

## Conversion of Alkyl Halides into the Corresponding Alcohols Under Mild Reaction Conditions

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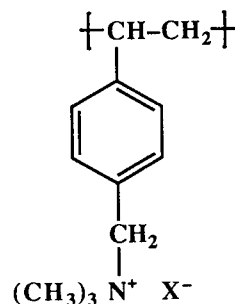
Reaction of primary, cyclopentyl, allyl and arylmethyl halides, but not an acyclic secondary halide or a tertiary halide, in acetone or tetrahydrofuran with the formate form of a commercial anion-exchange resin gave formate esters in good yields. The formates were hydrolysed efficiently to the corresponding alcohols by a brief treatment with hydrochloric acid. Reaction of primary alkyl bromides or iodides, secondary alkyl bromides, cinnamyl and arylmethyl halides in tetrahydropyran or 1,4-dioxane with the bicarbonate form of the same anion-exchange resin gave the corresponding alcohols directly in good yields. This latter reaction can be carried out successfully in the presence of ester or amide groups.

It is often necessary in organic synthesis to convert alcohols into alkyl halides, but only occasionally is it necessary to achieve the reverse conversion, i.e. the hydrolysis of alkyl halides. As a consequence relatively few reagents have been developed for this purpose.<sup>1</sup> The more reactive alkyl halides, i.e. tertiary, benzyl and allyl halides, can be hydrolysed easily by water in various solvents<sup>1</sup> or by aqueous bicarbonate.<sup>2,3</sup> However, the hydrolysis of primary alkyl halides is more difficult and has usually been achieved using alkali metal hydroxides,<sup>1</sup> though in a few simple cases it has been shown that water in *N*-methylpyrrolidone or hexamethylphosphoramide can achieve hydrolysis.<sup>4</sup> Primary bromides and iodides have also been 'hydrolysed' using bis(tributyltin) oxide in the presence of silver salts.<sup>5</sup>

In connection with our work on the synthesis of cyclic and linear oligoesters,<sup>6,7</sup> we wished to be able to convert primary alkyl bromide groups smoothly into hydroxyl groups under conditions that would not hydrolyse ester linkages in the same molecules. This has prompted us to look for new methods for the hydrolysis of alkyl halides and in this paper we report two new methods.

The ready availability of strong base anion-exchange resins designed for use in organic solvents has resulted in the study of a wide variety of anionic forms of such resins as polymer-supported (PS) reagents.<sup>8</sup> Such resins generally contain residues **1** and the reagents can be considered as PS benzyltrimethylammonium salts. In addition to being easy to prepare and convenient to use, since excess and spent reagent can be removed simply by filtering off the beads, they can be used successfully in various organic solvents. Examples of reactions using these PS quaternary ammonium salts are the synthesis of esters by reaction of PS carboxylates **1b** with alkyl halides in hexane or diethyl ether,<sup>9</sup> and the use of PS periodate **1c** to cleave 1,2-diols in dichloromethane, and to oxidise quinols in chloroform or thioethers in methanol.<sup>10</sup> The present study has involved reagents of this general type, namely PS carbonate **1d**, PS formate **1e** and PS bicarbonate **1f**. The last of these three proved to be very effective for hydrolysing alkyl halides.

The first method we investigated for hydrolysing primary alkyl bromides was to treat them with PS carbonate **1d**.



(1a): X<sup>-</sup> = Cl<sup>-</sup>

(1b): X<sup>-</sup> = RCO<sub>2</sub><sup>-</sup>

(1c): X<sup>-</sup> = IO<sub>4</sub><sup>-</sup>

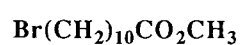
(1d): X<sup>-</sup> = [CO<sub>3</sub><sup>-</sup>]<sub>1/2</sub>

(1e): X<sup>-</sup> = HCO<sub>2</sub><sup>-</sup>

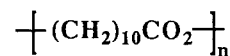
(1f): X<sup>-</sup> = HCO<sub>3</sub><sup>-</sup>

(1g): X<sup>-</sup> = I<sup>-</sup>

It has been shown before that PS carbonate **1d** hydrolyses allyl, benzyl and primary alkyl halides in tetrahydrofuran or benzene at reflux temperature.<sup>11</sup> In the present study we treated methyl 11-bromoundecanoate (**2**) with PS carbonate **1d**. The reaction was carried out for 24 h in 1,4-dioxane at 60°C. The desired methyl 11-hydroxyundecanoate was obtained but only in trace amounts (1% yield). The main product (~70% yield) was a mixture of oligoesters **3**. The latter presumably arise by hydrolysis of the ester group in **2** to give a PS carboxylate, then reaction of this with the alkyl bromide residues to generate new ester linkages.<sup>12</sup> Consistent with this it was found that methyl undecanoate reacted with PS carbonate **1d** under similar conditions to give undecanoic acid (15% yield). Since the PS carbonate **1d** clearly hydrolyses esters at a significant rate and overall the reactions are complex, this reagent was not further investigated.



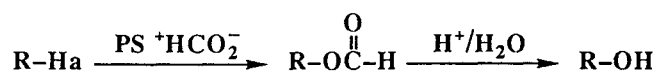
(2)



(3)

A second method investigated was the conversion of alkyl halides into formates followed by the hydrolysis of these relatively easily hydrolysed esters<sup>13</sup> (Scheme). Although numerous esters have been prepared previously by reaction of alkyl halides with PS carboxylates,<sup>7,9,14</sup> these esters do not include formates. PS formate **1e** was prepared in two steps. First, the commercially available chloride form **1a** of Amberlyst A26 was converted into the bicarbonate form **1f** by treatment of the former with a large excess of aqueous sodium bicarbonate using a column technique. The bicarbonate form **1f** was then neutralised with formic acid to give PS formate **1e**. Suspecting that the latter would have only a modest thermal stability its decomposition was investigated by thermogravimetric analysis (TGA). This showed that PS formate **1e** decomposes rapidly at temperatures above 100°C. Hence, reactions using this reagent need to be carried out at temperatures significantly lower than this. Of several reaction solvents investigated (THF, acetone, chloroform, toluene), THF and acetone proved to be the most effective.<sup>15</sup>

A range of alkyl halides were, therefore, treated with 3 to 4 molar equivalents of PS formate **1e** in either THF or acetone at reflux temperature. The reactions were monitored by GC and in selected cases the products were isolated. Due to the modest reaction temperatures and the modest nucleophilicity of the formate anion, in most cases reaction times of 72 h were necessary to obtain satisfactory yields. The results are summarised in Table 1, entries 1 to 11. It is evident that formates were obtained in satisfactory yields from the primary, cyclopentyl, allyl and arylmethyl halides but not from the acyclic secondary halide or the tertiary halide. The hydrolysis of the formate esters is so easy that in some cases (see entries 1,8 and 11) they hydrolysed partially under the reaction and "workup" conditions. In selected cases the formates were hydrolysed efficiently by treating the crude reaction products with hydrochloric acid at 20°C for 15 minutes.



#### Scheme

One attractive feature of PS reagents is that it is possible to use two PS reagents in the same reaction system without them reacting directly together.<sup>16,17</sup> In the present system it was shown (Table 1, entries 12, 13 and 14) that alkyl halides can be converted into alcohols satisfactorily in a one-pot procedure by using together PS formate **1e** and Amberlyst 15, a PS sulphonic acid suitable for cat-

alysing formate hydrolysis, in acetone containing 2% of water. As expected, with 1,2-dibromooctane reaction occurred mainly at the primary rather than the secondary position. The mixture of bromohydrins obtained could be useful as both isomers would be expected to react with base to generate the terminal epoxide.

The final method investigated, and the one which proved to be the most satisfactory, for converting alkyl halides into the corresponding alcohols was to treat the halide with PS bicarbonate **1f**. This reagent, prepared as described above, was shown by TGA to begin to decompose at ca. 120°C and so reaction temperatures up to ca. 100°C are satisfactory. Tetrahydropyran (bp 88°C) and 1,4-dioxane (bp 101°C) proved to be suitable reaction solvents and various alkyl halides in one or other of these solvents were treated with 2 to 3 molar equivalents of PS bicarbonate **1f** at reflux temperature for 3 to 24 h. The results are summarised in Table 2. These show that the primary alkyl bromides and an iodide, secondary alkyl bromides, cinnamyl and arylmethyl halides all react to give good isolated yields of the corresponding alcohols. It is noteworthy that the secondary alkyl halides reacted reasonably well. The primary alkyl chloride and tertiary alkyl halide reacted but in only poor yield, the former even in the presence of PS iodide **1g** as a catalyst.<sup>18</sup> The particularly attractive feature of this hydrolysis procedure is that the conversion of the halide group into a hydroxyl group took place without any significant hydrolysis of ester groups (see Table 2, entries 11,13 and 14) or an amide group (Table 2, entry 12) and, in the

**Table 1.** Synthesis and Hydrolysis of Various Formates

Entry	Substrate	Reaction Conditions <sup>a</sup>			Yield (%) <sup>b</sup>		Yield of Alcohol (%)	
		Solvent	Temp. (°C)	Time (h)	Formate	Alcohol	From Formate <sup>c</sup>	One-Pot Procedure <sup>d</sup>
1	butyl bromide	acetone	56	72	46 <sup>e</sup>	4	100	–
2	octyl bromide	THF	65	72	76 <sup>f</sup>	–	–	–
3	dodecyl bromide	THF	65	72	59	–	–	–
4	dodecyl bromide	acetone	56	72	100 <sup>f</sup>	–	100	–
5	2-bromooctane	THF	65	72	3 <sup>f</sup>	–	–	–
6	cyclopentyl bromide	THF	65	72	72 <sup>f</sup>	–	–	–
7	<i>t</i> -butyl chloride	THF	65	72	17 <sup>f</sup>	–	–	–
8	cinnamyl bromide	acetone	56	72	78	10	100	–
9	cinnamyl bromide	THF	65	72	92 <sup>f</sup>	–	–	–
10	benzyl chloride	acetone	56	72	100 <sup>f</sup>	0	–	–
11	2-(bromomethyl)naphthalene	acetone	56	72	70	20	100 <sup>g</sup>	–
12	octyl bromide	acetone	56	72	–	–	–	76
13	1,2-dibromooctane <sup>h</sup>	acetone	56	72	–	–	–	56 <sup>i</sup>
14	benzyl bromide	acetone	56	24	–	–	–	100

<sup>a</sup> The alkyl halide in the given solvent was treated with a 3–4 fold excess of PS formate **1e**.

<sup>b</sup> Unless indicated otherwise, yields quoted are isolated yields. See Experimental Section for full details.

<sup>c</sup> Yield of alcohol from hydrolysis of formate. Unless indicated otherwise, hydrolysis was carried out by stirring the filtered reaction mixture with 3M aq HCl at 20°C for 15 min. The yield is the percentage of the formate present that was hydrolysed.

<sup>d</sup> Yield of alcohol from one-pot synthesis. See experimental for full details.

<sup>e</sup> In a similar experiment carried out using THF as the solvent and monitored by GC, the yield of the formate was 84%.

<sup>f</sup> Estimated by GC and/or <sup>1</sup>H NMR spectral analysis.

<sup>g</sup> As *c*, except that 1M methanolic HCl was used.

<sup>h</sup> Starting material prepared by treating oct-1-ene in CCl<sub>4</sub> with a molar equivalent of Br<sub>2</sub> in CCl<sub>4</sub> for 10 min at 20°C, followed by evaporation of the solvent.

<sup>i</sup> By <sup>1</sup>H NMR spectroscopy the product was a mixture of 1-hydroxy-2-bromooctane (86%), 1-bromo-2-hydroxyoctane (4%) and octane-1,2-diol (10%).

**Table 2.** Hydrolysis of Alkyl Halides Using PS Bicarbonate **1f**

Entry	Substrate	Reaction Conditions <sup>a</sup>			Yield of Alcohol (%) <sup>b</sup>
		Solvent	Temp. (°C)	Time (h)	
1	undecyl chloride	D	101	24	26
2	undecyl bromide	D	101	24	68
3	undecyl iodide	D	101	24	68
4	2-bromoethylbenzene	THP	88	6	66
5	2-bromooctane	D	101	24	47
6	cyclopentyl bromide	D	101	24	54
7	<i>t</i> -butyl bromide	acetone	56	24	22 <sup>c</sup>
8	cinnamyl bromide	THP	88	3	64
9	<i>p</i> -bromobenzyl bromide	THP	88	2	71
10	2-bromomethylnaphthalene	THP	88	2	81
11	methyl 11-bromoundecanoate	D	101	24	77 <sup>d</sup>
12	<i>N</i> -butyl-11-bromoundecanamide	D	101	24	80 <sup>d</sup>
13	methyl 4-chloromethylbenzoate	THP	88	3	80 <sup>d</sup>
14	methyl undecanoate	D	101	24	92 <sup>e</sup>

<sup>a</sup> See Experimental Section for details. D = 1,4-dioxane THP = tetrahydropyran.

<sup>b</sup> Unless indicated otherwise yield quoted is of product isolated by distillation or column chromatography.

<sup>c</sup> Determined by <sup>1</sup>H NMR spectral analysis.

<sup>d</sup> Products were as starting material but with the halogen replaced by hydroxyl.

<sup>e</sup> Yield of recovered starting material.

case of (2-bromoethyl)benzene, without significant elimination of hydrogen bromide.

In summary, three methods have been investigated for converting alkyl halides into alcohols under mild conditions. PS carbonate **1d** brings about the hydrolysis of various alkyl halides<sup>12</sup> but it also brings about ester hydrolysis. The two-step procedure of reacting alkyl halides with PS formate **1e** followed by hydrolysis of the formate ester (see Scheme) can give good yields of alcohol but the first reaction tends to be sluggish. By using the PS formate **1e** and a strong acid cation exchange resin catalyst together both the reaction steps can be carried out successfully in 'one-pot'. The most satisfactory reagent is PS bicarbonate **1f**. This converts a wide variety of alkyl halides into alcohols without bringing about any significant elimination or ester or amide hydrolysis.

Unless indicated otherwise the alkyl halides, resins and solvents were purchased from either the Aldrich Chemical Company or Lancaster Synthesis and were used without further purification. Methyl 11-bromoundecanoate,<sup>19</sup> methyl undecanoate,<sup>20</sup> *N*-butyl-11-bromoundecanamide<sup>21</sup> and methyl 4-chloromethylbenzoate<sup>22</sup> were synthesised by known literature methods. Column chromatography was performed on silica gel 60, 230–400 mesh (Merck). Petroleum ether refers to the fraction bp 60–80°C. GC analysis was carried out using a Perkin-Elmer model 8300 equipped with a 10% PEG A on Chrom W-AW (80–100 mesh) column, length 2 m, internal diameter 2 mm. The oven temperature was set at 180°C, the injector port temperature at 230°C, the detector temperature at 230°C and the flow rate at 20 mL/min. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker AC 300 spectrometer operating at 300 and 75 MHz, respectively, or a Varian FX 100 for <sup>1</sup>H NMR operating at 100 MHz. Chemical shifts ( $\delta$ ) are quoted in ppm relative to TMS. Mass spectra were obtained with a Fisons VG TRIO 2000 spectrometer. IR spectra were recorded for evaporated films on a ATI Mattson Genesis Series FTIR spectrometer. Melting points were determined using an Electrothermal capillary apparatus and are uncorrected. Unless indicated otherwise, drying was achieved using a vacuum oven operating at 60°C/0.1 Torr. Elemental analyses

were carried out in-house using a Carlo Erba instrument. Where reaction products were well-known compounds, such as butanol and cinnamyl alcohol, they were identified by IR and <sup>1</sup>H NMR spectroscopy in comparison with authentic samples.

Amberlyst 15 was washed successively with dil HCl, water, acetone and Et<sub>2</sub>O, then dried by heating at 105°C for 6 h.

#### PS Carbonate **1d**:

Amberlyst A-26 in the chloride form **1a** was packed into a column. It was then washed successively with water (2 volumes), sat Na<sub>2</sub>CO<sub>3</sub> solution (10 volumes over 24 h), water until the washings were neutral, acetone (2 volumes) and finally Et<sub>2</sub>O (2 volumes). The resin was then dried at 40°C in a vacuum oven for 48 h. By back titration using 2 N HCl, the final resin contained 3.40 mmol of carbonate per g.

#### Reaction of Methyl 11-Bromoundecanoate (**2**) with PS Carbonate **1d**:

A mixture of ester **2** (1.9 g), PS carbonate **1d** (9.18 g) and 1,4-dioxane (50 mL) was stirred and heated at 60°C for 24 h. The mixture was then cooled and the resin filtered and washed with CHCl<sub>3</sub>. Evaporation of the combined filtrate and washings gave an oil (1.01 g, ca. 70% yield) which was shown by <sup>1</sup>H NMR spectroscopy and gel permeation chromatography<sup>23</sup> to be a mixture of oligomers **3** with *n* = 1 to 6, mainly Br(CH<sub>2</sub>)<sub>10</sub>CO<sub>2</sub>(CH<sub>2</sub>)<sub>10</sub>CO<sub>2</sub>Me. The resin was washed with 2 N HCl (20 mL) and the acid was extracted with CHCl<sub>3</sub>. The extracts were dried and the solvent evaporated. This gave 11-hydroxyundecanoic acid (10 mg, 1%); mp 65–66°C, identified by IR spectroscopy in comparison with an authentic sample.<sup>24</sup>

#### PS Bicarbonate **1f**:

PS bicarbonate **1f** was prepared in an analogous way to the PS carbonate **1d** as described above, except that NaHCO<sub>3</sub> was used in place of Na<sub>2</sub>CO<sub>3</sub>. The final resin contained 3.45 mmol of bicarbonate per g.

#### PS Formate **1e**:

The bicarbonate form **1f** of Amberlyst A-26, prepared as above, was treated with an excess of formic acid in MeOH for 18 h. It was then washed successively with deionised water to neutrality, with acetone, and finally with Et<sub>2</sub>O, and dried at 40°C for 48 h. Given the loading of the bicarbonate form, it was calculated that the product contained 3.65 mmol of formate anion per g.

**Butyl Formate; Typical Procedure:**

A mixture of butyl bromide (530 mg, 386 mmol), the formate form of Amberlyst A-26 resin **1e** (3.87 g, 3.65 mmol formate per gram of resin) and acetone (30 mL) was stirred and heated at reflux temperature for 72 h. The resin was then removed by filtration and washed with acetone. The filtrate and washings were combined and the solvent evaporated to leave the crude product as a clear liquid (224 mg). This was subjected to column chromatography using petroleum ether as the eluent. Initially butyl formate (182 mg, 46%) was eluted; bp 107°C/760 Torr (Lit.<sup>25</sup> bp 105–106°C/760 Torr).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ = 8.05 (s, 1H, CH<sub>2</sub>O<sub>2</sub>CH), 4.05 (t, 2H, *J* = 7.5 Hz, CH<sub>2</sub>O<sub>2</sub>CH), 1.7–1.5 (quint, 2H, *J* = 7.5 Hz, OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 1.38 (sext, 2H, *J* = 7.5 Hz, CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 0.9 (t, 3H, *J* = 7.5 Hz, CH<sub>3</sub>).

IR: ν = 1694 cm<sup>-1</sup>.

Subsequently butanol (10 mg recovered, 4%) was eluted. The results of this and other experiments are summarised in Table 1, entries 1–11.

**Dodecan-1-ol; Typical Procedure:**

The above crude formate product (1.03 g) in acetone (40 mL) was stirred with aq 3M HCl (1 mL) at 20°C for 15 min. Most of the solvent was evaporated, water (5 mL) was added and the product was extracted with Et<sub>2</sub>O. Evaporation of the dried extracts gave dodecanol (900 mg, 100% yield). The results obtained with this and other formates are summarized in Table 1, entries 1, 4, 8 and 11.

**One-Pot Procedure for the Conversion of Alkyl Halides into Alcohols:***Hydrolysis of octyl bromide; Typical Procedure:*

Octyl bromide (741 mg, 3.84 mmol) was dissolved in a mixture of acetone (50 mL) and water (0.5 mL). The formate form of Amberlyst A-26 resin (3.0 g, 3.65 mmol formate per gram resin) **1e** and Amberlyst A-15 resin (2.0 g) were added and the mixture was stirred and heated under reflux for 24 h. The resin was filtered and washed with acetone. The combined filtrate and washings were evaporated to dryness. The residue (490 mg), analysed by GC in comparison with authentic samples, was a mixture of octan-1-ol (76%) and octyl bromide (20%). The results obtained with this and the other alkyl halides are summarized in Table 1, entries 12–14.

**PS Iodide 1g:**

The iodide form of Amberlyst A-26 was prepared by neutralising the bicarbonate form of the resin with an excess of HI (57% solution in water). The resin was washed thoroughly and successively with water, acetone and finally Et<sub>2</sub>O. It was dried at 40°C for 48 h. By titration using AgNO<sub>3</sub> solution, the final product was shown to contain 2.85 mmol of iodide per g.

**Reaction of the Bicarbonate Form of Amberlyst A-26 1f with Alkyl Halides; Typical Procedures:***Undecan-1-ol:*

Undecyl chloride (2.22 g, 11.64 mmol) in 1,4-dioxane (100 mL) was treated with the bicarbonate form of Amberlyst A-26 resin **1f** (8.16 g, 28.1 mmol HCO<sub>3</sub><sup>-</sup>) and the iodide form of Amberlyst A-26 resin **1g** (11.51 g, 2.85 mmol iodide per gram of resin, 32.8 mmol of I<sup>-</sup>) and the mixture was heated under reflux for 24 h. The resin was filtered and the beads washed with 1,4-dioxane. The filtrate and washings were combined and the solvent evaporated to leave the crude product. This was purified by column chromatography. Elution with petroleum ether/EtOAc (10:1) afforded unreacted starting material (1.07 g, 48%). Elution with petroleum ether/EtOAc (6:1) gave the desired product as a colourless liquid (510 mg, 2.97 mmol, 25%). (Lit.<sup>26</sup> bp 123–125°C/6 Torr).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ = 3.6 (t, 2H, *J* = 2 Hz, CH<sub>2</sub>OH), 1.75 (br s, OH), 1.55 (quint, 2H, *J* = 2 Hz, CH<sub>2</sub>CH<sub>2</sub>OH), 1.3 (s, 16H, CH<sub>2</sub>), 0.9 (t, 3H, *J* = 2 Hz, CH<sub>3</sub>CH<sub>2</sub>).

MS (CI): *m/z* = 190 [(M + NH<sub>4</sub>)<sup>+</sup>].

IR: ν = 3600–3100 (v br, OH), 2954, 2854, 1465, 1377, 1056 cm<sup>-1</sup>.

*Methyl 11-Hydroxyundecanoate:*

The crude material obtained by reacting methyl 11-bromoundecanoate (1.04 g, 3.72 mmol) with the bicarbonate form of Amberlyst

A-26 **1f** (3.25 g) in 1,4-dioxane (50 mL) at reflux temperature for 24 h was purified by distillation. This gave the desired compound as a colourless liquid (616 mg, 77%); bp > 200°C/15 Torr. (Lit.<sup>27</sup> bp 168–169°C/8 Torr).

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ = 3.7–3.5 (t + s, 5H, *J* = 2.4 Hz, CH<sub>2</sub>OH + CH<sub>3</sub>CO<sub>2</sub>), 2.35 (t, 2H, *J* = 2.4 Hz, CH<sub>2</sub>CO<sub>2</sub>Me), 1.60 (quint, 4H, *J* = 2.4 Hz, CH<sub>2</sub>), 1.30 (s, 12H, CH<sub>3</sub>).

MS (CI): *m/z* = 234 [(M + NH<sub>4</sub>)<sup>+</sup>].

IR: ν = 3700–3100 (v br, OH, 2927) 1740 cm<sup>-1</sup> (ester C=O).

*N-Butyl-11-hydroxyundecanamide:*

The crude material obtained by treating *N*-butyl-11-bromoundecanamide (1.71 g, 5.34 mmol) with the bicarbonate form of Amberlyst A-26 **1f** (4.50 g) in tetrahydropyran (50 mL) at reflux temperature for 24 h was recrystallised from EtOH to give the desired compound as colourless cubic crystals (1.09 g, 80%); mp 68–69°C.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ = 5.55–5.45 (br s, 1H, NH), 3.65 (t, 2H, *J* = Hz, CH<sub>2</sub>OH), 3.25 (q, 2H, *J* = 2 Hz, NHCH<sub>2</sub>CH<sub>2</sub>), 2.15 (t, 2H, *J* = 2 Hz, CH<sub>2</sub>CO), 1.7–1.5 (m, 20H, CH<sub>2</sub>), 0.90 (t, 3H, *J* = 2 Hz, CH<sub>3</sub>).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ = 173.1, 63.05, 39.2, 36.9, 32.8, 31.8, 29.5, 29.4, 29.3, 29.2, 25.8, 25.7, 20.1 and 13.7.

MS (EI): *m/z* = 258 ((M + 1)<sup>+</sup>).

IR: ν = 3393, 3316, 1639 cm<sup>-1</sup>.

C <sub>15</sub> H <sub>31</sub> NO <sub>2</sub>	calc.	C 70.00	H 12.06	N 5.40
	found	69.97	12.13	5.43

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