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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 , ^{13}C 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: Infracum FT-801 and Shimadzu IRAffinity-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	<i>The synthesized compounds were characterized by NMR and IR spectroscopy</i>
Data source location	<i>Novosibirsk, Russian Federation</i>
Data accessibility	<i>Data are available with this article</i>

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.

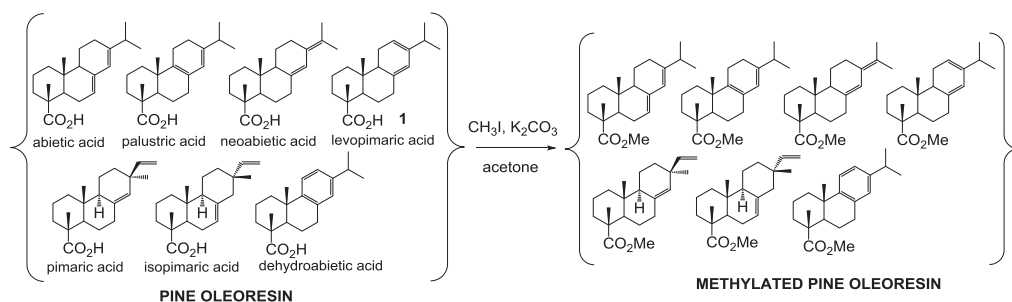
1. Data

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 ^1H , ^{13}C NMR and IR (Figs. 2-1, 2-2 and 2-3). Scheme 3 illustrates the synthetic route to the 1,2-diisocyanate **3**, which was characterized using ^1H , ^{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 ^1H , ^{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 ^1H , ^{13}C NMR and IR spectra of the compounds **5a-f** and **6a-f**. Analyses of the 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://>



Scheme 1. Methylation of resin acids.

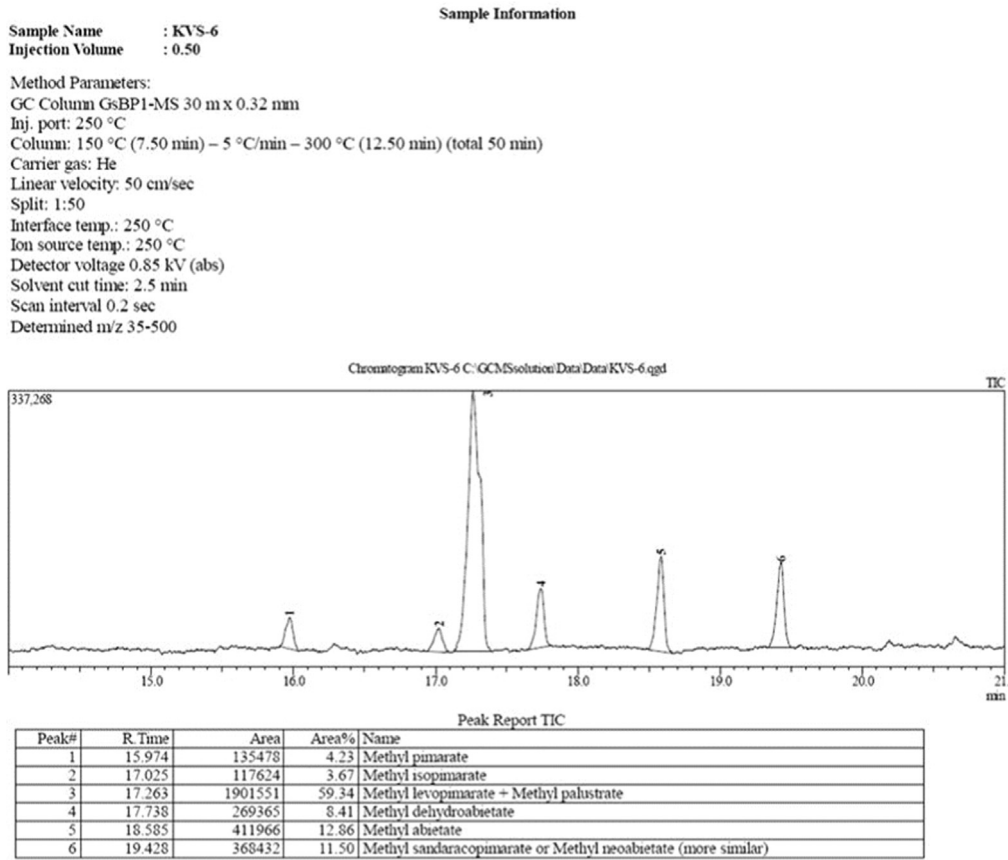


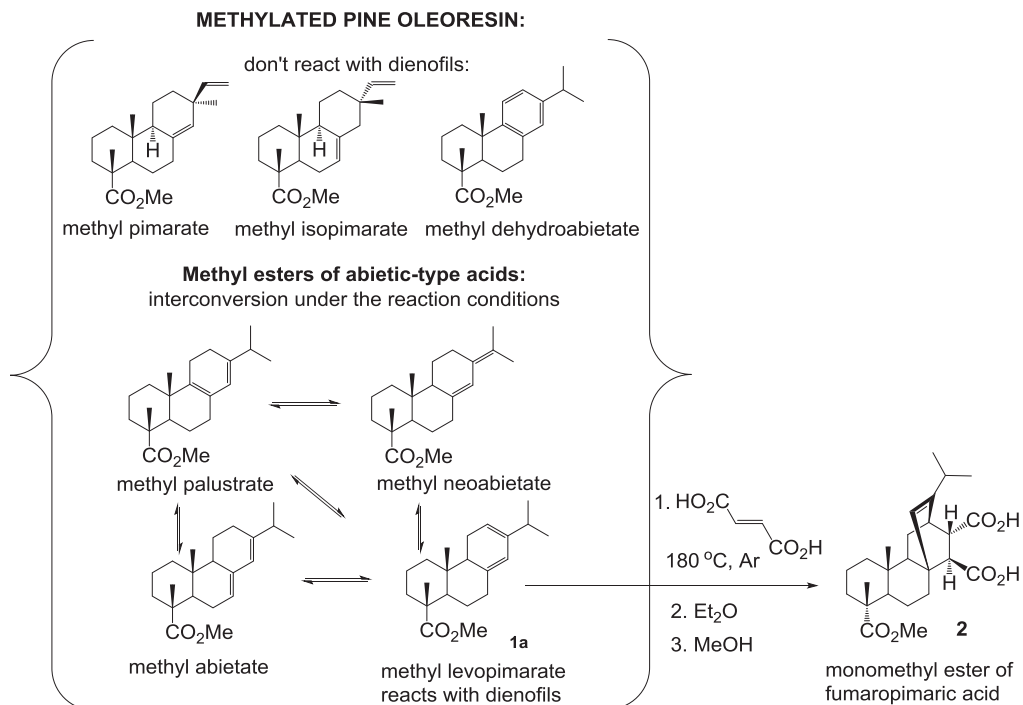
Fig. 1. Chromatogram of methylated oleoresin (methyl esters of resin acids).

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). ¹H NMR and ¹³C 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 CHCl₃ as a reference (726 ppm) for ¹H NMR spectra and the middle signal in the triplet of CDCl₃ (77.00 ppm) for ¹³C NMR samples. IR spectra were recorded using Infracum 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



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

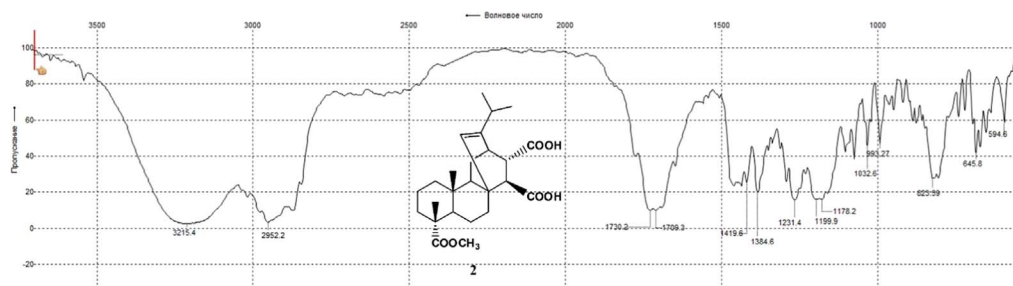


Fig. 2-1. IR spectrum of compound **2**.

3.3. 13-isopropyl-17,18-dinor-atris-13-ene-15 β ,16 α -diisocyanato-4-carboxylic acid methyl ester **3**

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3.4. 13-isopropyl-17,18-dinor-atris-13-ene-15 β ,16 α -diamino-4-carboxylic acid methyl ester **4**

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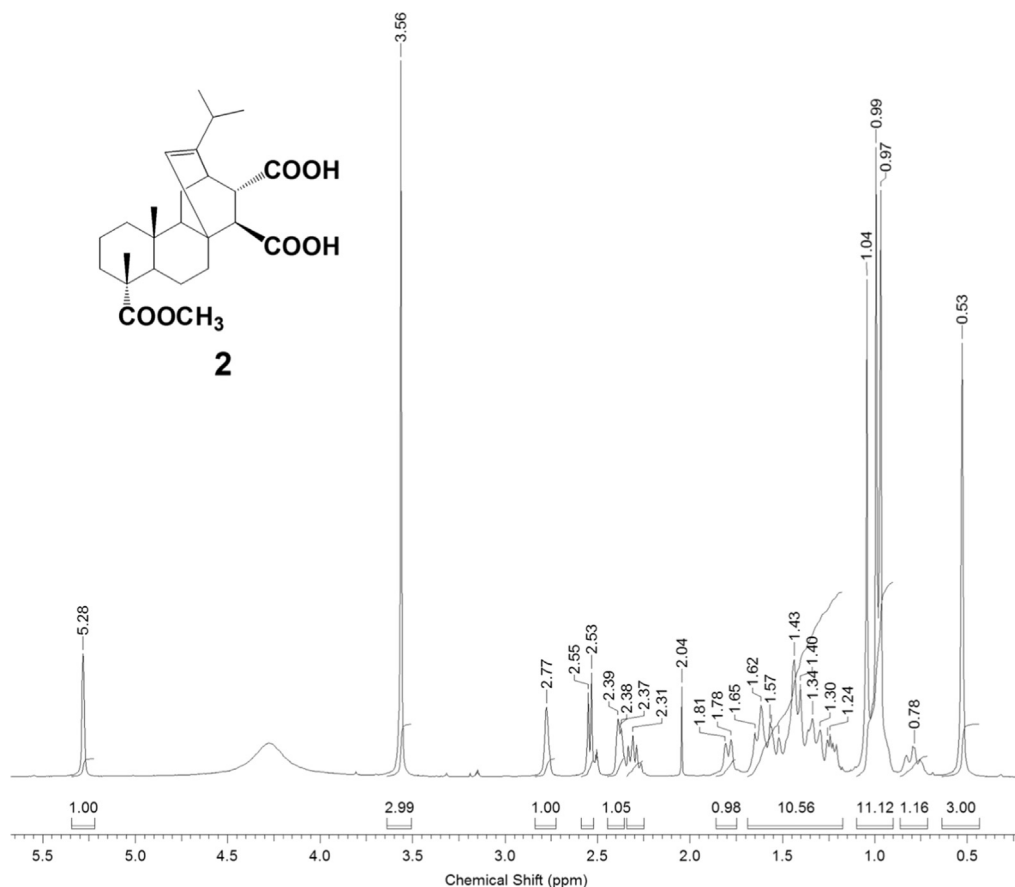


Fig. 2-2. ^1H NMR spectrum of compound **2**.

3.5. Imines **5a-f**

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

^1H , ^{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-15 β ,16 α -di(2-hydroxy-3-methoxybenzylideneamino)-4-carboxylic acid methyl ester **5b**

^1H , ^{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-15 β ,16 α -di(2-hydroxy-3,5-di-tert-butylbenzylideneamino)-4-carboxylic acid methyl ester **5c**

^1H , ^{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-15 β ,16 α -di(2-hydroxy-1-naphthylmethyleneamino)-4-carboxylic acid methyl ester **5d**

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

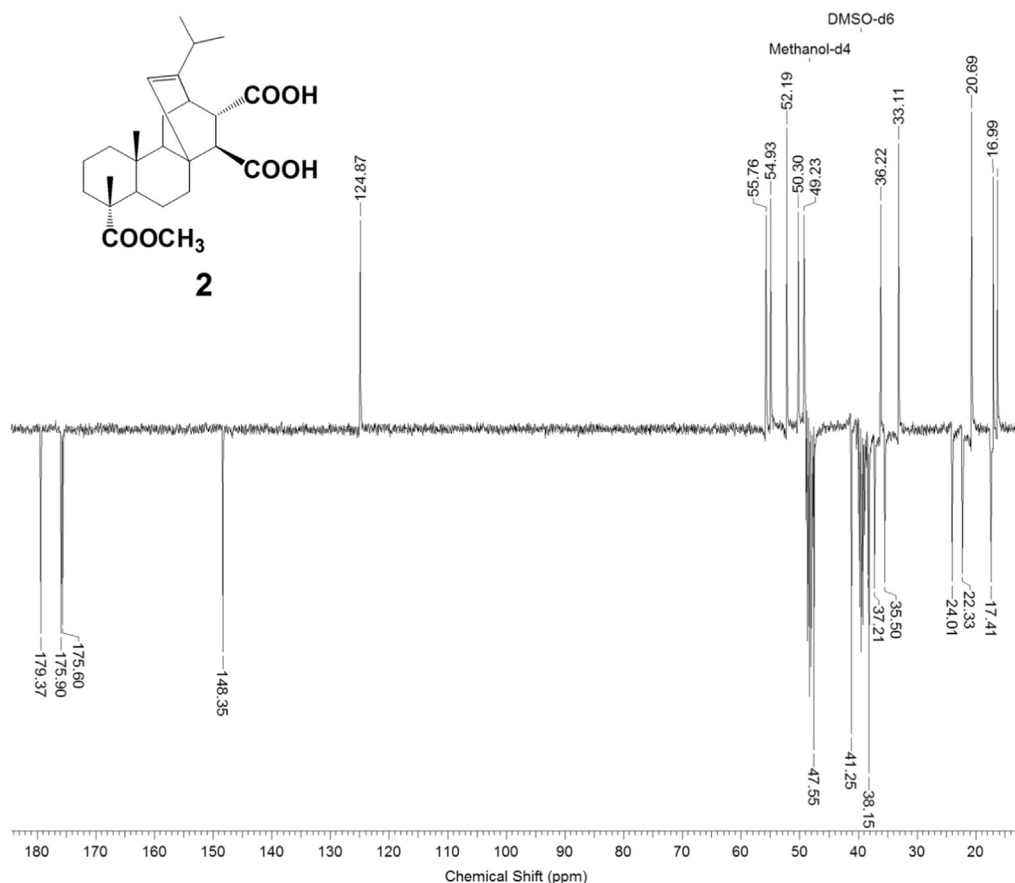
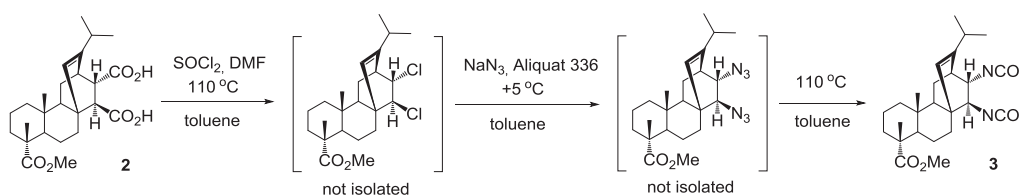


Fig. 2-3. ^{13}C NMR spectrum of compound **2**.



Scheme 3. Synthesis of diisocyanate **3** from diacid **2** via Curtius rearrangement.

3.5.5. 13-isopropyl-17,18-dinor-atis-13-ene-15 β ,16 α -di(2'-pyridyl-methyleneamino)-4-carboxylic acid methyl ester **5e**

^1H , ^{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-15 β ,16 α -di(thioph-2-ylidenamino)-4-carboxylic acid methyl ester **5f**

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

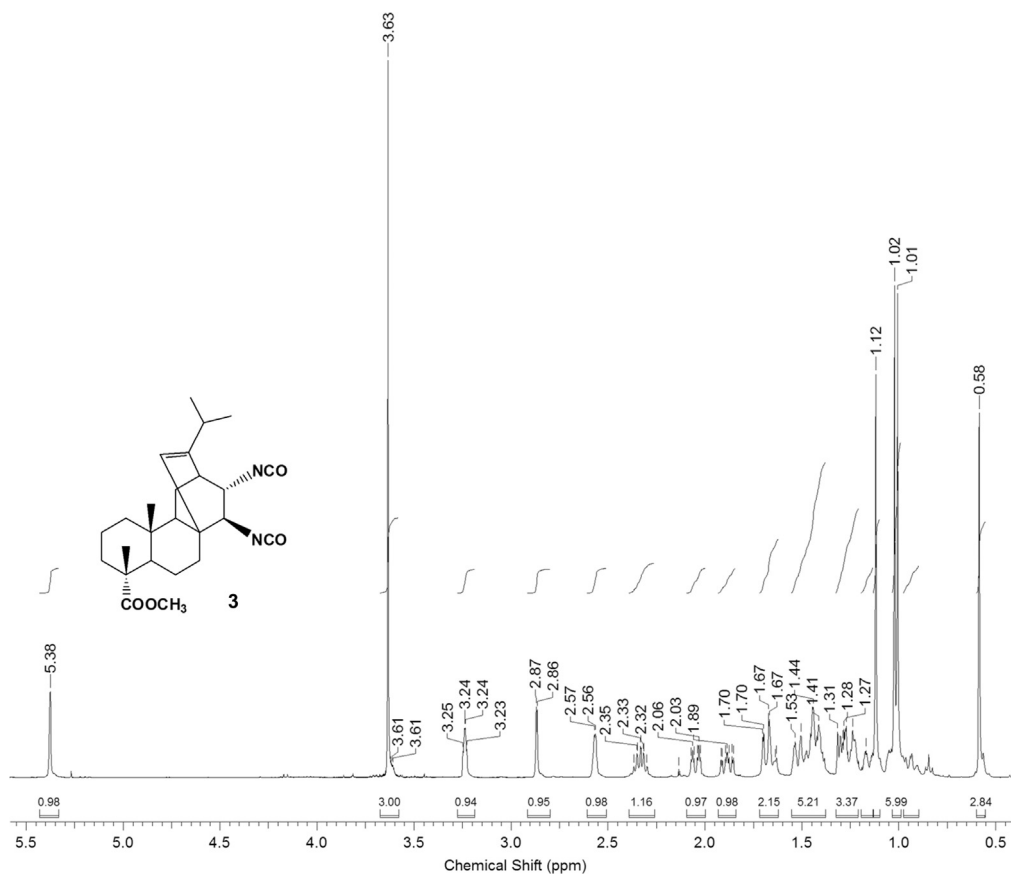


Fig. 3-1. ^1H NMR spectrum of compound **3**.

3.6. Aminophenols 6a-f

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

^1H , ^{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-15 β ,16 α -di[(2-hydroxy-3-methoxybenzyl)amino]-4-carboxylic acid methyl ester 6b

^1H , ^{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-15 β ,16 α -di[(2-hydroxy-3,5-di-tert-butylbenzyl)amino]-4-carboxylic acid methyl ester 6c

^1H , ^{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-15 β ,16 α -di[(2-hydroxynaphthalen-1-yl)methylamino]-4-carboxylic acid methyl ester 6d

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

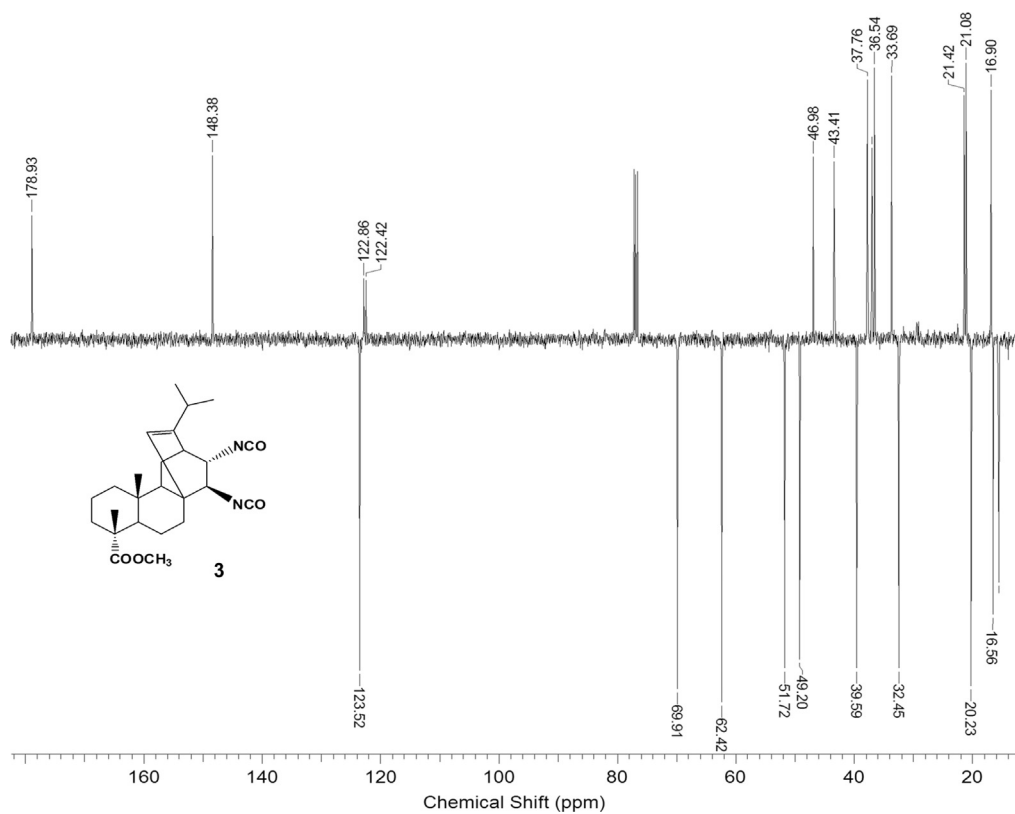
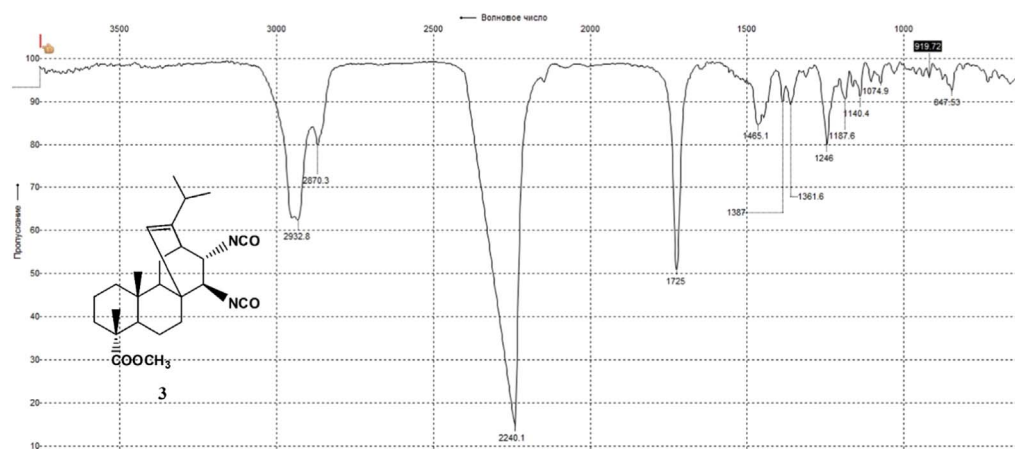
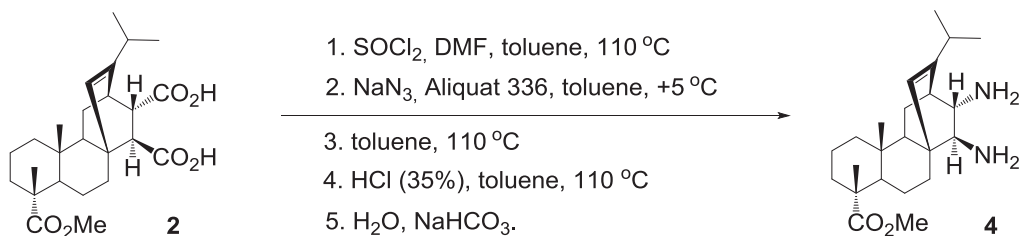
Fig. 3-2. ^{13}C NMR spectrum of compound 3.

Fig. 3-3. IR spectrum of compound 3.



Scheme 4. Synthesis of diamine **4** from diacid **2**.

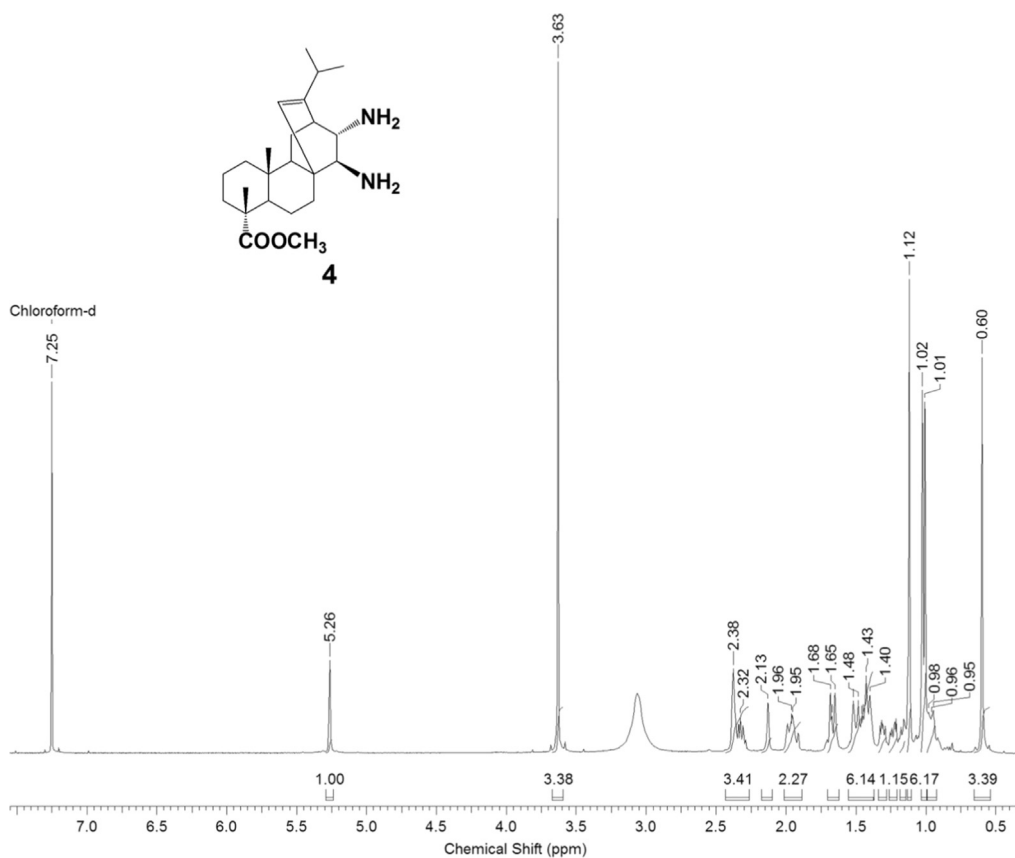


Fig. 4-1. ^1H NMR spectrum of compound **4**.

3.6.5. 13-isopropyl-17,18-dinor-atis-13-ene-15 β ,16 α -di((2-pyridylmethyl)amino)-4-carboxylic acid methyl ester **6e**

^1H , ^{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-15 β ,16 α -di(((thiophen-2-yl)-methyl)amino)-4-carboxylic acid methyl ester **6f**

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

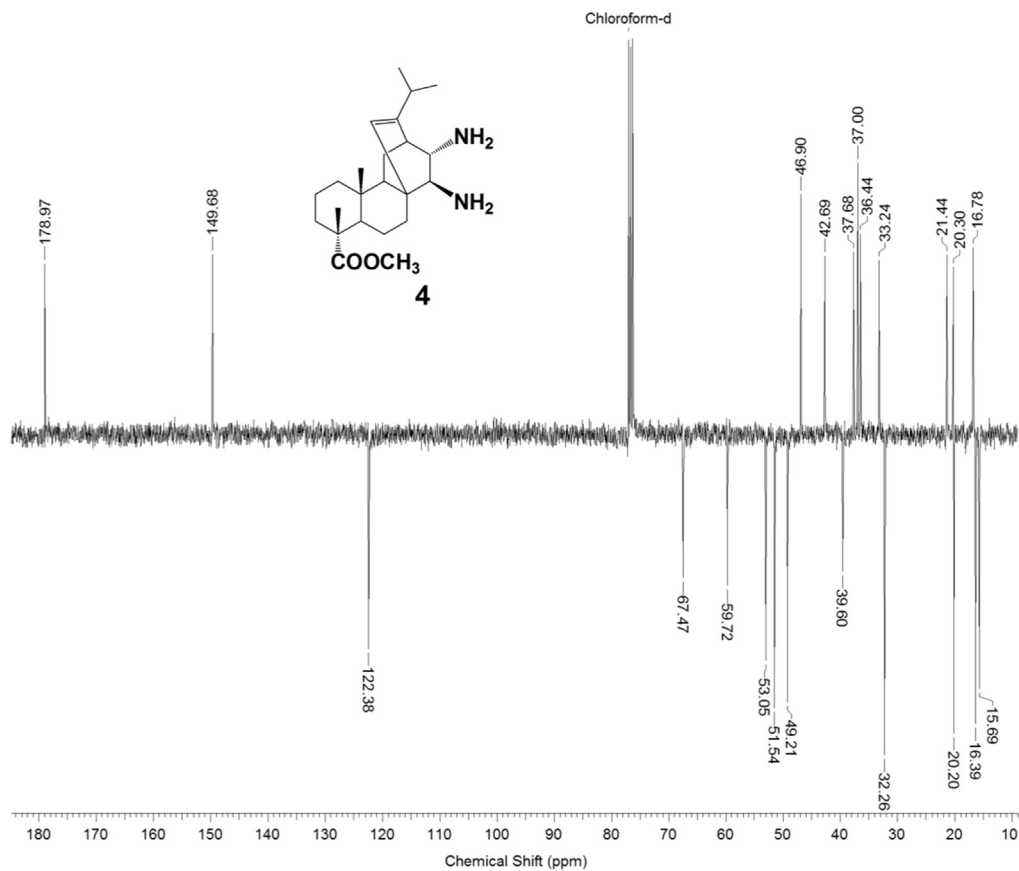


Fig. 4-2. ^{13}C NMR spectrum of compound **4**.

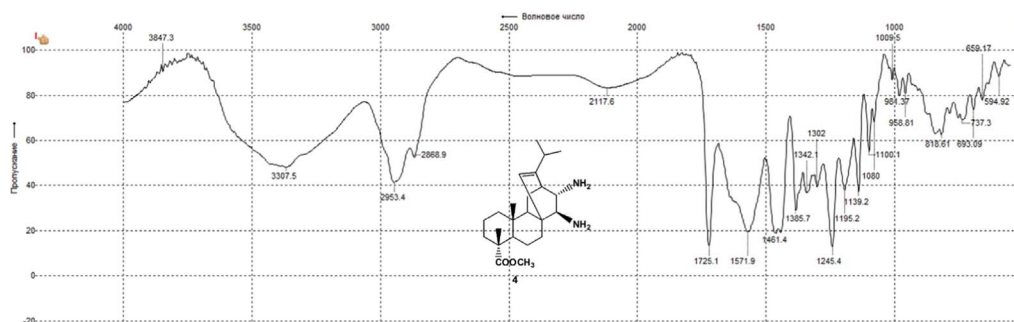


Fig. 4-3. IR spectrum of compound **4**.

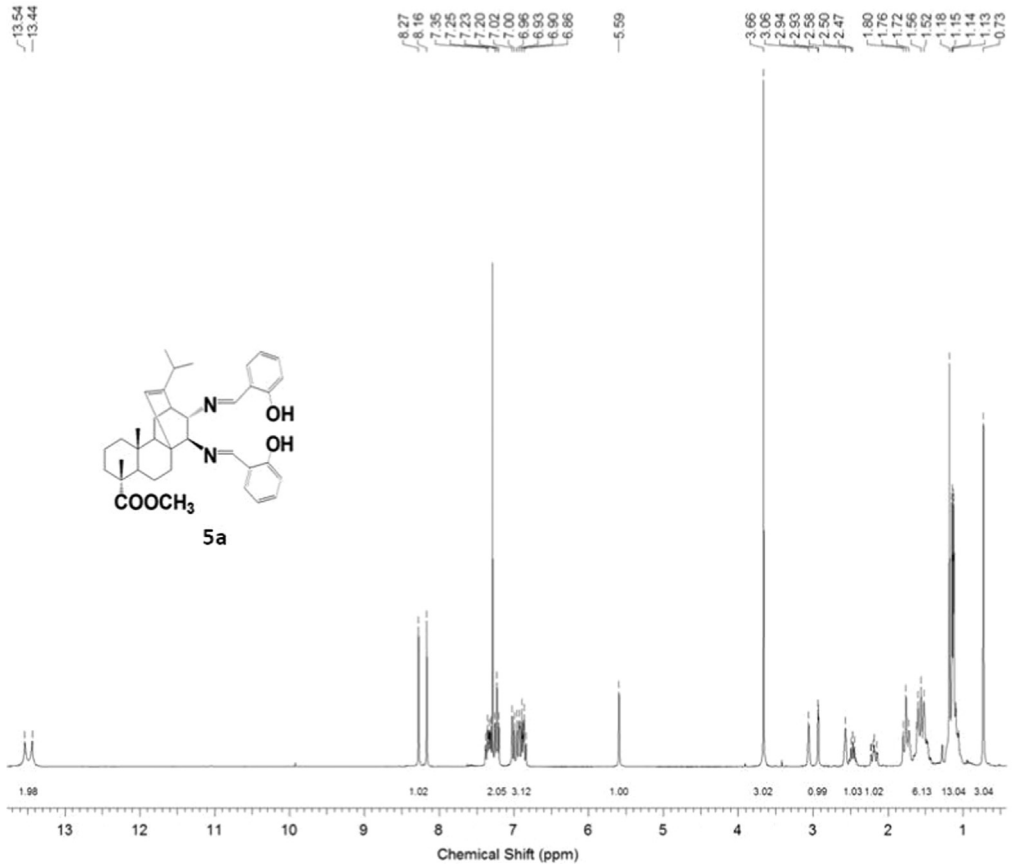
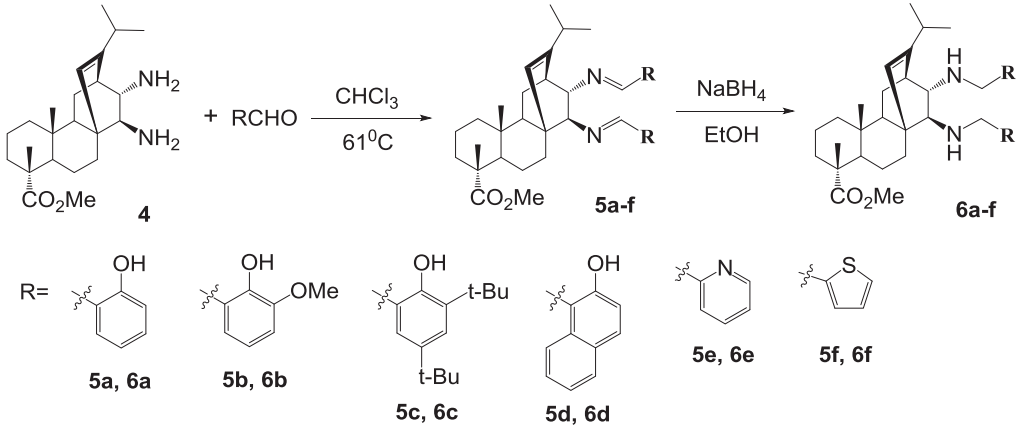


Fig. 5a-1. ^1H NMR spectrum of compound **5a**.

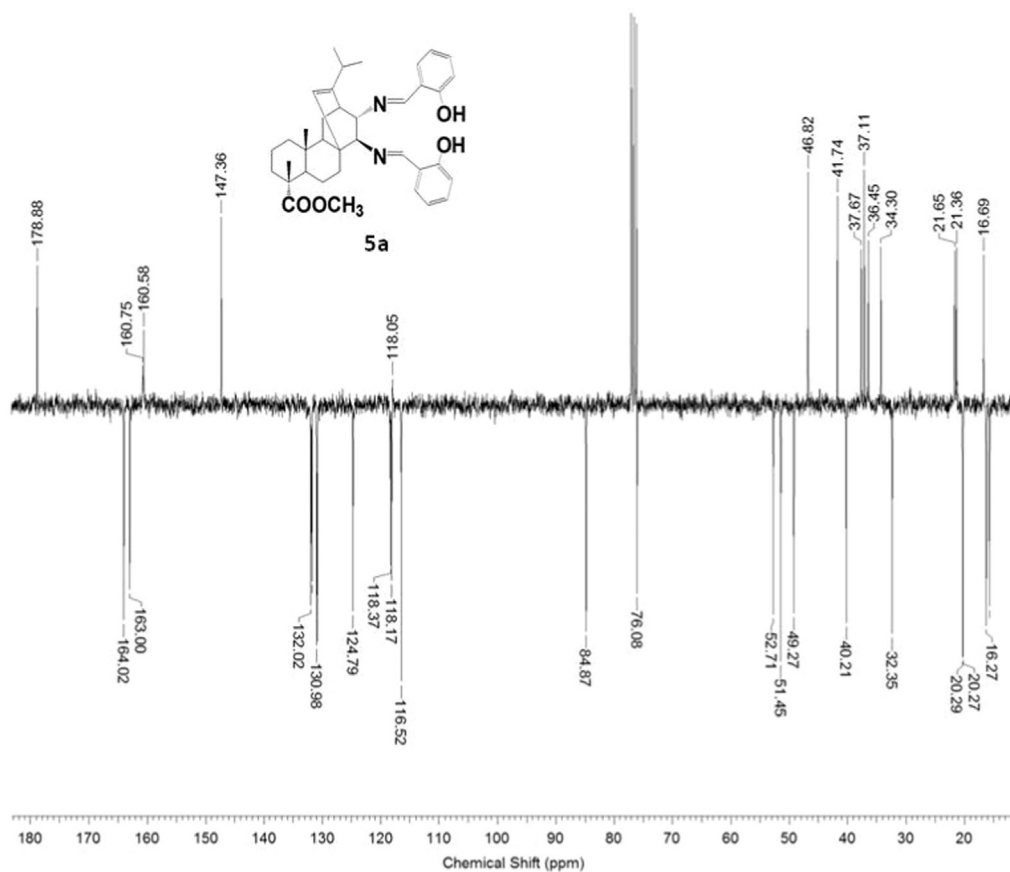
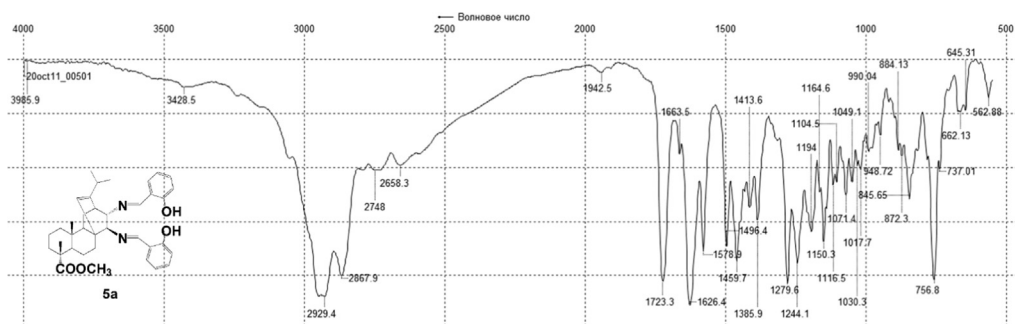
Fig. 5a-2. ^{13}C NMR spectrum of compound 5a.

Fig. 5a-3. IR spectrum of compound 5a.

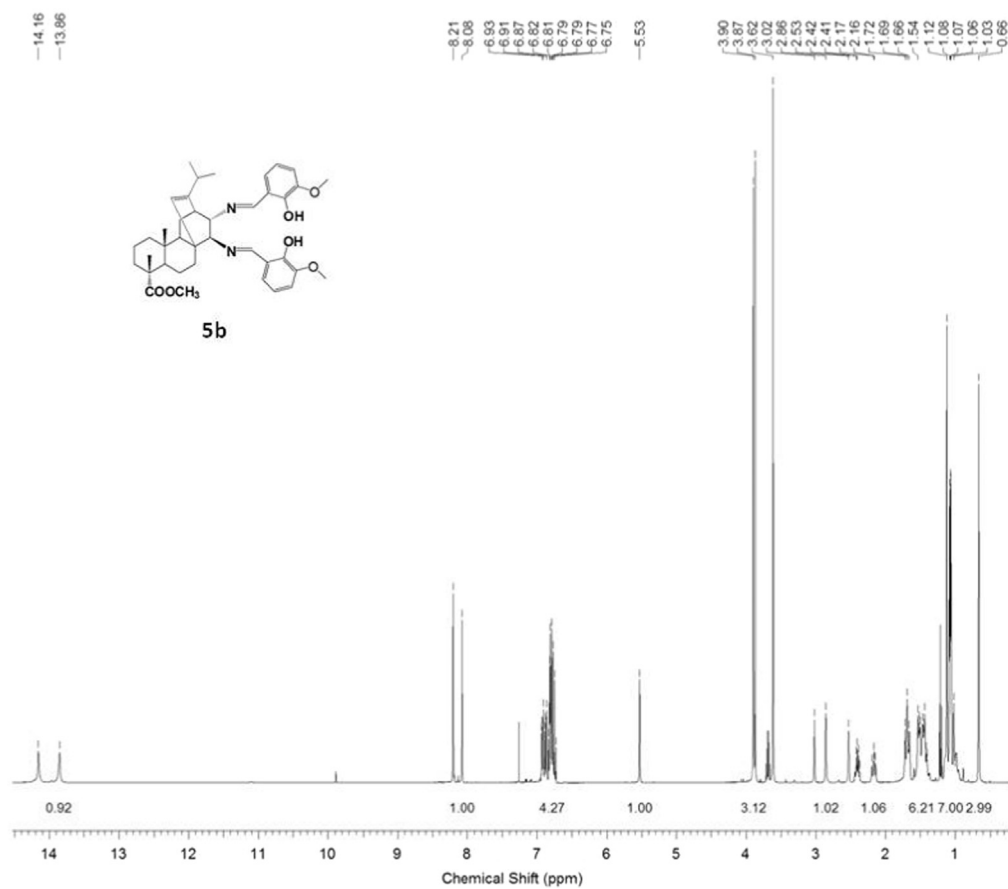


Fig. 5b-1. ^1H NMR spectrum of compound **5b**.

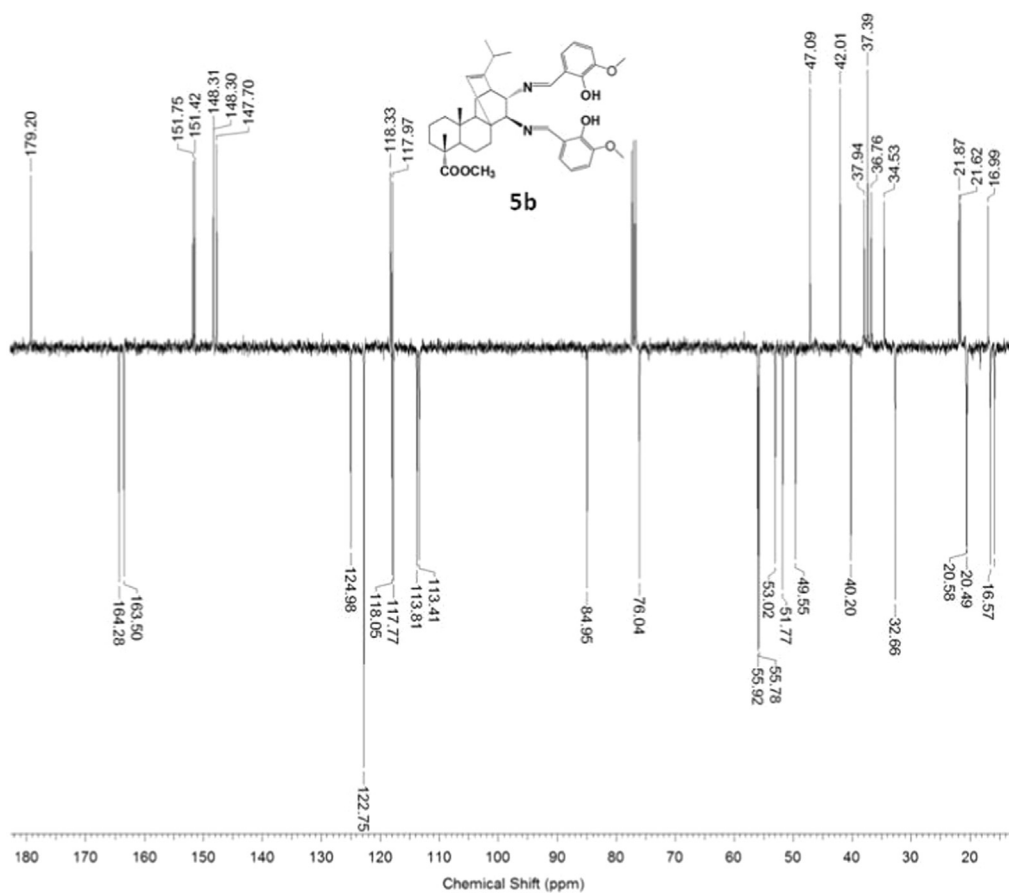
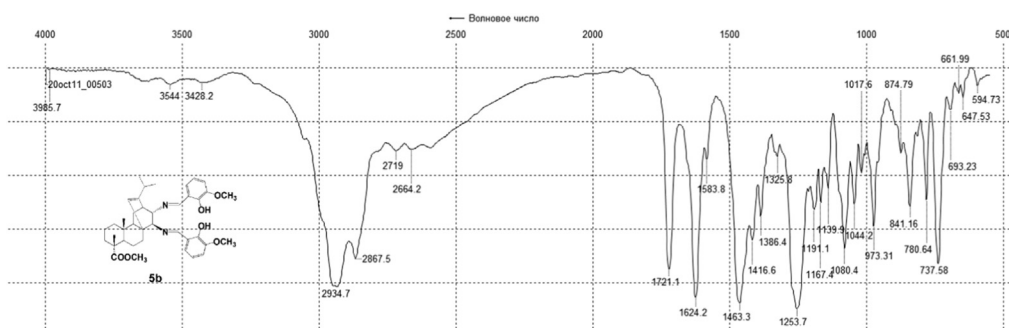
Fig. 5b-2. ^{13}C NMR spectrum of compound 5b.

Fig. 5b-3. IR spectrum of compound 5b.

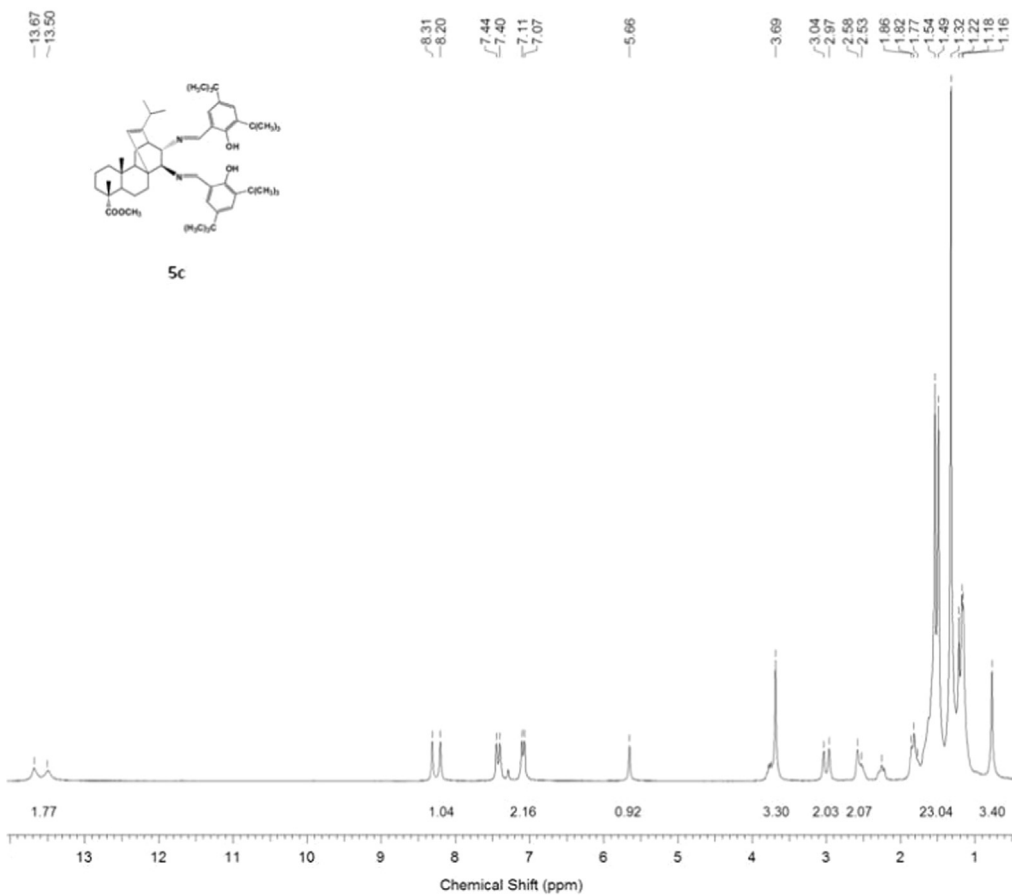


Fig. 5c-1. ¹H NMR spectrum of compound 5c.

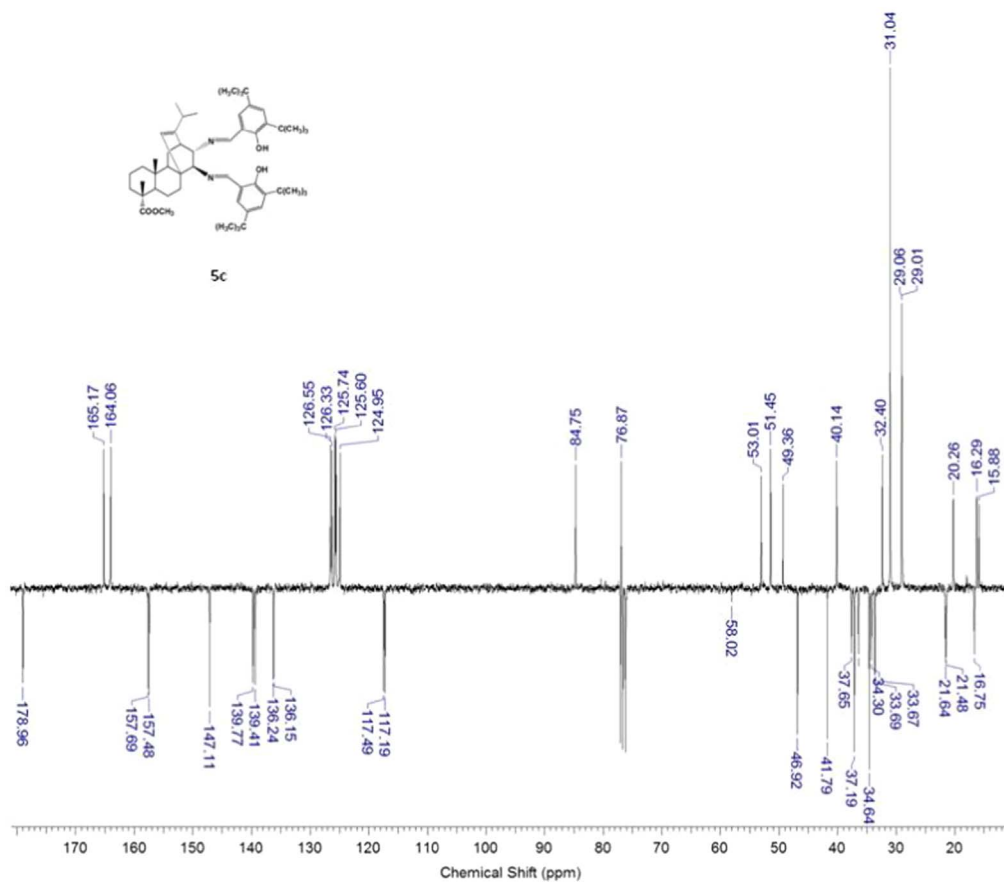
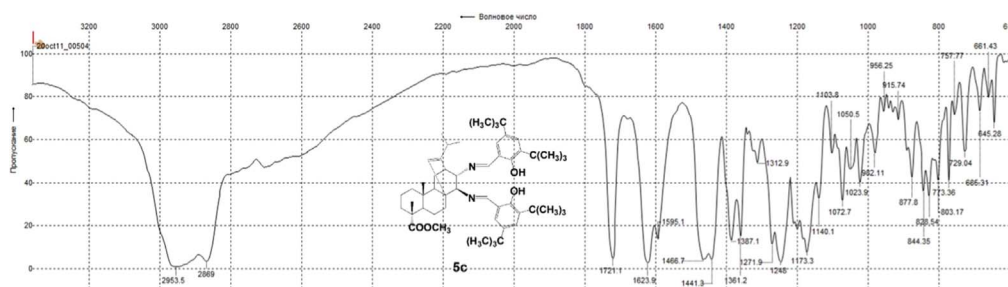


Fig. 5c-2. ^{13}C NMR spectrum of compound **5c**.



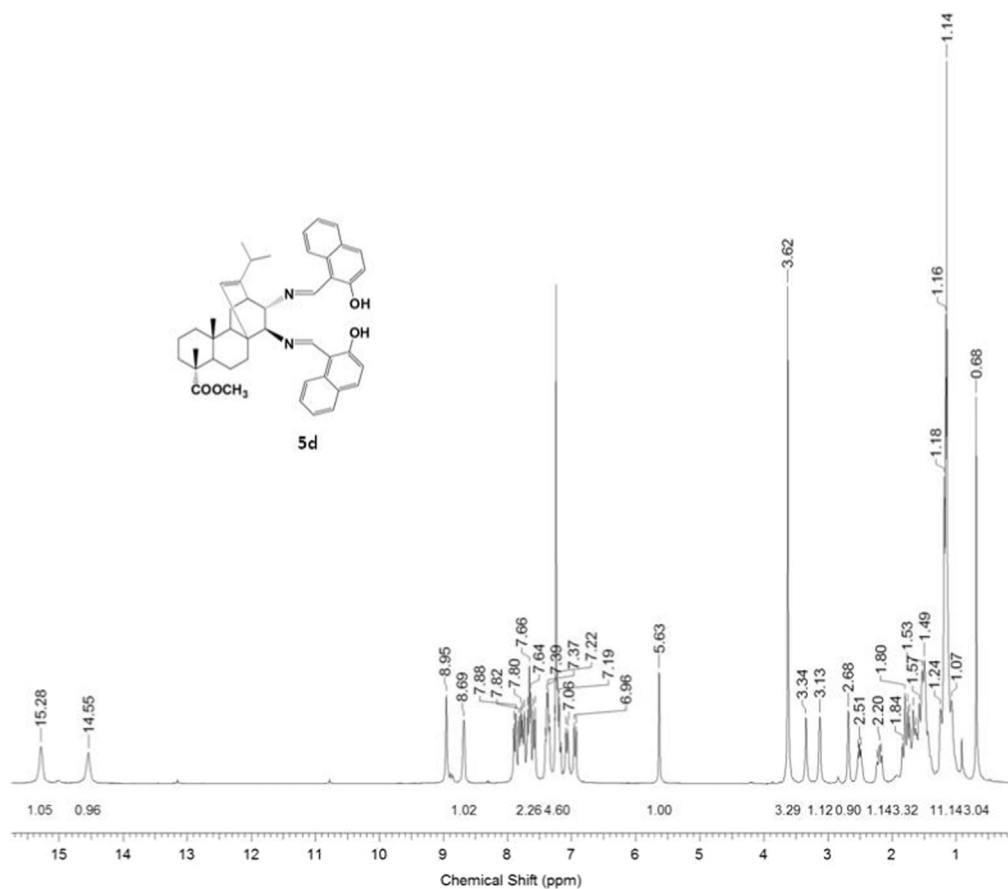
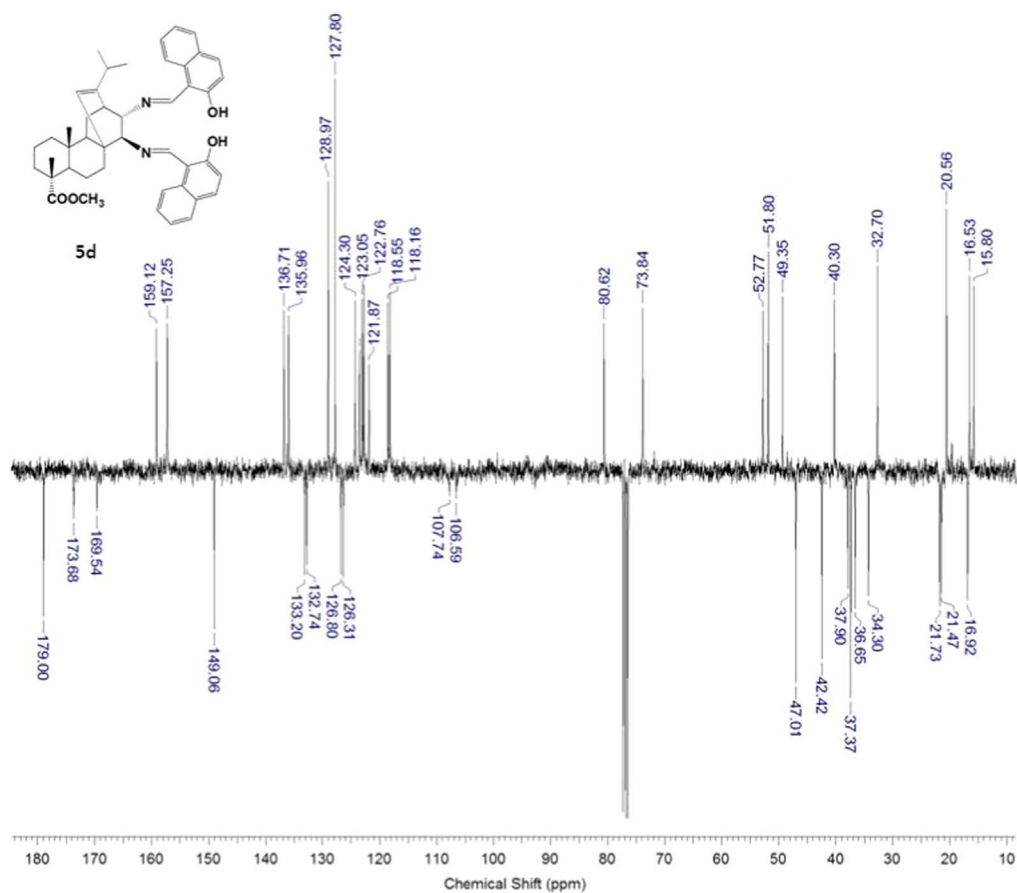
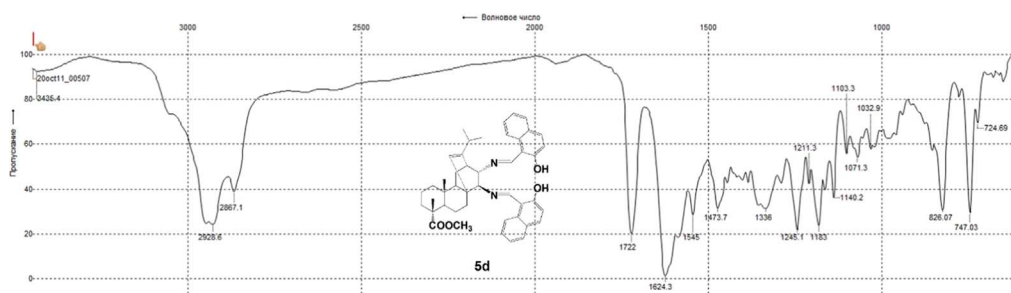


Fig. 5d-1. ¹H NMR spectrum of compound **5d**.

Fig. 5d-2. ^{13}C NMR spectrum of compound **5d**.Fig. 5d-3. IR spectrum of compound **5d**.

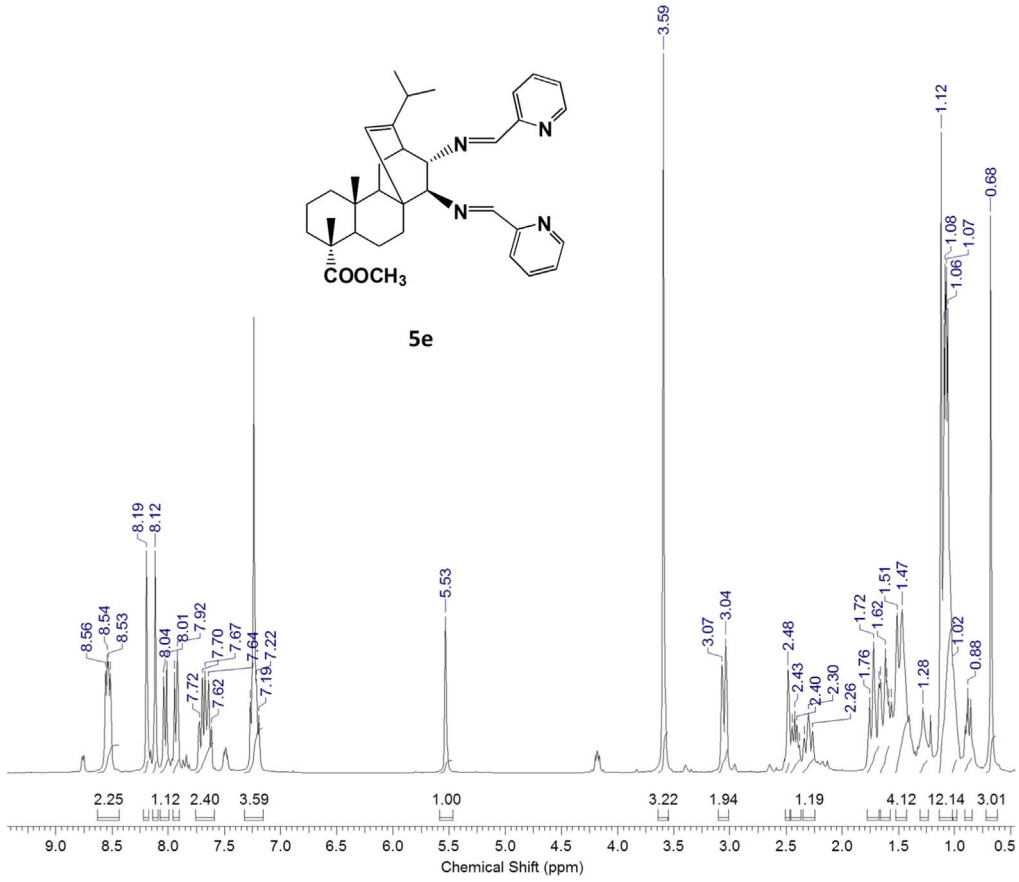


Fig. 5e-1. ¹H NMR spectrum of compound **5e**.

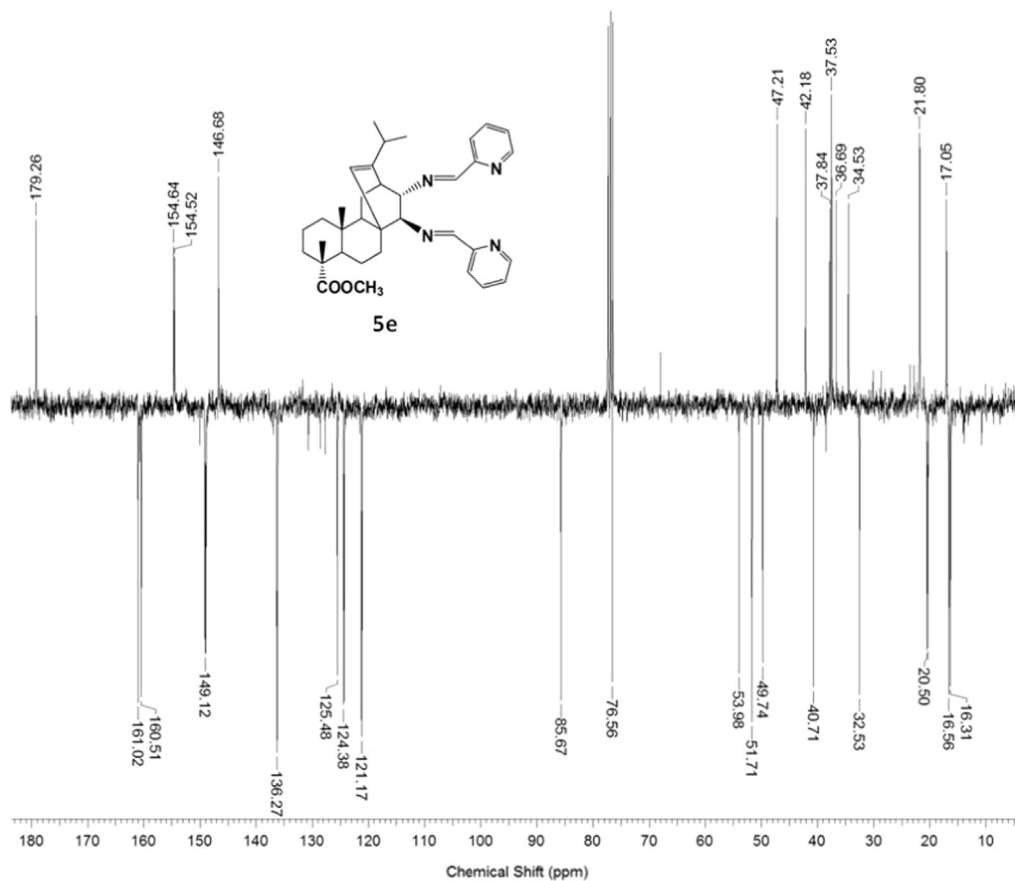
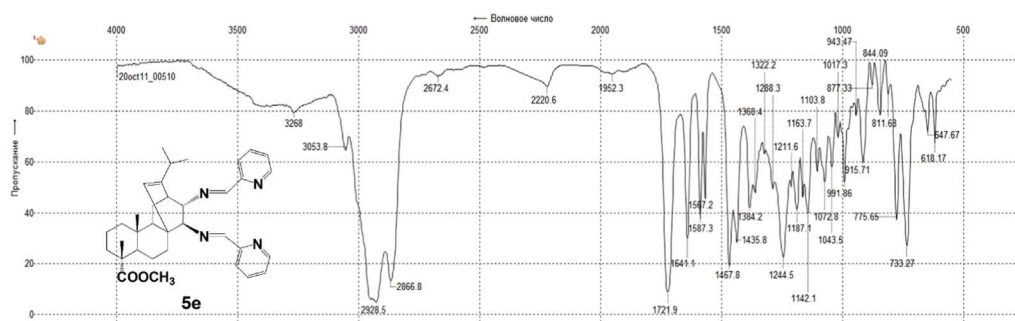
Fig. 5e-2. ¹³C NMR spectrum of compound 5e.

Fig. 5e-3. IR spectrum of compound 5e.

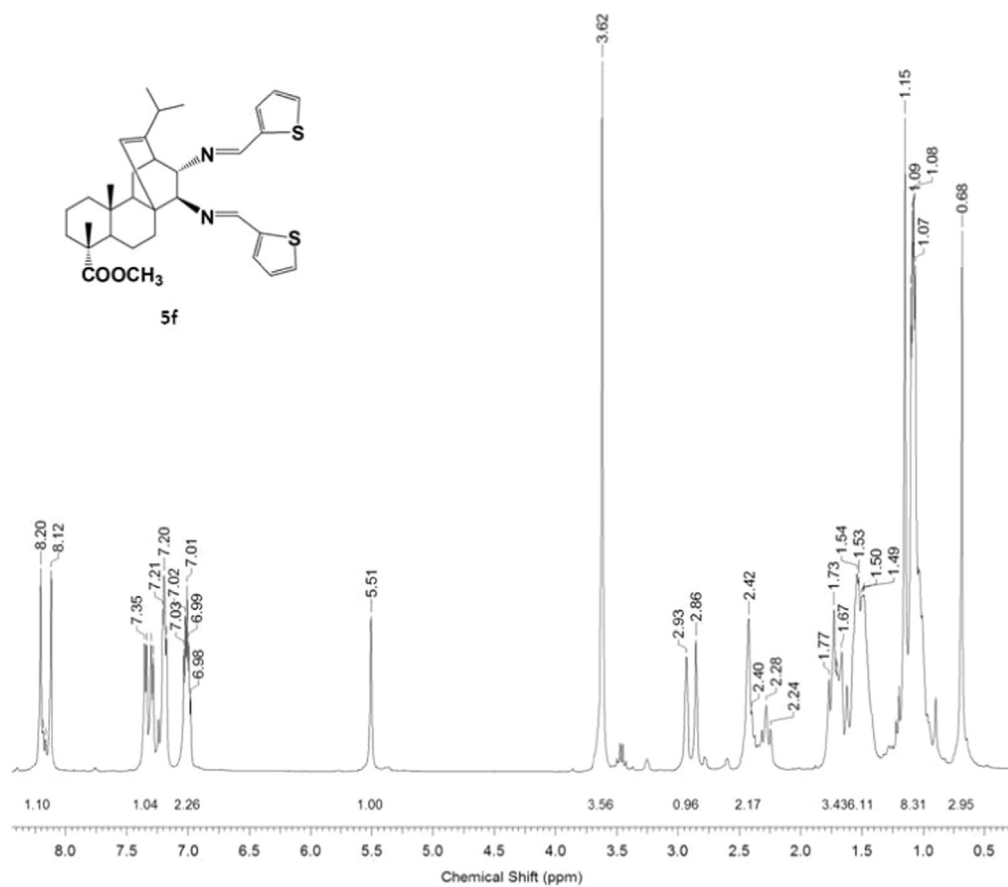


Fig. 5f-1. ¹H NMR spectrum of compound **5f**.

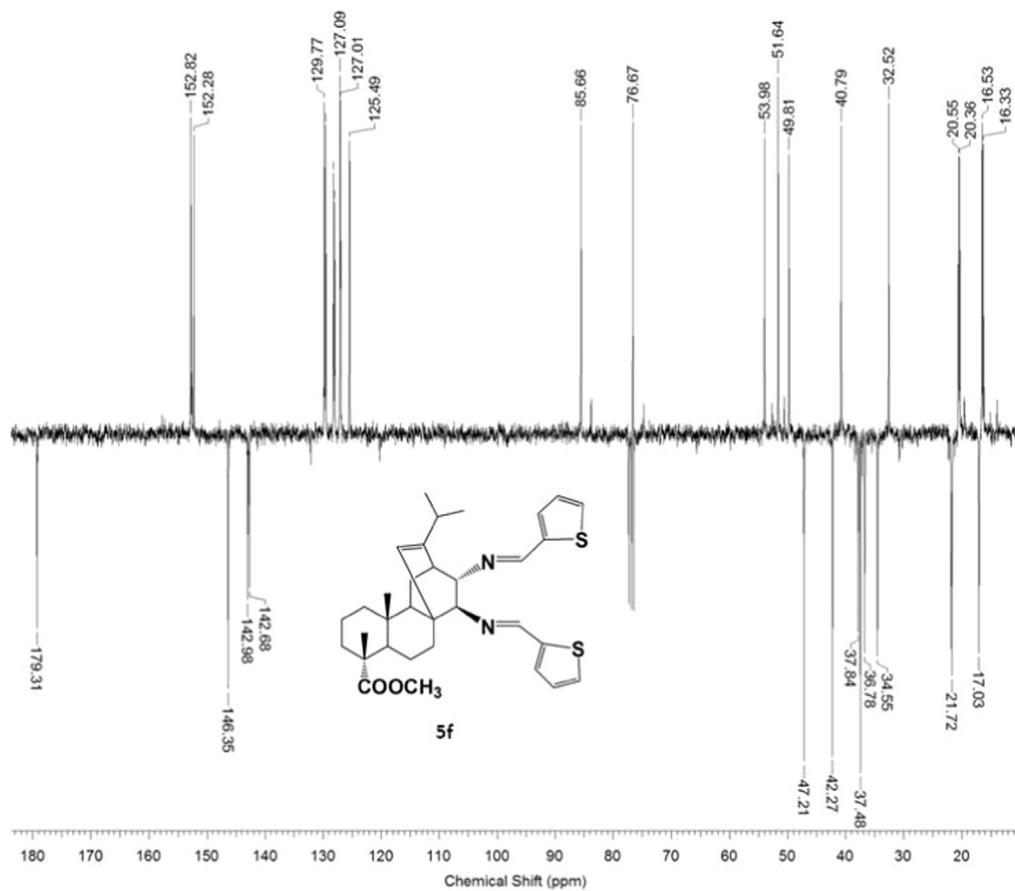
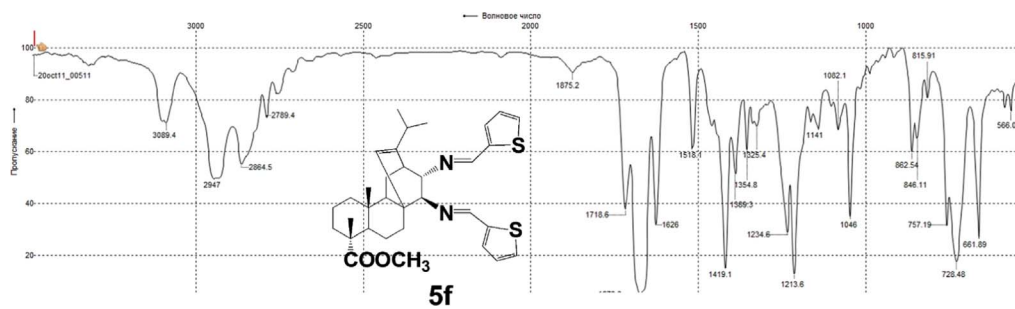
Fig. 5f-2. ¹³C NMR spectrum of compound 5f.

Fig. 5f-3. IR spectrum of compound 5f.

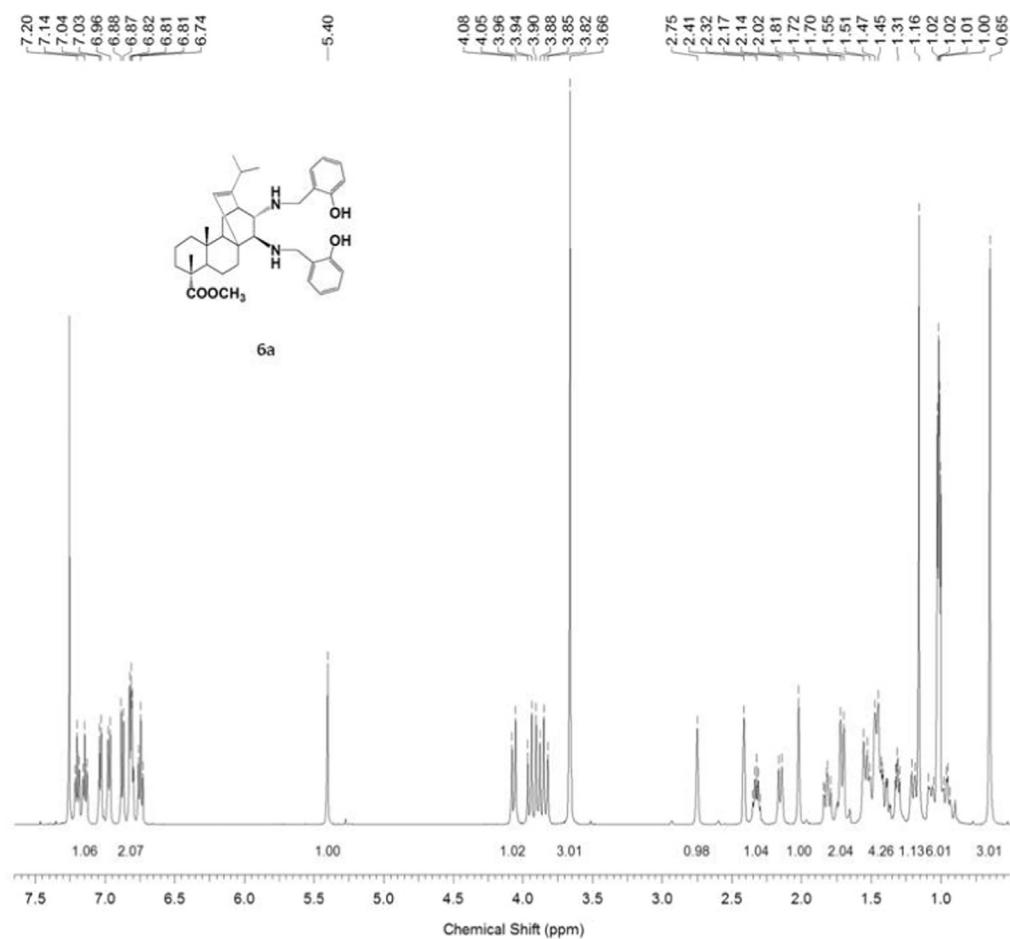


Fig. 6a-1. ^1H NMR spectrum of compound **6a**.

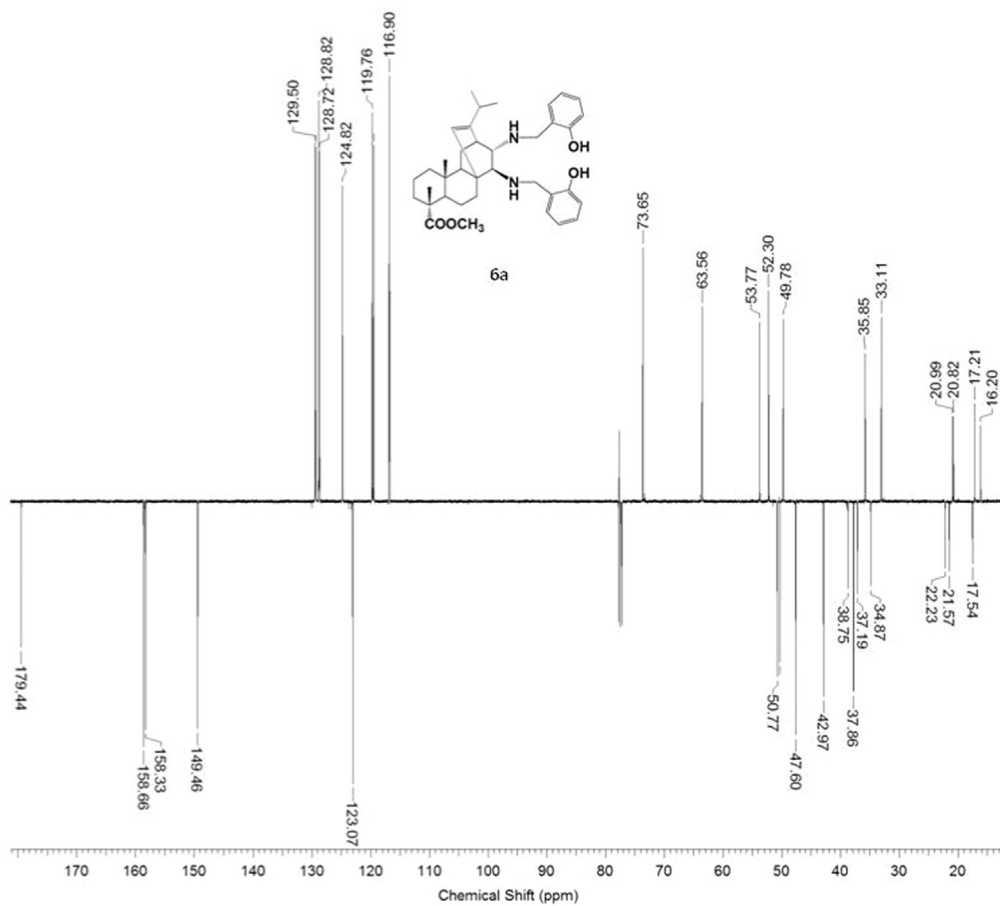
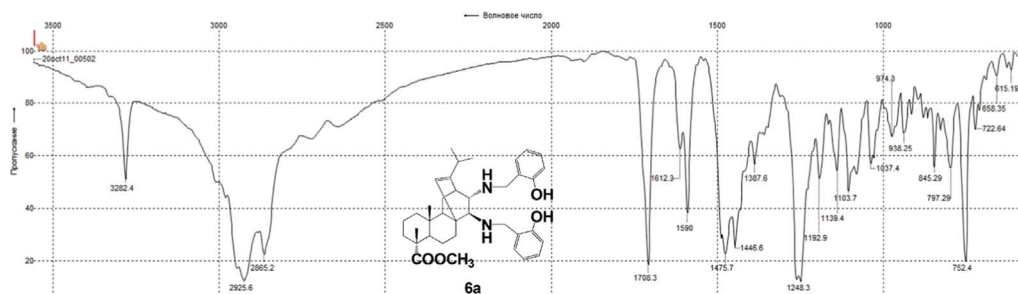
Fig. 6a-2. ^{13}C NMR spectrum of compound 6a.

Fig. 6a-3. IR spectrum of compound 6a.

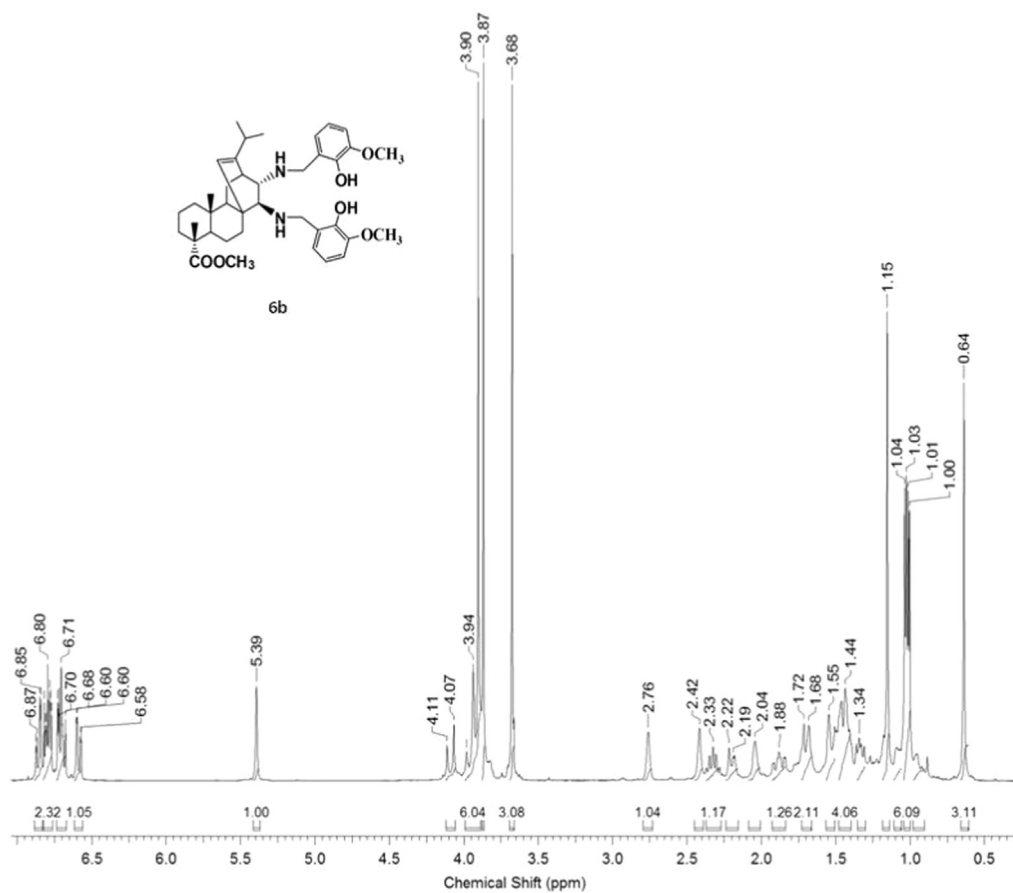


Fig. 6b-1. ^1H NMR spectrum of compound **6b**.

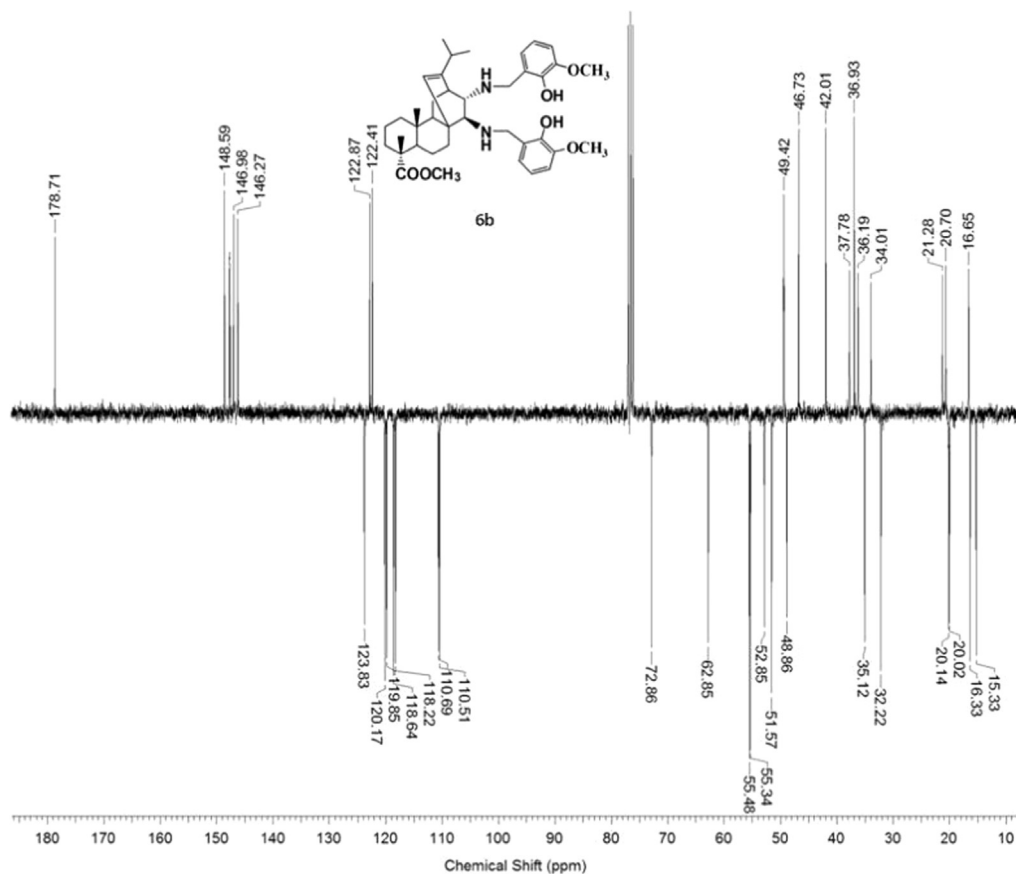
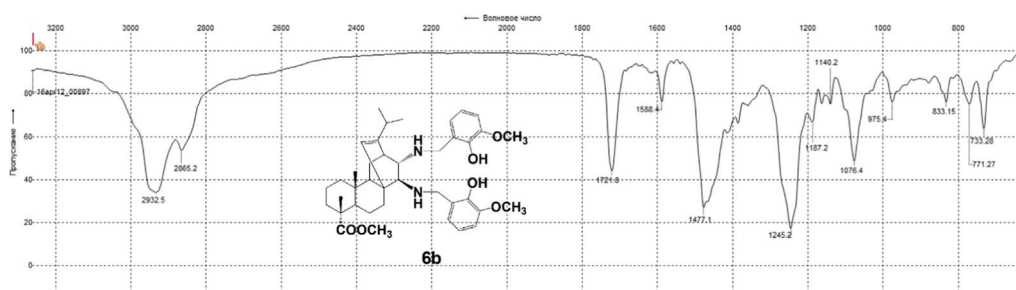
Fig. 6b-2. ¹³C NMR spectrum of compound 6b.

Fig. 6b-3. IR spectrum of compound 6b.

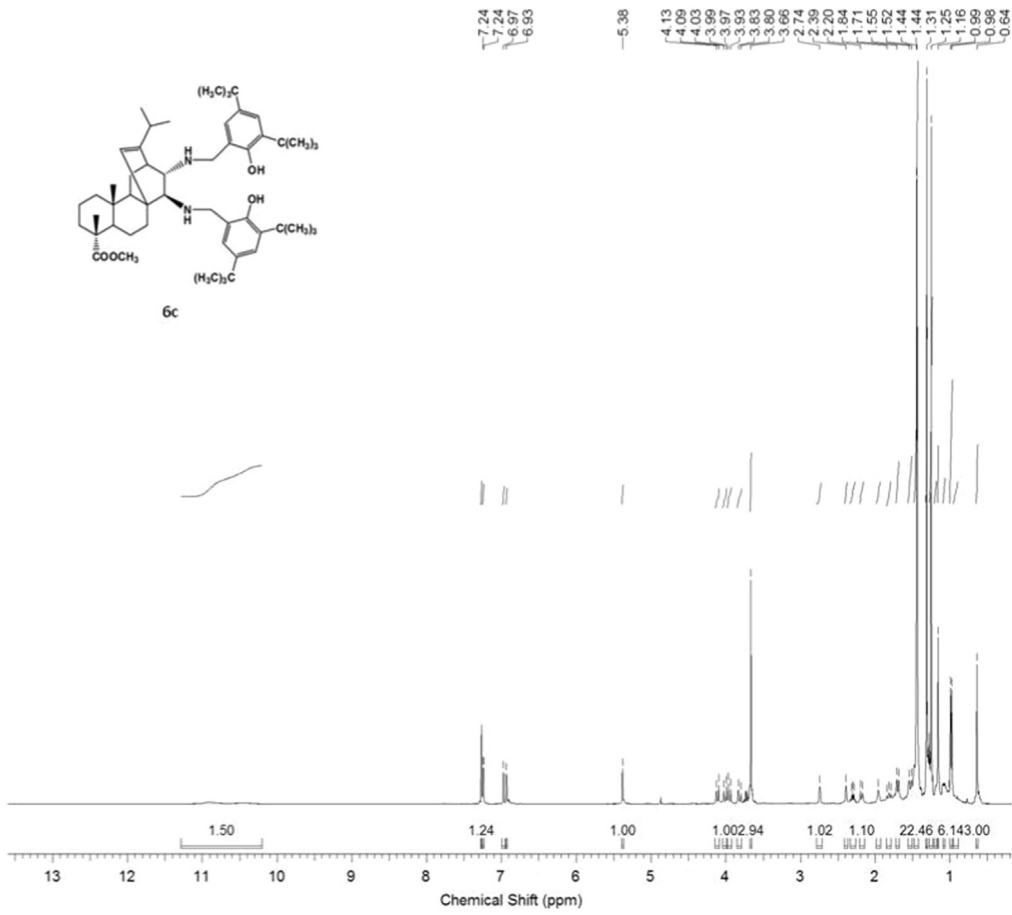
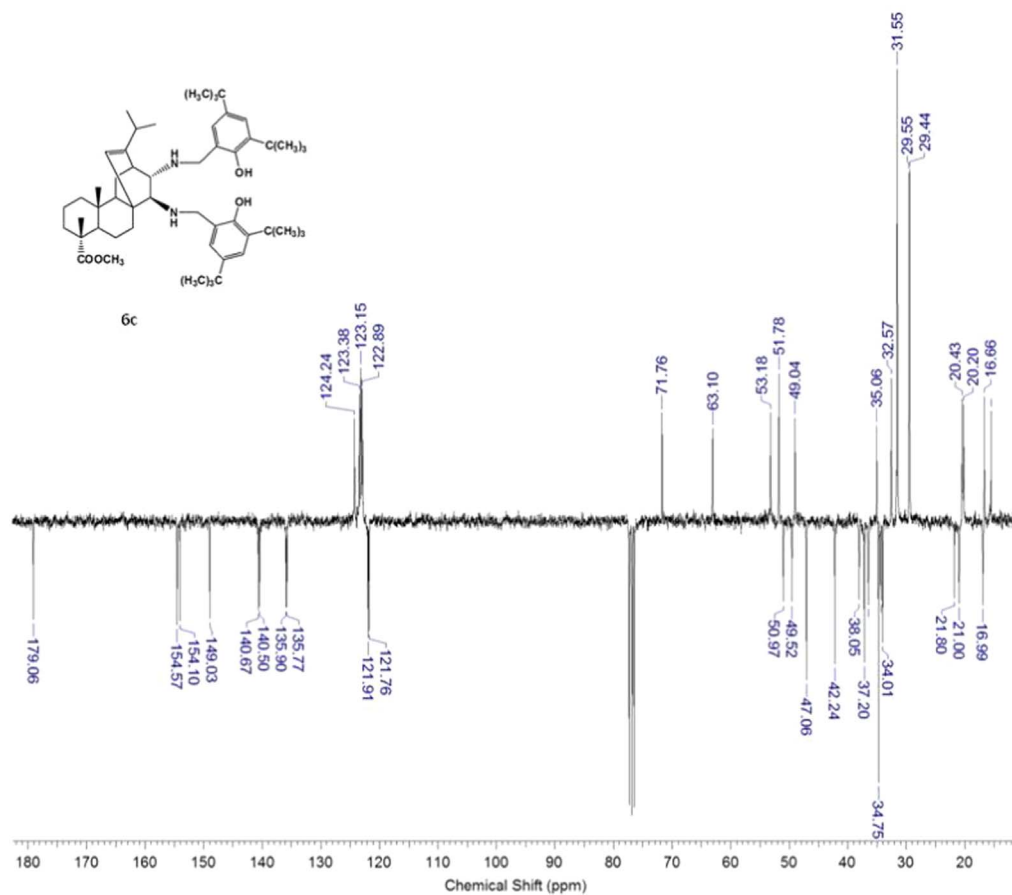
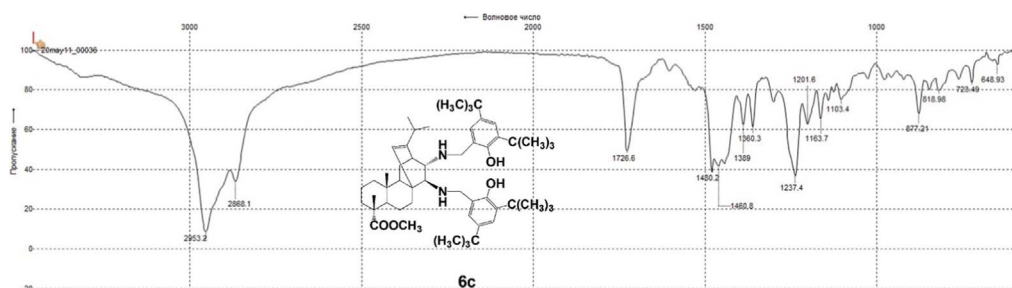


Fig. 6c-1. ^1H NMR spectrum of compound **6c**.

Fig. 6c-2. ^{13}C NMR spectrum of compound **6c**.Fig. 6c-3. IR spectrum of compound **6c**.

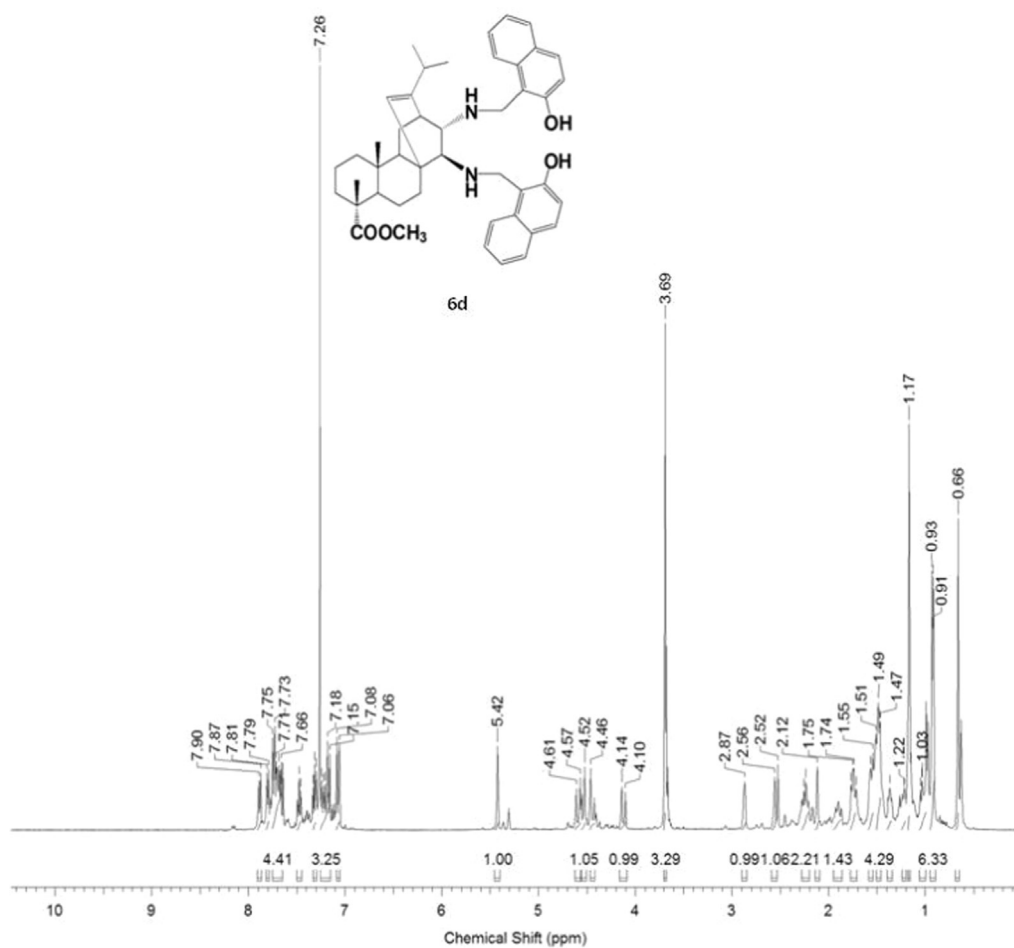


Fig. 6d-1. ^1H NMR spectrum of compound **6d**.

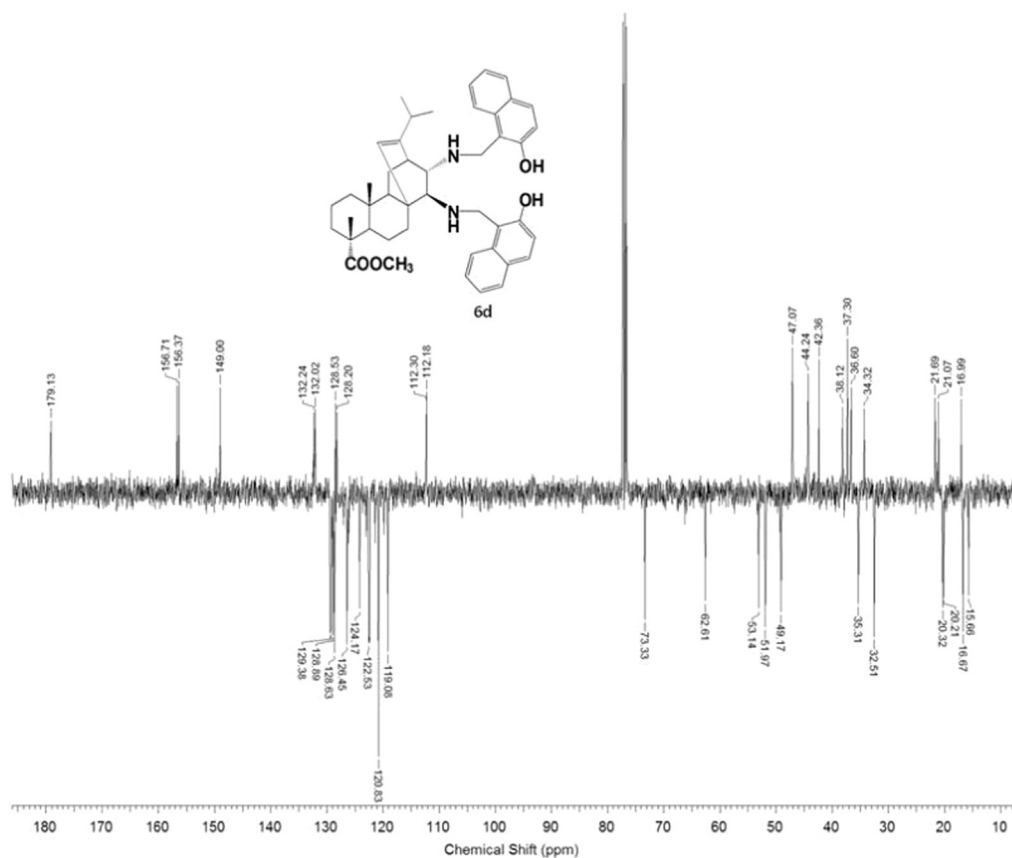
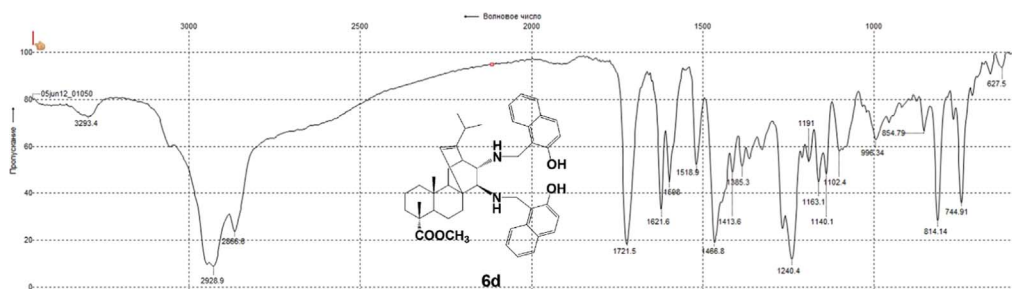
Fig. 6d-2. ^{13}C NMR spectrum of compound 6d.

Fig. 6d-3. IR spectrum of compound 6d.

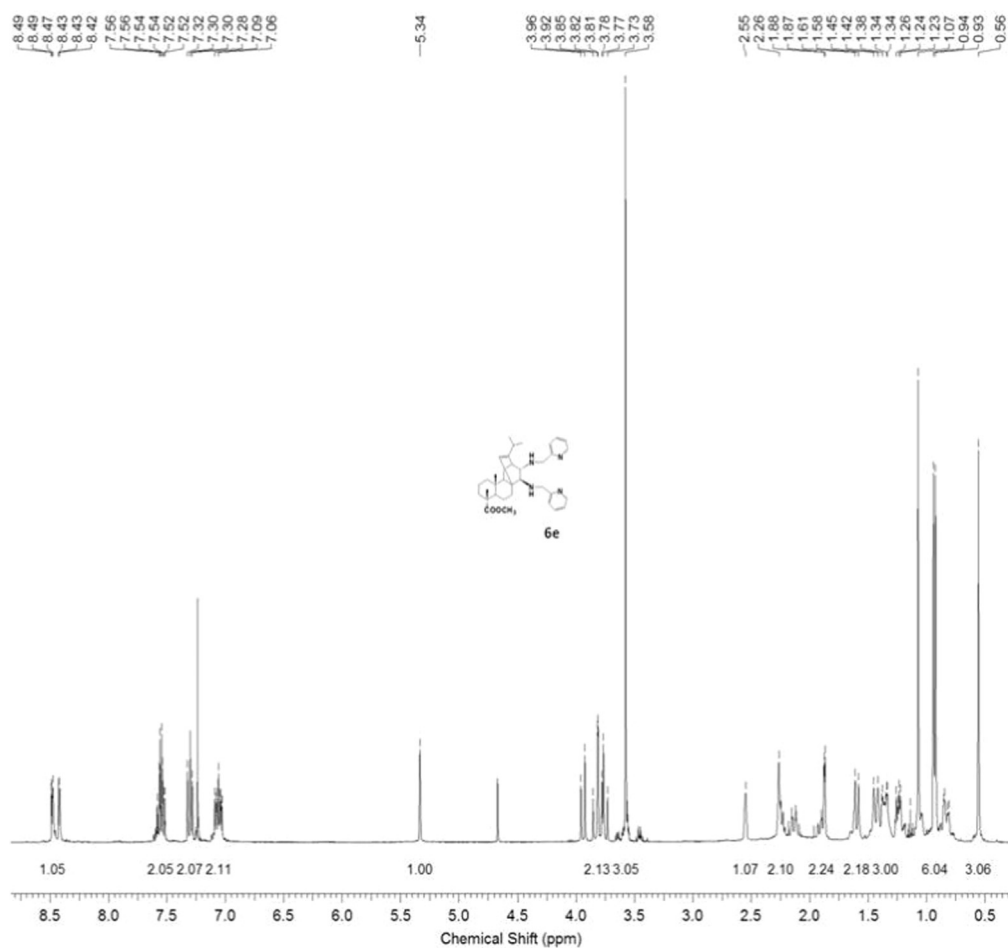


Fig. 6e-1. ^1H NMR spectrum of compound **6e**.

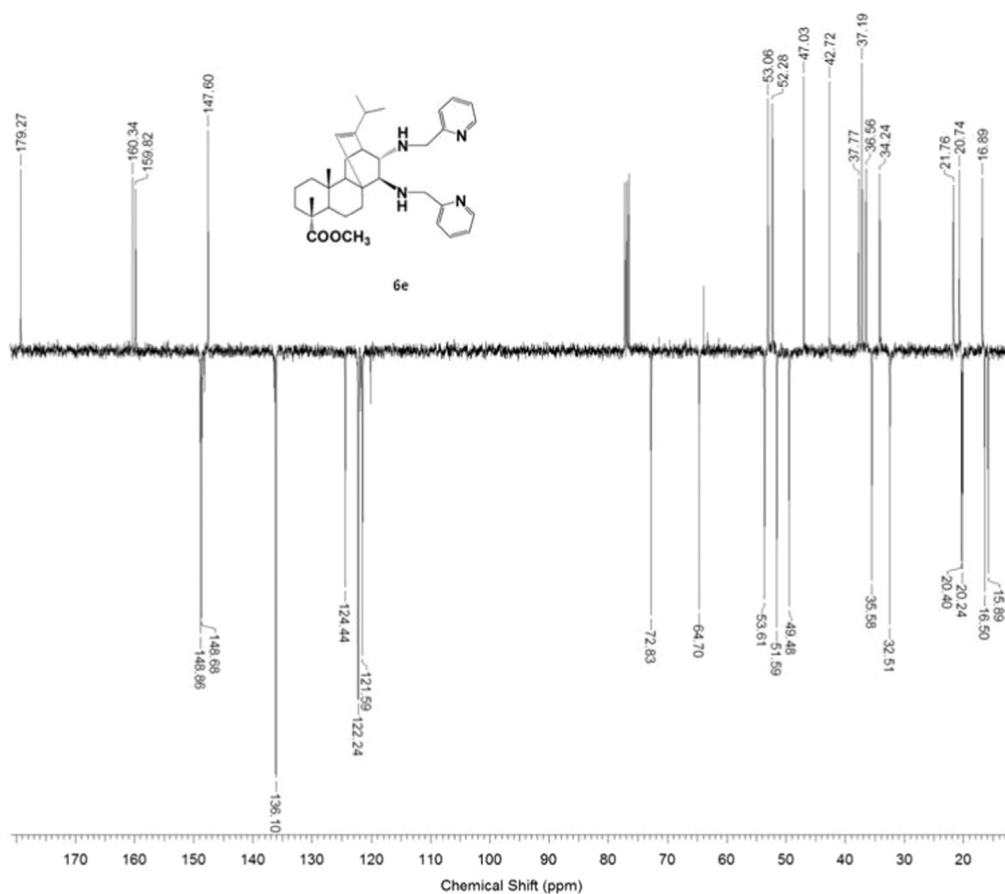
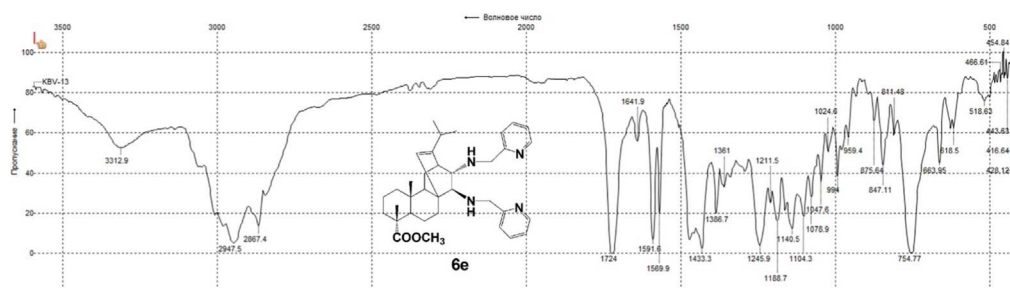
Fig. 6e-2. ^{13}C NMR spectrum of compound 6e.

Fig. 6e-3. IR spectrum of compound 6e.

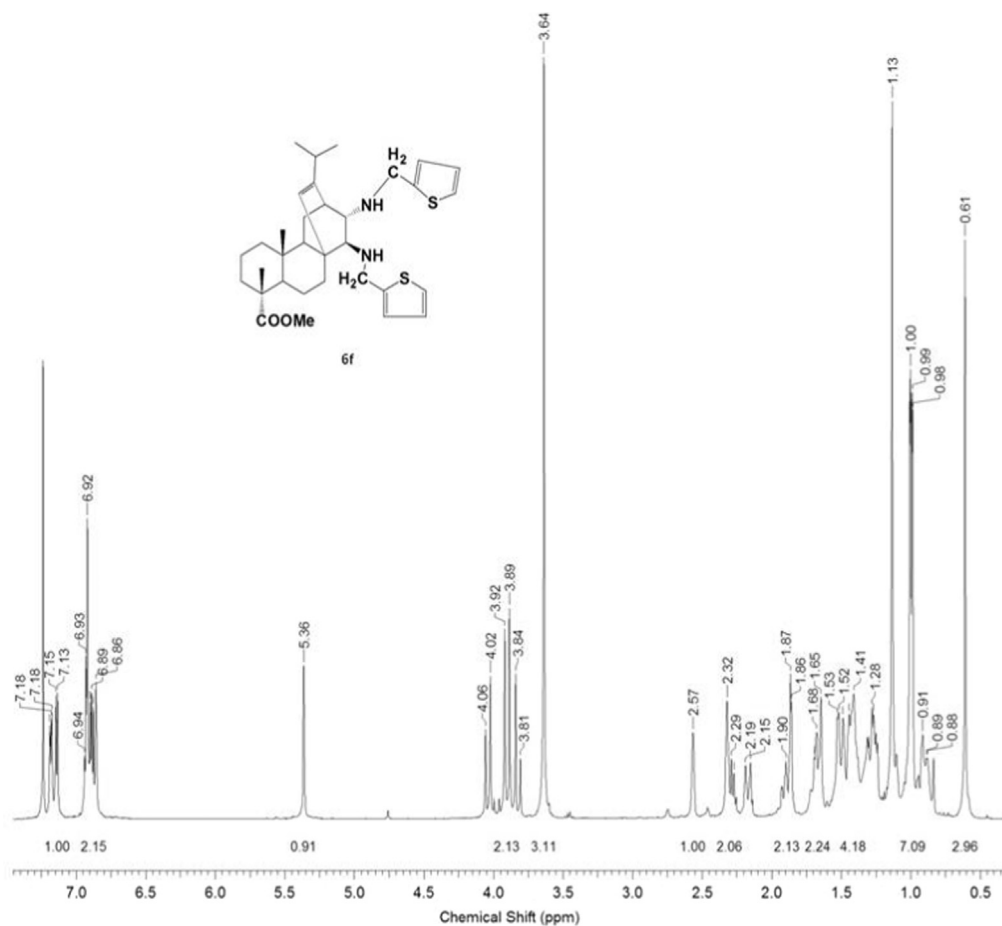


Fig. 6f-1. ¹H NMR spectrum of compound 6f.

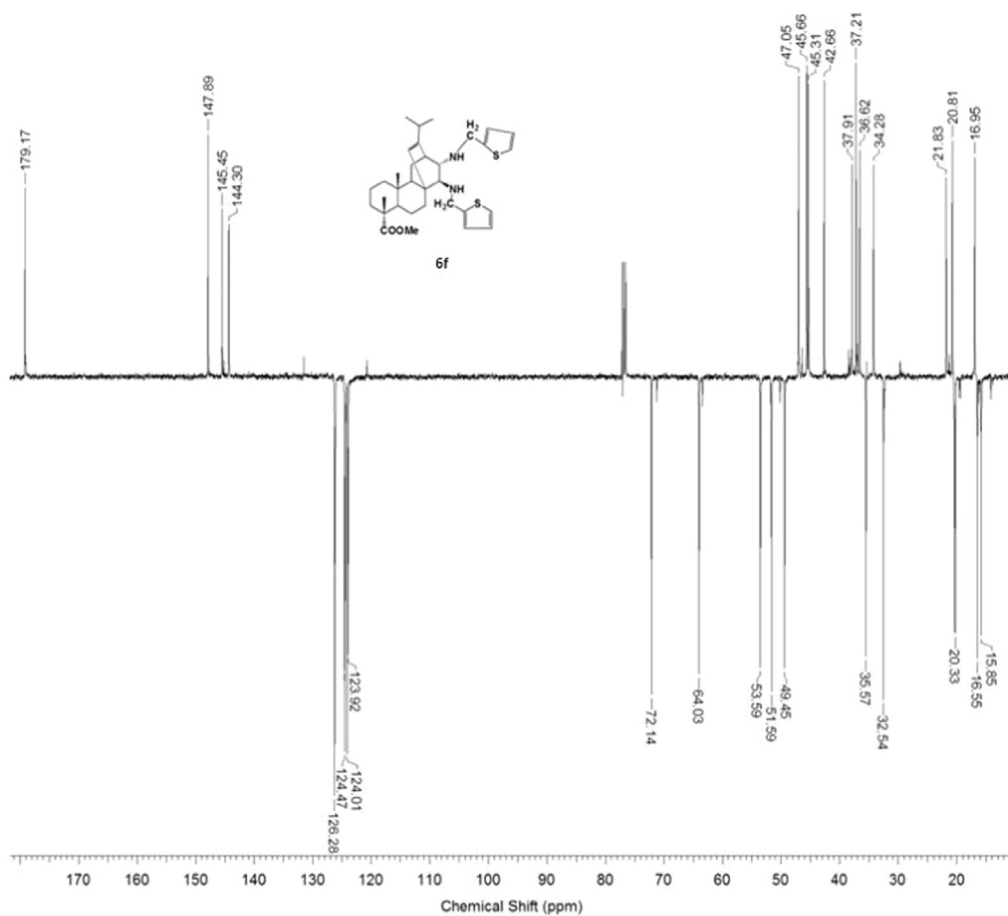
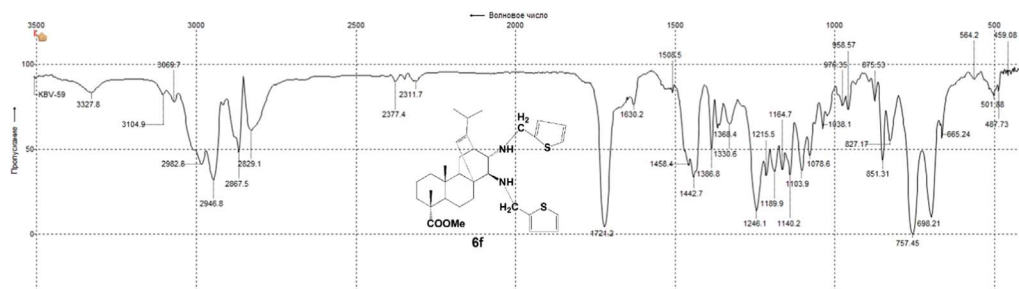
Fig. 6f-2. ¹³C NMR spectrum of compound 6f.

Fig. 6f-3. IR spectrum of compound 6f.

Acknowledgements

This work was carried out within the framework of the budget project N AAAA-A17-117041710081-1 for Boreskov Institute of Catalysis. We are grateful to Orgsyntez OJSC for supplying the pine oleoresin. We are grateful to Dr. L.V. Malysheva for IR measurements and Dr. S.A. Prihod'ko for GC–MS measurements.

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>.

References

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