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# **Condensations of γ-Acetylacetonephosphonium Ylide (1-(Triphenylphosphoranylidene) pentane-2,4-dione) with Carbonyl Compounds**

# Cornelis M. Moorhoff\*

Department of Chemistry, University of Tasmania, GPO Box 252-75C, Hobart, Tasmania, Australia 7001

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**Summary.** 2,4-Dioxopentyltriphenylphosphonium bromide reacted with aqueous sodium hydrogencarbonate in dichloromethane to give, in almost quantitative yield, 1-(triphenylphosphoranylidene) pentane-2,4-dione. Dianion *Wittig-* and *Michael Wittig* condensations of the last mentioned phosphonium ylide with carbonyl compounds gave unsaturated 2,4-diketones which were almost totally enolised in deutero chloroform as 4-hydroxy-2-oxo-3,5-dienes (24–70% yields). These products closely resembled the type of compounds derived from similar condensations with alkyl 3-oxo-4-(triphenylphosphoranylidene)butanoate.

**Keywords.**  $\gamma$ -Acetylacetonephosphonium ylide; 1-(Triphenylphosphoranylidene)pentane-2,4-dione; Unsaturated 2,4-diketone; 4-Hydroxy-2-oxo-3,5-diene; 4-Hydroxy-2-oxo-3,5,7-triene; *Michael Wittig* condensation.

## Introduction

Perhaps it is not surprising that  $\gamma$ -acetylacetonephosphonium ylide (1-(triphenylphosphoranylidene)pentane-2,4-dione) (**2a**) is unrepresented in the large arsenal of phosphonium ylides (Scheme 1). A literature search revealed that only the chloride salt **1a** has been prepared *via* a detour [1]. We prepared the bromide salt **1b** [2] and its treatment with aqueous sodium hydrogencarbonate in dichloromethane gave the stable  $\gamma$ -acetylacetonephosphonium ylide **2a**. This new phosphonium ylide has the potential to become a very useful compound for the transformation of carbonyl compounds **3** into  $\gamma$ , $\delta$ -unsaturated- $\beta$ -diketones (unsaturated 2,4-diketones) **4** (Scheme 1).

<sup>\*</sup> E-mail: cmoorhoff@boronmolecular.com

Present address: Boron Molecular Pty. Ltd., P.O. Box 756, Noble Park, Victoria, Australia 3174



Scheme 1

# **Results and Discussion**

As can be expected, this phosphonium ylide 2a showed similarities with esters of  $\gamma$ -acetoacetatephosphonium ylide (alkyl 3-oxo-4-(triphenylphosphoranylidene)butanoate) (2b) [3, 4]. For example, condensation of 2a with 3-methyl-2-butenal (3a) gave a 22% yield of (3Z,5E)-4-hydroxy-2-oxo-3,5,7-triene 4a and with 2butenal (3b), no product could be isolated (Table 1). Last mentioned result was not unforeseen, since the analogous phosphonium ylide 2b gave, under the socalled mono anion condensation conditions, also low yields of condensation products [3, 4]. Condensation of the phosphonium ylide **2a** with aliphatic aldehydes **3c** and **3d** gave the wanted (3Z,5E)-4-hydroxy-2-oxo-3,5-dienes **4c** and **4d** in only low yields of 11% and 15% (Table 1). Our knowledge of the formation and use of the dianion of the phosphonium ylide 2b was applied to 2a [3, 4]. To increase the yield of Wittig condensation product, the dianion of the phosphonium ylide 2a was generated. The reaction of the phosphonium ylide 2a with lithium diisopropylamide in THF was exothermic and resulted in an intense orange solution of the dianion of **2a**. Subsequent condensations of the dianion of **2a** with  $\alpha,\beta$ -unsaturated aldehydes **3a** and **3b**, now gave a much higher yield of **4a** and **4b** (64% and 29%). In these cases 5:1 and 4:1 mixtures of (3Z,5E)- and (3Z,5Z) and (3Z,5E,7E)- and (3Z,5Z,7E) isomers of **4a** and **4b** were obtained. With 2-butenal (**3b**), no *Michael Wittig* condensation product was detected. The dianion of **2a** condensed also with aliphatic aldehydes 3c and 3d in a much higher yield of 4c (55%) and 4d (70%), but in mixtures of 3:2 and 2:1 (3Z,5E)- and (3Z,5Z) isomers (Table 1). The dianion of 2a condensed with the aldehyde 3e to give a 1:1 (E)- and (Z)-isomeric mixture

Carbonyl compound 3	Product 4	Yield of product 4			
		Wittig	( <i>3Z</i> , <i>5E</i> ):( <i>3Z</i> , <i>5Z</i> )	Dianion Wittig	( <i>3Z</i> , <i>5E</i> ):( <i>3Z</i> , <i>5Z</i> )
(3a)	OH O (4a)	22%	1:0	64%	5:1
(3b)	OH O (4b)	$\sim 0\%$	_	29%	4:1
CHO (3c)	OH O (4c)	11%	1:0	55%	3:2
СНО	ОН О	15%	1:0	70%	2:1
	(40)	-	-	49%	1:1
( <b>3e</b> )	( <b>4e</b> )				

Table 1. Yield of Wittig- and dianion Wittig condensations of carbonyl compounds 3 and the phosphonium ylide 2a

of 4e. We have not yet explored *Michael Wittig* condensations of 2a in great detail, however, preliminary results seem to suggest that the phosphonium ylide 2a is less able to condense in a *Michael Wittig* fashion than the corresponding phosphonium ylide 2b [3, 5, 6]. In an analogous manner as for the condensation of the phosphonium ylide 2b and 2*H*-pyran-5-carboxylates [7], the *Michael Wittig* condensation of the 2*H*-pyran ester 5 and 2a gave the cyclohexenonecarboxylate 6 (70% enolised in CDCl<sub>3</sub>) in a modest yield of 24%.

## **Experimental**

All reactions were carried out under N<sub>2</sub>. <sup>1</sup>H NMR (with SiMe<sub>4</sub> as an internal standard) and <sup>13</sup>C NMR spectra ( $\delta$ , ppm) were recorded in CDCl<sub>3</sub> on a Varian Gemini 200 spectrometer at 200 MHz and 50.3 MHz. Infrared spectra were obtained on a Hitachi 270-30 FTIR spectrophotometer (film, NaCl plates). Ultraviolet absorbance was measured as solutions in 96% EtOH on a Shimadzu UV-150 spectrophotometer. Column chromatography was performed using Merck Si-60 (40–63 mm) silica gel. Petroleum spirits is the fraction distilled between 40–60°C.

*1-(Triphenylphosphoranylidene)pentane-2,4-dione* (2a, $C_{23}H_{21}O_2P$ ): (2,4-Dioxopentyl)triphenylphosphonium bromide (1) [1, 2] (0.58 g, 1.314 mmol) in 30 cm<sup>3</sup> of CH<sub>2</sub>Cl<sub>2</sub> was treated with an aqueous solution of 0.16 g of NaHCO<sub>3</sub> (1.90 mmol) in 30 cm<sup>3</sup> of H<sub>2</sub>O and stirred vigorously for 30 min at room temperature. The CH<sub>2</sub>Cl<sub>2</sub> layer was separated and dried (Na<sub>2</sub>SO<sub>4</sub>). Evaporation of the solvent gave 0.47 g of crude **2a** (99.4%). This residue was recrystallised from benzene [8] to give pure **2a**.

<sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 2.263$  (s, 3H), 3.425 (s, 2H), 3.5–4.0 (broad, 1H), 7.44–7.65 (m, 15H) (Note: **2a** is 10% enolised) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 29.92$ , 53.65 (d,  $J_{CP} = 106.9$  Hz), 58.20 (d,  $J_{CP} = 15.2$  Hz), 126.17 (d,  $J_{CP} = 90.4$  Hz), 128.78 (d,  $J_{CP} = 12.3$  Hz), 132.30 (d,  $J_{CP} = 2.6$  Hz), 132.89 (d,  $J_{CP} = 10.3$  Hz), 184.09 (d,  $J_{CP} = 3.2$  Hz), 206.29 ppm.

#### Wittig Condensations of 2a with Carbonyl Compounds

(3Z,5E)-4-Hydroxy-8-methylnona-3,5,7-triene-2-one (4a, C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>): 0.19 g 2,4-Dioxo-1-(triphenylphosphoranylidene)pentane (2a) (0.527 mmol) in 1 cm<sup>3</sup> of benzene [8] was treated with 60 mg of 3methyl-2-butenal (3a) (0.74 mmol) and heated for 5 h at 75°C. The reaction mixture was diluted with 10 cm<sup>3</sup> of light petroleum and filtered over silica gel. The filtrate was concentrated and chromatographed over silica gel and eluted with ethyl acetate:light petroleum (1:9) to give 19 mg of 4a (21.7%).

UV (EtOH):  $\lambda_{max} = 240.5$  (263.8 enol) nm; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 1.89$  (sm, 3H), 1.9 (s, 3H), 2.1 (s, 3H), 5.52 (s, 1H), 5.8 (d, J = 15.1 Hz, 1H), 6.0 (dm, J = 11.7 Hz, 1H), 7.49 (dd, J = 15.1, 11.7 Hz, 1H), ~12 (s, OH) ppm; IR:  $\bar{\nu} = 2980$  (s), 2933 (s), 2880 (m), 1712 (s), 1600 (m), 1369 (s), 913 (s), 738 (s) cm<sup>-1</sup>.

The procedure for other aldehyde-*Wittig* condensations is exactly the same as for the example described above.

(3Z,5E)-4-Hydroxyundeca-3, 5-diene-2-one (**4c**, C<sub>11</sub>H<sub>18</sub>O<sub>2</sub>): UV (EtOH):  $\lambda_{max} = 242$ , (~270 enol) nm; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 0.85$  (sm, 3H), 1.3–1.5 (m, 6H), 2.08 (s, 3H), 2.1–2.25 (m, 2H), 5.463 (s, 1H), 5.799 (d, J = 15.4 Hz, 1H), 6.823 (dt, J = 15.4, 7.2 Hz, 1H), ~12 (s, OH) ppm; IR:  $\bar{\nu} = 2936$  (s), 2880 (s), 1709 (s), 1615 (m), 1466 (m), 1376 (s), 1240 (s), 1165 (s), 732 (m) cm<sup>-1</sup>.

(3Z,5E)-7-*Ethyl*-4-hydroxynona-3,5-diene-2-one (**4d**, C<sub>11</sub>H<sub>18</sub>O<sub>2</sub>): UV (EtOH):  $\lambda_{max} = 223.5$  (~305 enol) nm; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 0.858$  (t, J = 7.3 Hz, 6H), 1.2–1.6 (m, 2 pentets, 4H), 1.9–2.1 (m, 1H), 2.127 (s, 3H), 5.518 (s, 1H), 5.810 (d, J = 15.4 Hz, 1H), 6.617 (dt, J = 15.4, 9.2 Hz, 1H), ~12 (s, OH) ppm; IR:  $\bar{\nu} = 2962$  (s), 2930 (s), 2880 (m), 1710 (s), 1659 (m), 1430 (m), 1379 (m), 1120 (s), 722 (m) cm<sup>-1</sup>.

#### Dianion Condensations of 2a with Carbonyl Compounds

(3Z,5E)- and (3Z,5Z)-4-Hydroxy-8-methylnona-3,5,7-triene-2-one (4a): A yellow suspension of 1.00 g of 2a (2.78 mmol) in *THF* (20 cm<sup>3</sup>) was added within 1 min to a freshly prepared solution of lithium diisopropylamide (3 mmol) in *THF* (10 cm<sup>3</sup>) at room temperature. The resulting warm (~35°C), orange solution of the dianion was treated with is 0.35 g of 3-methyl-2-butenal (3a) (4.2 mmol) in *THF* (0.5 cm<sup>3</sup>) within 2 minutes. The condensation mixture was stirred for another 20 min at room temperature and monitored by thin layer chromatography. The reaction mixture was quenched with brine (50 cm<sup>3</sup>) and cautiously acidified with aqueous 2M H<sub>2</sub>SO<sub>4</sub> to pH 3. 50 cm<sup>3</sup> of petroleum spirits:ether (1:1) was added to the acidified aqueous mixture and the condensation products were extracted. The extraction process was repeated. The combined extract was dried and the solution filtered over silica gel to remove Ph<sub>3</sub>P=O. The evaporated residue was separated on silica gel and eluted with petroleum spirits:ethyl acetate (9:1) to give a 5:1 mixture of (*3Z*,*5E*)-**4a** and (*3Z*,*5Z*)-**4a** (0.305 g, 63.9%).

(3Z,5E)-(**4a**): <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 1.766$  (sm, 6H), 1.986 (s, 3H), 5.405 (s, 1H), 5.682 (d, J = 15.0 Hz, 1H), 5.874 (dm, J = 11.7 Hz, 1H), 7.365 (dd, J = 15.0, 11.7 Hz, 1H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 18.57$ , 26.23, 26.38 (3 CH<sub>3</sub>), 100.18 (=CH), 123.25, 124.31, 135.21 (3 =CH), 145.21 (=C-), 177.86 (=C(OH)), 196.62 (C=O) ppm.

(3Z,5Z)-(4a): <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 1.766$  (sm, 6H), 1.971 (s, 3H), 5.394 (s, 1H), 5.38 (d,  $J = \sim 12.0$  Hz, 1H), 6.601 (dd,  $J = \sim 12.0$ , 11.5 Hz, 1H), 7.105 (dm, J = 11.5, 1.1 Hz, 1H),  $\sim 12$  (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 17.70$ , 25.66, 26.62 (3 CH<sub>3</sub>), 101.73 (=CH), 119.32, 123.05, 137.39 (3 =CH), 145.73 (=C-), 180.68 (=C(OH)), 195.56 (C=O) ppm.

Condensations of  $\gamma$ -Acetylacetonephosphonium Ylide

Some 3,6,6-trimethylcyclohexa-1,3-diene-1-carbaldehyde as a self-condensation product of 3-methyl-2-butenal (3a) was also isolated [3].

The procedure for other aldehyde-dianion *Wittig* condensations is exactly the same as for the example described above.

(3Z,5E,7E)- and (3Z,5Z,7E)-4-Hydroxynona-3,5,7-triene-2-one (**4b**): For the 4:1 mixture of (3Z,5E,7E)-**4b** and (3Z,5Z,7E)-**4b**.

(3Z,5E,7E)-**4b**: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 1.781$  (d, J = 5.5, 3H), 2.031 (s, 3H), 5.455 (s, 1H), 5.735 (d, J = 15.3 Hz, 1H), 5.9–6.2 (m, 2H), 7.107 (dd, J = 15.3, 9.7 Hz, 1H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>): 18.48, 26.62, (2 CH<sub>3</sub>), 100.37 (=CH), 123.63, 130.34, 138.25, 140.24 (4 =CH), 177.47 (=C(OH)), 197.13 (C=O) ppm.

(3Z,5Z,7E)-**4b**: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz):  $\delta = 1.801$  (d, J = 5.5, 3H), 2.041 (s, 3H), 5.430 (s, 1H), ~5.4 (m, 1H), 5.9–6.2 (m, 2H), 6.350 (t,  $J = \sim 11$  Hz, 1H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 17.50$ , 26.31 (2 CH<sub>3</sub>), 101.76 (=CH), 119.83, 129.45, 139.55, 142.03 (4 =CH), 179.93 (=C(OH)), 192.67 (C=O) ppm.

(3Z,5E)- and (3Z,5Z)-4-Hydroxyundeca-3,5-diene-2-one (4c): For the 3:2 mixture (3Z,5E)-4c and (3Z,5Z)-4c.

(3Z,5E)-4c: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 0.84$  (sm, 3H), 1.2–1.5 (m, 6H), 2.02 (s, 3H), 2.2 (m, 2H), 5.44 (s, 1H), 5.77 (d, J = 15.5 Hz, 1H), 6.78 (dt, J = 15.5, 7.6 Hz, 1H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 13.40$  (CH<sub>3</sub>), 22.06 (CH<sub>2</sub>), 25.71 (CH<sub>3</sub>), 27.65, 31.00, 32.23 (3 CH<sub>2</sub>), 99.17 (=CH), 125.32, 144.11 (2 =CH), 177.63 (=C(OH)), 196.46 (C=O) ppm.

(3Z,5Z)-4c: <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta = 0.84$  (sm, 3H), 1.2–1.5 (m, 6H), 2.2 (m, 2H), 2.02 (s, 3H), 5.43 (s, 1H), 5.65 (d, J = 12.2 Hz, 1H), 5.99 (dt, J = 12.2, 7.8 Hz, 1H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 13.40$  (CH<sub>3</sub>), 22.06 (CH<sub>2</sub>), 25.99 (CH<sub>3</sub>), 28.70, 29.31, 31.13 (3 CH<sub>2</sub>), 101.21, 123.60, 146.96 (3 =CH), 180.46 (=C(OH)), 195.71 (C=O) ppm.

(*3Z*,*5E*)- and (*3Z*,*5Z*)-7-Ethyl-4-hydroxynona-3,5-diene-2-one (**4d**): For the 2:1 mixture of (*3Z*,*5E*)-**4d** and (*3Z*,*5Z*)-**4d**.

(3Z,5E)-4d: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 0.779$  (t, J = 7.3 Hz, 6H), 1.2–1.6 (m, 4H), 1.9 (m, 1H), 2.026 (s, 3H), 5.437 (s, 1H), 5.731 (d, J = 15.5 Hz, 1H), 6.533 (dd, J = 15.5, 9.3 Hz, 1H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 11.25$  (2 CH<sub>3</sub>), 26.13 (CH<sub>3</sub>), 26.62 (2 CH<sub>2</sub>), 46.05 (CH), 99.27, 125.45, 148.20 (3 =CH), 177.64 (=C(OH)), 196.44 (C=O) ppm.

(3Z,5Z)-4d: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 0.771$  (t, J = 7.3 Hz, 6H), 1.2–1.6 (m, 4H), 1.9 (m, 1H), 2.012 (s, 3H), 5.408 (s, 1H), 5.60–5.74 (m, 2H), ~12 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 11.25$  (2 CH<sub>3</sub>), 26.00 (CH<sub>3</sub>), 27.58 (2 CH<sub>2</sub>), 41.40 (CH), 101.36, 123.89, 151.58 (3 =CH), 180.25 (=C(OH)), 196.10 (C=O) ppm.

(*3Z*,*5E*)- and (*3Z*,*5Z*)-4-Hydroxy-7-phenylhepta-3,5-diene-2-one (**4e**): For the 1:1 mixture (*3Z*,*5E*)-**4e** and (*3Z*,*5Z*)-**4e**.

(3Z,5E)-4e: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 2.097$  (s, 3H, CH<sub>3</sub>), 3.525 (d, J = 6.5 Hz, 2H, CH<sub>2</sub>), 5.545 (s, 1H, =CH), 5.838 (dt, J = 15.4, 1.1 Hz, 1H, =CH), 7.012 (dt, J = 15.4, 6.5 Hz, 1H, =CH), 7.4 – 7.1 (m, 5H, Ar), 12.0 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta = 26.08$  (CH<sub>3</sub>), 38.49 (CH<sub>2</sub>), 101.81, 125.96, 144.48 (3 =CH), 180.08, (=C(OH)), 196.38 (C=O), and 126.28, 128.28 (2C), 128.35 (2C), 139.50 (Ar) ppm.

(3Z,5Z)-4e: <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  = 2.118 (s, 3H, CH<sub>3</sub>), 4.071 (d, J = 7.7 Hz, 2H, CH<sub>2</sub>), 5.489 (s, 1H, =CH), 5.806 (dt, J = 11.5, 1.6 Hz, 1H, =CH); 6.203 (dt, J = 11.5, 7.7 Hz, 1H, =CH), 7.4–7.1 (m, 5H, Ar), 12.0 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  = 26.43 (CH<sub>3</sub>), 35.59 (CH<sub>2</sub>), 99.82, 123.90, 142.18 (3 =CH), 177.02, (=C(OH)), 197.28 (C=O), and 126.28, 128.28 (2C), 128.47 (2C), 137.77 (Ar) ppm.

### Michael Wittig Condensation of 2a with 2H-Pyranester 5

*Ethyl 5-acetyl-2-methyl-6-(2-methyl-1-propenyl)-4-oxo-2-cyclohexene carboxylate* (6): 0.19 g of **2a** (0.527 mmol) in 1 cm<sup>3</sup> of benzene [8] was treated with 50 mg of ethyl 2,2,6-trimethyl-2*H*-pyran-5-carboxylate (5) [7] (0.223 mmol) and heated for 5 h at 75°C. The reaction mixture was diluted with 10 cm<sup>3</sup> of light petroleum and filtered over silica gel. The filtrate was concentrated and

chromatographed over silica gel and eluted with ethyl acetate:light petroleum (1:9) to give 16 mg of **6** (23.5%) ( $\sim$ 70% enol tautomer).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, enol tautomer):  $\delta = 1.200$  (t, J = 7.1 Hz, 3H, CH<sub>3</sub>), 1.640 (sm, J = 1.3 Hz, 3H, CH<sub>3</sub>), 1.917 (sm, J = 1.1 Hz, 3H, CH<sub>3</sub>), 1.994 (d, J = 1.5 Hz, 3H, CH<sub>3</sub>), 2.014 (s, CH<sub>3</sub>), 2.896 (d, J = 1.5 Hz, 1H, ring-CH), 3.909 (dd, J = 9.9, 1.5 Hz, 1H, ring-CH), 4.15 (q, J = 7.1 Hz, 2H, CH<sub>2</sub>), 5.072 (dm, J = 9.9, 1.6 Hz, 1H, =CH), 5.977 (q, J = 1.5 Hz, 1H, =CH), 12.0 (s, OH) ppm; <sup>13</sup>C NMR (CDCl<sub>3</sub>, enol tautomer):  $\delta = 13.93$ , 17.64, 21.84, 25.46, 27.55 (5 CH<sub>3</sub>), 35.62, 51.91 (2 ring CH), 61.12 (CH<sub>2</sub>), ~105.4 (=C), 124.37, 125.54 (2 =CH), 139.54, 149.96 (2 =C), 170.49 (C=O), 180.15 (=C(OH)), 188.77 (C=O) ppm; IR:  $= \bar{\nu} = \sim 3400$  (w), 2984 (s), 2865 (m), 1735 (s), 1715 (s), 1610 (m), 1445 (m), 1369 (m) cm<sup>-1</sup>.

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