 17.8 | 1 | | | 22.7 | | | 457.1 | 5444 | 0 |) | | 21704 | 15.1 | 12 | .9 | | 457.1544 | 48 -0.08 | | |
| | | 6 | 27.6 | | 3.7 | | | | 4.7 | | | 458.1 | 5661 | 0 | .9 | | 3032.9 | 2.1 | 1.0 | 3 | | 458.1557 | 72 1.94 | | |

Figure S22: Isotopic distribution of 456 ion at RT 4.17