Data article

Data on nitrogen-containing derivatives of fumaropimaric acid

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ABSTRACT

The data presented here are related to the research paper entitled “Levopimaric Acid Derived 1,2-Diamines and Their Application in the Copper-Catalyzed Asymmetric Henry Reaction” [1]. In this data article, we provide 1H, 13C NMR and IR data for the diterpene derivatives described in [1]. The GC–MS analysis of pine oleoresin used as a starting material of the syntheses is also included in the data article.

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Specifications table

| Subject area                        | Chemistry                                      |
|-------------------------------------|------------------------------------------------|
| More specific subject area          | Organic synthesis, natural products           |
| Type of data                        | Synthetic schemes, NMR and IR spectra, GC-chromatogram |
| How data was acquired               | NMR spectroscopy: Bruker DRX-500, AM-400 and AV-300; IR spectroscopy: Infralum FT-801 and Shimadzu IRAfinity-1 FT-IR; GC–MS analysis: SHIMADZU GCMS-QP2010 Ultra instrument on the basis of gas chromatograph GC-2010 plus with mass detector |
| Data format                         | Raw, analyzed                                  |
| Experimental factors                | The new diterpene derivatives were synthesized and purified by column chromatography or crystallization |

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Experimental features  The synthesized compounds were characterized by NMR and IR spectroscopy
Data source location  Novosibirsk, Russian Federation
Data accessibility  Data are available with this article

Value of the data

- The data presents NMR, IR spectra of newly synthesized diterpene derivatives and GC–MS analysis of methylated pine oleoresin and could be used by other researchers.
- The provided information on the structural data of diterpenes could be useful for the analysis of spectra and determination of the structure of other diterpene derivatives.
- The data could be helpful for other researchers to identify the compounds described in the research article [1] and to reproduce the experiments reported therein.

1. Data

The dataset presented in this article focuses on characterization of the new diterpene derivatives described in [1]. The article provides the information on the composition of natural raw material (pine oleoresin) and the structural data of the functionalized diterpenes. Scheme 1 illustrates the preparation of mixture of methylated resin acids. The GC–MS analysis of this mixture is given in Fig. 1. Scheme 2 illustrates the method of preparation and isolation of monomethyl ester of fumaropimaric acid 2. The compound 2 was characterized using $^1$H, $^{13}$C NMR and IR (Figs. 2-1, 2-2 and 2-3). Scheme 3 illustrates the synthetic route to the 1,2-disocyanate 3, which was characterized using $^1$H, $^{13}$C NMR and IR (Figs. 3-1, 3-2 and 3-3). Scheme 4 illustrates the synthetic route to the 1,2-diamine 4, which was characterized using $^1$H, $^{13}$C NMR and IR (Figs. 4-1, 4-2 and 4-3). Scheme 5 illustrates the method of preparation of imines 5a-f and aminophenols 6a-f. Figs. 5a-f and 6a-f shows $^1$H, $^{13}$C NMR spectra of the compounds 2 and 4 are provided in [2]. Analyses of the spectra of the compounds 3, 5a-f and 6a-f are provided in [1]. The synthetic procedures for the compounds 2–6 are described in the research article [1].

2. Experimental design, materials and methods

2.1. General information

The chemicals were of reagent purity grade, obtained from commercial sources, and used without further purification. Pine oleoresin OST 13-128-93 (Russian industry standard; oleoresin contains at least 80% of abietic-type acids) was obtained from Orgsyntez OJSC (available on request at http://
orgsyntez.ru/en) and used as received. Solvents were distilled from appropriate drying agents prior to use, unless otherwise noted. Flash column chromatography was performed on silica gel (Panreac 40–63 µm). $^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker DRX-500, AM-400 and AV-300 spectrometers. Chemical shifts were reported in the δ scale using the residual solvent peak of the CHCl$_3$ as a reference (726 ppm) for $^1$H NMR spectra and the middle signal in the triplet of CDCl$_3$ (77.00 ppm) for $^{13}$C NMR samples. IR spectra were recorded using Infralum FT-801 or Shimadzu IRAffinity-1 FT-IR spectrometers. GC–MS analysis of methyl esters of resin acids was carried out on SHIMADZU GCMS-QP2010 Ultra instrument on the basis of gas chromatograph GC-2010 plus with mass detector and with chromatographic column GsBP1-MS 30 m × 0.32 mm.

3. Fumaropimaric acid derivatives

3.1. Methylated pine oleoresin

3.2. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-dicarboxy-4-carboxylic acid methyl ester 2
3.3. 13-isopropyl-17,18-dinor-atis-13-ene-15\(\beta\),16\(\alpha\)-diisocyanato-4-carboxylic acid methyl ester 3

3.4. 13-isopropyl-17,18-dinor-atis-13-ene-15\(\beta\),16\(\alpha\)-diamino-4-carboxylic acid methyl ester 4

Scheme 2. Synthesis of monomethyl ester of fumaropimaric acid 2 via Diels-Alder reaction of methyl levopimarate 1a with fumaric acid.

Fig. 2-1. IR spectrum of compound 2.
3.5. Imines 5a-f

3.5.1. 13-Isopropyl-17,18-dinor-atis-13-ene-15β,16α-di(2-hydroxybenzylideneamino)-4-carboxylic acid methyl ester 5a

$^{1}$H, $^{13}$C NMR and IR spectra of the compound 5a are presented in Figs. 5a-1, 5a-2 and 5a-3.

3.5.2. 13-Isopropyl-17,18-dinor-atis-13-ene-15β,16α-di(2-hydroxy-3-methoxybenzylideneamino)-4-carboxylic acid methyl ester 5b

$^{1}$H, $^{13}$C NMR and IR spectra of the compound 5b are presented in Figs. 5b-1, 5b-2 and 5b-3.

3.5.3. 13-Isopropyl-17,18-dinor-atis-13-ene-15β,16α-di(2-hydroxy-3,5-di-tert-butylbenzylideneamino)-4-carboxylic acid methyl ester 5c

$^{1}$H, $^{13}$C NMR and IR spectra of the compound 5c are presented in Figs. 5c-1, 5c-2 and 5c-3.

3.5.4. 13-Isopropyl-17,18-dinor-atis-13-ene-15β,16α-di(2-hydroxy-1-naphthylmethyleneamino)-4-carboxylic acid methyl ester 5d

$^{1}$H, $^{13}$C NMR and IR spectra of the compound 5d are presented in Figs. 5d-1, 5d-2 and 5d-3.

Fig. 2-2. $^{1}$H NMR spectrum of compound 2.
3.5.5. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di(2’-pyridyl-methyleneamino)-4-carboxylic acid methyl ester 5e

$^1$H, $^{13}$C NMR and IR spectra of the compound 5e are presented in Figs. 5e-1, 5e-2 and 5e-3.

3.5.6. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di(thioph-2-ylideneda)-4-carboxylic acid methyl ester 5f

$^1$H, $^{13}$C NMR and IR spectra of the compound 5f are presented in Figs. 5f-1, 5f-2 and 5f-3.
3.6. Aminophenols 6a-f

3.6.1. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di[(2-hydroxybenzyl)amino]-4-carboxylic acid methyl ester 6a

$^1$H, $^{13}$C NMR and IR spectra of the compound 6a are presented in Figs. 6a-1, 6a-2 and 6a-3.

3.6.2. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di[(2-hydroxy-3-methoxybenzyl)amino]-4-carboxylic acid methyl ester 6b

$^1$H, $^{13}$C NMR and IR spectra of the compound 6b are presented in Figs. 6b-1, 6b-2 and 6b-3.

3.6.3. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di[(2-hydroxy-3,5-di-tert-butylbenzyl)amino]-4-carboxylic acid methyl ester 6c

$^1$H, $^{13}$C NMR and IR spectra of the compound 6c are presented in Figs. 6c-1, 6c-2 and 6c-3.

3.6.4. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di[(2-hydroxynaphthalen-1-yl)methylamino]-4-carboxylic acid methyl ester 6d

$^1$H, $^{13}$C NMR and IR spectra of the compound 6d are presented in Figs. 6d-1, 6d-2 and 6d-3.
Fig. 3-2. $^{13}$C NMR spectrum of compound 3.

Fig. 3-3. IR spectrum of compound 3.
3.6.5. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di[(2-pyridylmethyl)amino]-4-carboxylic acid methyl ester 6e

$^1$H, $^{13}$C NMR and IR spectra of the compound 6e are presented in Figs. 6e-1, 6e-2 and 6e-3.

3.6.6. 13-isopropyl-17,18-dinor-atis-13-ene-15β,16α-di[[(thiophen-2-yl)-methyl]amino]-4-carboxylic acid methyl ester 6f

$^1$H, $^{13}$C NMR and IR spectra of the compound 6f are presented in Figs. 6f-1, 6f-2 and 6f-3.
Fig. 4-2. $^{13}$C NMR spectrum of compound 4.

Fig. 4-3. IR spectrum of compound 4.
Scheme 5. Synthesis of imines 5a-f and aminophenols 6a-f.

Fig. 5a-1. $^1$H NMR spectrum of compound 5a.
Fig. 5a-2. $^{13}$C NMR spectrum of compound 5a.

Fig. 5a-3. IR spectrum of compound 5a.
Fig. 5b-1. $^1$H NMR spectrum of compound 5b.
Fig. 5b-2. $^{13}$C NMR spectrum of compound 5b.

Fig. 5b-3. IR spectrum of compound 5b.
Fig. 5c-1. $^1$H NMR spectrum of compound 5c.
Fig. 5c-2. $^{13}$C NMR spectrum of compound 5c.

Fig. 5c-3. IR spectrum of compound 5c.
Fig. 5d-1. $^1$H NMR spectrum of compound 5d.
Fig. 5d-2. $^{13}$C NMR spectrum of compound 5d.

Fig. 5d-3. IR spectrum of compound 5d.
Fig. 5e-1. $^1$H NMR spectrum of compound 5e.
Fig. 5e-2. $^{13}$C NMR spectrum of compound 5e.

Fig. 5e-3. IR spectrum of compound 5e.
Fig. 5f-1. $^1$H NMR spectrum of compound 5f.
Fig. 5f-2. $^{13}$C NMR spectrum of compound 5f.

Fig. 5f-3. IR spectrum of compound 5f.
Fig. 6a-1. $^1$H NMR spectrum of compound 6a.
Fig. 6a-2. $^{13}$C NMR spectrum of compound 6a.

Fig. 6a-3. IR spectrum of compound 6a.
Fig. 6b-1. $^1$H NMR spectrum of compound 6b.
Fig. 6b-2. $^{13}$C NMR spectrum of compound 6b.

Fig. 6b-3. IR spectrum of compound 6b.
Fig. 6c-1. $^1$H NMR spectrum of compound 6c.
Fig. 6c-2. $^{13}$C NMR spectrum of compound 6c.

Fig. 6c-3. IR spectrum of compound 6c.
Fig. 6d-1. $^1$H NMR spectrum of compound 6d.
Fig. 6d-2. $^{13}$C NMR spectrum of compound 6d.

Fig. 6d-3. IR spectrum of compound 6d.
Fig. 6e-1. $^1$H NMR spectrum of compound 6e.
Fig. 6e-2. $^{13}$C NMR spectrum of compound 6e.

Fig. 6e-3. IR spectrum of compound 6e.
Fig. 6f-1. $^1$H NMR spectrum of compound 6f.
Fig. 6f-2. $^{13}$C NMR spectrum of compound 6f.

Fig. 6f-3. IR spectrum of compound 6f.
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Transparency document. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2018.04.059.

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