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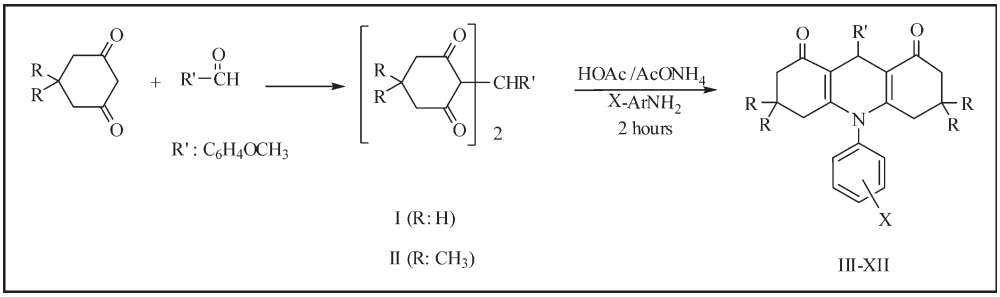

Synthesis of 10-(halophenyl)-9-(4-methoxyphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8-(2H,5H)-dione derivates have been prepared and their absorption, emission, and laser properties have been evaluated. The structures of all the synthesized compounds were characterized by spectroscopic methods IR, ^{1}H NMR, ^{13}C-APT, MS, and elemental analysis.

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INTRODUCTION

Many organic compounds demonstrated laser activity in the 310–1100 nm region [1]. These laser dyes have been classified as xanthene dyes, cyanine or polymethine dyes, linear and condensed polybenzoid compounds, and heterocyclic compounds. In the heterocyclic series, rhodamine, coumarin, and acridinedione have been known as efficient laser dyes. So far, the acridinedione ring system reported possesses laser activity in the 475–500 nm region [2–5]. The effectiveness of lasing can be controlled by substituents at the 9- and 10-positions of the acridine chromophore [6]. Therefore, acridinediones with substituents at 9- or 10-positions have already been synthesized by using different methods [7–13]. Photochemical properties of some acridinedione dyes were reported in literature [14–16]. In this study, we reported the synthesis of novel halogen substitute acridinediones. In addition to UV, fluorescence spectra and laser activity of the acridinedione dyes were examined (Table 1).

RESULTS AND DISCUSSION

The tetraketones were formed by the condensation of cyclohexane-1,3-dione or 5,5-dimethyl-1,3-cyclohexanedione with 4-methoxybenzaldehyde furnishing compounds **I** and **II**. Then, tetraketones were refluxed together various halo-anilines with acetic acid-ammonium acetate system in toluene for the syntheses of acridinediones (Scheme 1). Acridinediones were obtained with high yield (from 75 to 92%) and short reaction time 2 h data for all compounds. Physical and analytical data of compounds were given in Table 1.

The purity of the compounds was checked by using TLC. All of the products were characterized by IR, ^{1}H NMR, ^{13}C-APT, mass spectrometry, and elemental analyses.

All spectral data are in accordance with assigned structures. In IR spectra, aromatic C—H, aliphatic C—H, and C=O stretching bands were observed at expected values. In the ^{1}H NMR spectra, methyl protons were seen at ~0.85–1.10 ppm as two singlet peaks. Aromatic, methylene, methane, and OCH_3 protons were seen at expected values. The ^{13}C-APT spectra of the compounds displayed the number of resonance that fit exactly with the number of expected carbon resonances. Mass spectra of the compounds were taken using the chemical ionization (CI) technique. In general molecular ion peaks were seen in spectra and the base peaks were found by cleavage of the aryl ring from the parent molecule. The structure of compounds IX was further confirmed by an X-ray crystallographic analysis [17]. In summary, all compounds show good florescence and laser activity in chloroform ($1 \times 10^{-4}M$). Table 1 gives the absorption, emission, and laser activity of the dyes that have high

Table 1

Yields, melting points, UV, fluorescence, laser, and elemental analyses data for **III–XIV**.

Compound	X	R	Yield (%)	mp (°C) (Ref.)	λ_{UV} (nm)	λ_{Flu} (nm)	λ_{laser} (nm)	Molecular formula	Elemental analyses (%) Calc./Found C	H	N
III	4-F	H	79	265–266	272, 356	424	554	$C_{26}H_{24}FNO_3$	74.80	5.79	3.36
									74.51	5.77	3.34
IV	4-Cl	H	90	283, 285 [18]	238, 360	431	546	$C_{26}H_{24}ClNO_3$	71.97	5.57	3.23
									71.70	5.55	3.19
V	2-Cl	H	77	248	242, 361	443	548	$C_{26}H_{24}ClNO_3$	71.97	5.57	3.23
									71.89	5.49	3.22
VI	4-Br	H	92	217–219	243, 357	423	558	$C_{26}H_{24}BrNO_3$	65.28	5.06	2.93
									65.20	5.04	2.89
VII	4-I	H	85	242–243	247, 361	422	534	$C_{26}H_{24}INO_3$	59.44	4.60	2.67
									59.37	4.58	2.66
VIII	4-F	CH_3	77	230 (dec)	240, 358	423	540	$C_{30}H_{32}FNO_3$	76.08	6.81	2.96
									75.85	6.79	2.93
IX	2-F	CH_3	75	209	244, 364	433	535	$C_{30}H_{32}FNO_3$	76.08	6.81	2.96
									76.01	6.77	2.95
X	4-Cl	CH_3	80	220–221	248, 367	434	550	$C_{30}H_{32}ClNO_3$	73.53	6.58	2.86
									73.36	6.56	2.81
XI	2-Cl	CH_3	90	245	247, 366	421	530	$C_{30}H_{32}ClNO_3$	73.53	6.58	2.86
									73.45	6.56	2.82
XII	4-Br	CH_3	81	245, 247–248 [19]	250, 369	425	542	$C_{30}H_{32}BrNO_3$	67.41	6.03	2.62
									67.20	6.01	2.60
XIII	2-Br	CH_3	86	268	249, 369	427	530	$C_{30}H_{32}BrNO_3$	67.41	6.03	2.62
									67.28	5.97	2.61
XIV	4-I	CH_3	75	263, 259–263 [20]	240, 363	422	536	$C_{30}H_{32}INO_3$	61.97	5.55	2.41
									61.78	5.53	2.39

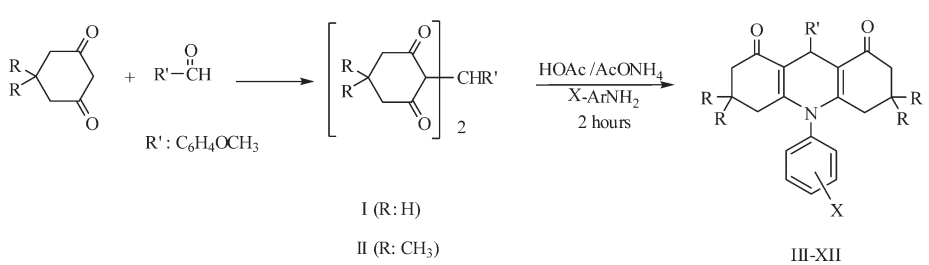
lasing efficiencies using chloroform as solvent. The tuning range for the dyes lies between 530 and 558 nm (Fig. 1).

EXPERIMENTAL

Melting points were measured on an Electrothermal 9100 apparatus. Absorption spectra were performed on an ATI Unicam UV-100 spectrophotometer. Infrared absorption spectra were recorded from a Mattson 1000-FTIR spectrometer, using KBr pellets. ^{1}H NMR (300 MHz) and ^{13}C-APT (75 MHz) spectra were obtained with a Bruker DPX-300 FT-NMR instrument with $CDCl_3$ as solvent. Mass spectra with CI were recorded on a AGILENT 1100 MSD instrument. The elemental analyses (C, H, N) were conducted using the Elemental Analyser LECO CHNS-932. Fluorescence spectra were obtained with Varian CARY Eclipse Fluorescence Spectrophotometer. The dye solutions (in a 2 cm × 2 cm quartz cell) were excited by using Ar ion laser; its wavelength was 488 nm and the pulse duration 6 ns and were detected by a diode. All measurements were performed in the presence of air at room temperature.

General procedure. The syntheses of compounds **I** and **II** were achieved according to the procedure described in the literature [20]. For that purpose, 4-methoxybenzaldehyde (3.40 g, 25 mmol) was added to the solution of cyclohexane-l,3-dione (5.60 g, 50 mmol) in aq. methanol (20 mL) and warmed until the solution became cloudy. The tetraketone started to separate out. Then, the reaction mixture was diluted with water to 250 mL and allowed to stand overnight; the tetraketone was collected by filtration and dried and recrystallized from methanol (**I:** yield 92%, mp 196°C; lit. mp for **I:** 196°C [20] and **II:** yield 90%, mp 142–143°C; lit. mp for **II:** 142–143°C [21], 125.5–126.5°C [21]).

Scheme 1



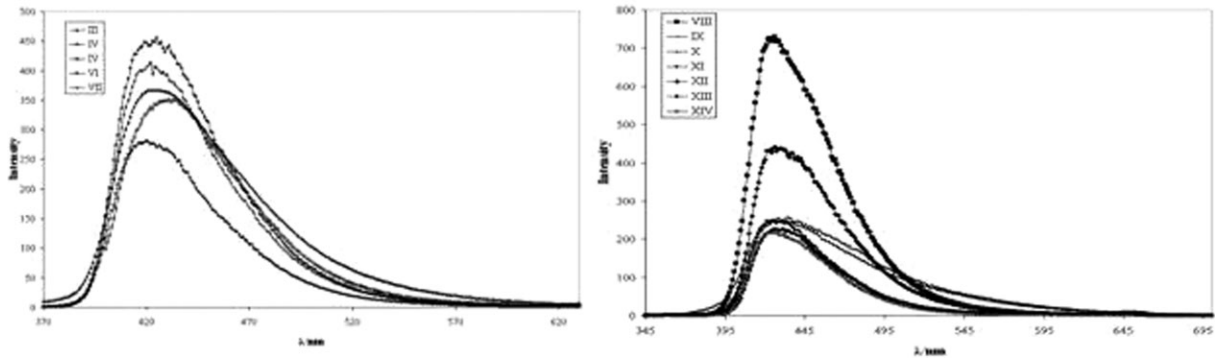


Figure 1. Fluorescence spectra of the dyes **III–XIV.**

10-(4-Fluorophenyl)-9-(4-methoxyphenyl)-3,4,6,7-hexadroacridine-1,8-(2*H*,5*H*-dione (III). The solution of 2,2'-((4-methoxyphenyl)methylene) dicyclohexane-1,3-dione **(I)** (1.03 g, 3 mmol), 4-fluorobenzenamine (0.34 g, 3.0 mmol), and excess amount of ammonium acetate were prepared with 20 mL toluene-acetic acid mixture (1:1). The formed solution was refluxed for 2 h by using a Dean-Stark apparatus. Then, the reaction mixture was poured into 100 mL water. Afterwards, last mixture was taken to a separation flask and 25 mL $CHCl_3$ was added. The organic phase was separated and evaporated. Halo-acridinedione **(III)** was separated from residue by thin layer chromatography and dried and then recrystallized from $CHCl_3$–MeOH (9:1). This compound was obtained as yellow solid, mp 265–266°C; IR: 3063 (Ar-H), 2954 (C—H), 1631 (C=O), 1585 (C=C) cm^{-1}. ^1H NMR: δ 1.73–1.97 (m, 4H, 2 × CH_2), 2.03–2.47 (m, 8H, 4 × CH_2), 3.77 (s, 3H, OCH_3), 5.32 (s, 1H, CH), 6.82 (d, 2H, J = 8.5 Hz, ArH), 7.18–7.36 (m, 6H, ArH). ^{13}C-APT: δ 21.10 (CH_2), 28.33 (CH_2), 31.18 (CH), 36.65 (CH_2), 55.18 (OCH_3), 113.63 (CH), 116.04 (C), 116.98 (CH), 129.33 (CH), 131.67 (CH), 135.07 (C), 138.79 (C), 151.30 (C), 160.90 (C), 164.23 (C), 196.14 (C). MS: *m*/*z* 417 (M^+), 291 (M^+-$C_6H_4FOCH_3$).

10-(4-Chlorophenyl)-9-(4-methoxyphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (IV). This compound was obtained as yellow solid (chloroform–methanol), mp 283°C Ref. [17] 285°C. IR: 3034 (Ar-H), 2947 (C—H), 1640 (C=O), 1573 (C=C) cm^{-1}. ^1H NMR: δ 1.76–2.00 (m, 4H, 2 × CH_2), 2.05–2.47 (m, 8H, 4 × CH_2), 3.78 (s, 3H, OCH_3), 5.34 (s, 1H, CH), 6.78 (d, 2H, J = 8.7 Hz, ArH), 7.20 (d, 2H, J = 8.5 Hz, ArH), 7.33 (d, 2H, J = 8.7 Hz, ArH), 7.54 (d, 2H, J = 8.5 Hz, ArH). ^{13}C-APT: δ 21.10 (CH_2), 28.33 (CH_2), 31.18 (CH), 36.67 (CH_2), 55.18 (OCH_3), 113.50 (CH), 116.07 (C), 128.66 (CH), 129.32 (CH), 130.80 (CH), 135.49 (C), 137.59 (C), 138.75 (C), 151.00 (C), 157.86 (C), 196.10 (C). MS: *m*/*z* 433 (M^+), 326 (M^+-$C_6H_4OCH_3$).

10-(2-Chlorophenyl)-9-(4-methoxyphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (V). This compound was obtained as yellow solid (chloroform–methanol), mp 248°C. IR: 3030 (Ar-H), 2940 (C—H), 1635 (C=O), 1575 (C=C) cm^{-1}. ^1H NMR: δ 1.66–1.94 (m, 4H, 2 × CH_2), 2.25–2.59 (m, 8H, 4 × CH_2), 3.68 (s, 3H, OCH_3), 4.56 (s, 1H, CH), 6.81 (d, 2H, J = 8.5 Hz, ArH), 7.28–7.38 (m, 6H, ArH). ^{13}C-APT: δ 21.18 (CH_2), 26.54 (CH_2), 31.45 (CH), 36.79 (CH_2), 55.12 (OCH_3), 113.57 (CH), 113.28 (CH), 116.04 (C), 128.01 (CH), 129.59 (CH), 130.83 (CH), 130.97 (CH), 134.87 (C), 137.14 (C), 139.15 (C), 150.89 (C), 157.78 (C), 196.10 (C). MS: *m*/*z* 433 (M^+), 326 (M^+-$C_6H_4OCH_3$), 215 (M^+-$C_{12}H_8ClOCH_3$).

10-(4-Bromophenyl)-9-(4-methoxyphenyl)-3,4,6,7,9,10-hexadroacridine-1,8-(2*H*,5*H*)-dione (VI). This compound was obtained as yellow solid (chloroform–methanol), mp 217–219°C. IR: 3031 (Ar-H), 2940 (C—H), 1638 (C=O), 1573 (C=C) cm^{-1}. ^1H NMR: δ 1.75–1.98 (m, 4H, 2 × CH_2), 2.20–2.47 (m, 8H, 4 × CH_2), 3.90 (s, 3H, OCH_3), 5.32 (s, 1H, CH), 6.80 (d, 2H, J = 8.7 Hz, ArH), 7.17 (d, 2H, J = 8.4 Hz, ArH), 7.32 (d, 2H, J = 8.7 Hz, ArH), 7.7 (d, 2H, J = 8.4 Hz, ArH). ^{13}C-APT: δ 21.10 (CH_2), 28.33 (CH_2), 31.18 (CH), 36.67 (CH_2), 55.18 (OCH_3), 113.52 (CH), 116.47 (C), 124.66 (C), 128.02 (CH), 131.20 (CH), 133.47 (CH), 137.59 (C), 138.75 (C), 151.00 (C), 157.86 (C), 196.10 (C). MS: *m*/*z* 477 (M^+), 370 (M^+-$C_6H_4OCH_3$), 215 (M^+-$C_{12}H_8BrOCH_3$).

10-(4-Iodophenyl)-9-(4-methoxyphenyl)-3,4,6,7,9,10-hexa-oacridine-1,8-(2*H*,5*H*)-dione (VII). This compound was obtained as yellow solid (chloroform–methanol), mp 242–243°C. IR: 3050 (Ar-H), 2940 (C—H), 1645 (C=O), 1580 (C=C) cm^{-1}. ^1H NMR: δ 1.72–1.96 (m, 4H, 2 × CH_2), 2.18–2.47 (m, 8H, 4 × CH_2), 3.78 (s, 3H, OCH_3), 5.29 (s, 1H, CH), 6.80 (d, 2H, J = 8.7 Hz, ArH), 7.04 (d, 2H, J = 8.4 Hz, ArH), 7.32 (d, 2H, J = 8.7 Hz, ArH), 7.88 (d, 2H, J = 8.4 Hz, ArH): ^{13}C-APT: δ 21.04 (CH_2), 28.36 (CH_2), 31.19 (CH), 36.41 (CH_2), 55.19 (OCH_3), 95.13 (C), 113.67 (CH), 116.12 (C), 128.67 (CH), 131.35 (CH), 138.49 (C), 138.71 (C), 139.24 (CH), 151.44 (C), 157.92 (C), 196.23 (C). MS: *m*/*z* 525 (M^+), 418 (M^+-$C_6H_4OCH_3$).

10-(4-Fluorophenyl)-9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (VIII). This compound was obtained as yellow solid (chloroform–methanol), dec. 230°C. IR: 3028 (Ar-H), 2944 (C—H), 1637 (C=O), 1580 (C=C) cm^{-1}. ^1H NMR: δ 0.86 (s, 6H, 2 × CH_3), 1.02 (s, 6H, 2 × CH_3), 1.80–2.06 (d, 4H, 2 × CH_2), 2.25–2.40 (d, 4H, 2 × CH_2), 3.83 (s, 3H, OCH_3), 4.71 (s, 1H, CH), 6.76 (d, 2H, J = 8.5 Hz, ArH), 7.20–7.37 (m, 6H, ArH). ^{13}C-APT: δ 26.92 (CH_3), 27.33 (CH_3), 29.30 (CH), 32.21 (C), 41.85 (CH_2), 50.10 (CH_2), 55.14 (OCH_3), 113.50 (CH), 115.07 (C), 123.47 (C), 128.77 (CH), 131.27 (CH), 133.40 (CH), 138.15 (C), 138.44 (C), 149.05 (C), 157.71 (C), 195.87 (C). MS: *m*/*z* 473 (M^+), 378 (M^+-C_6H_4F), 271 (M^+-$C_{12}H_8FOCH_3$).

10-(2-Fluorophenyl)-9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (IX). This compound was obtained as yellow solid (ethanol), mp. 209°C. IR: 3032 (Ar-H), 2944 (C—H), 1645 (C=O), 1575 (C=C) cm^{-1}. ^1H NMR: δ 0.85 (s, 6H, 2 × CH_3), 0.91 (s, 6H, 2 × CH_3), 1.64 (s, 2H, CH_2), 2.09–2.24 (m, 6H, 3 × CH_2), 3.75 (s, 3H, OCH_3), 5.21 (s, 1H, CH), 6.79 (d, 2H, J = 8.4 Hz, ArH), 7.27 (d, 1H, ArH), 7.49–7.52 (m, 4H, ArH), 7.68 (d, 1H, ArH). ^{13}C-APT: δ 26.80 (CH_3), 29.30 (CH_3), 30.96 (CH), 32.48 (C), 40.63 (CH_2), 50.76 (CH_2), 55.14 (OCH_3), 113.46 (CH), 115.07 (C), 116.75 (CH), 117.02 (CH), 128.78 (CH), 129.31 (CH), 131.37 (CH), 138.46 (C), 140.48 (C), 140.60 (C), 40.87 (C), 157.73 (C), 195.88 (C). MS: *m*/*z* 472 (M^+-H), 366 (M^+-$C_6H_4OCH_3$), 347 (M^+-$C_6H_4OCH_3F$).

10-(4-Chlorophenyl)-9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (X). This compound was obtained as yellow solid (chloroform–methanol), mp 220–221°C. IR: 3030 (Ar-H), 2940 (C—H), 1638 (C=O), 1580 (C=C) cm^{-1}. ^1H NMR: δ 0.9 (s, 6H, 2 × CH_3), 1.1 (s, 6H, 2 × CH_3), 2.09–2.36 (m, 8H, 4 × CH_2), 3.73 (s, 3H, OCH_3), 4.71 (s, 1H, CH), 6.77 (d, 2H, J = 8.5 Hz, ArH), 7.16 (d, 2H, J = 8.5 Hz, ArH), 7.25 (d, 2H, J = 8.6 Hz, ArH), 7.46 (d, 2H, J = 8.6 Hz, ArH). ^{13}C-APT: δ 27.33 (CH_3), 29.27 (CH_3), 30.97 (CH), 32.20 (C), 40.86 (CH_2), 50.77 (CH_2), 55.11 (OCH_3), 113.48 (CH), 115.79 (C), 121.03 (C), 123.04 (CH), 129.00 (CH), 129.30 (CH), 136.45 (C), 158.20 (C), 162.11 (C), 168.00 (C), 196.58 (C). MS: *m*/*z* 489 (M^+), 271 (M^+-$C_{12}H_8ClOCH_3$), 256 (M^+-$C_{12}H_8ClOCH_3CH_3$).

10-(2-Chlorophenyl)-9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (XI). This compound was obtained as yellow solid (ethanol), mp. 245°C. IR: 3042 (Ar-H), 2955 (C—H), 1640 (C=O), 1585 (C=C) cm^{-1}. ^1H NMR: δ 0.84 (s, 6H, 2 × CH_3), 0.92 (s, 6H, 2 × CH_3), 1.63 (s, 2H, CH_2), 2.05–2.20 (d, 6H, 3 × CH_2), 3.74 (s, 3H, OCH_3), 5.17 (s, 1H, CH), 6.77 (d, 2H, J = 8.7 Hz, ArH), 7.32 (d, 1H, ArH), 7.45–7.52 (m, 4H, ArH), 7.63 (d, 1H, ArH). ^{13}C-APT: δ 26.28 (CH_3), 30.11 (CH_3), 32.46 (C), 32.84 (CH), 41.77 (CH_2), 50.01 (CH_2), 55.09 (OCH_3), 113.21 (CH), 115.04 (C), 128.79 (CH), 129.67 (CH), 130.80 (CH), 130.91 (CH), 131.17 (CH), 134.70 (C), 136.57 (C), 138.72 (C), 148.31 (C), 157.65 (C), 195.81 (C). MS: *m*/*z* 489 (M^+), 474 (M^+-CH_3), 367 (M^+-$CH_3C_6H_4OCH_3$).

10-(2-Bromophenyl)-9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2*H*,5*H*)-dione (XIII). This compound was obtained as yellow solid (ethanol), mp. 268°C. IR: 3049 (Ar-H), 2955 (C—H), 1660 (C=O), 1595 (C=C) cm^{-1}. ^1H NMR: δ 0.87 (s, 6H, 2 × CH_3), 0.94 (s, 6H, 2 × CH_3), 1.77 (s, 2H, CH_2), 1.92–2.07 (d, 2H, CH_2), 2.35–2.47 (d, 4H, 2 × CH_2), 3.70 (s, 3H, OCH_3), 5.20 (s, 1H, CH), 6.77 (d, 2H, J = 8.5 Hz, ArH), 7.31 (d, 1H, ArH), 7.42–7.60 (m, 4H, ArH), 7.85 (d, 1H, ArH). ^{13}C-APT: δ 26.79 (CH_3), 27.30 (CH_3), 29.75 (CH), 31.82 (C), 41.84 (CH_2), 50.76 (CH_2), 55.15 (OCH_3), 113.60 (CH), 115.02 (C), 126.43 (C), 129.14 (CH), 130.70 (CH), 132.18 (CH), 132.45 (CH), 134.51 (CH), 138.21 (C), 138.47 (C), 147.82 (C), 157.49 (C), 195.82 (C). MS: *m*/*z* 533 (M^+), 426 (M^+-$C_6H_4OCH_3$), 347 (M^+-$C_6H_4OCH_3Br$).

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