

## Synthesis of *N*-Vinylalkoxyalkyl Imines

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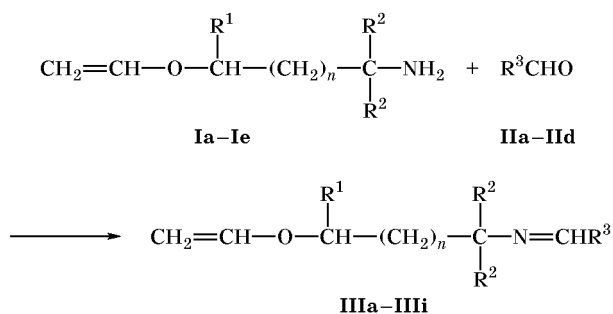
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**Abstract**—*N*-Alkylidene-2(3 or 4)-vinylalkoxyalkylamines were synthesized in 60–93% yield by reactions of 2(3 or 4)-vinylalkoxyalkylamines with aliphatic aldehydes. The stability of the resulting Schiff bases and their further transformations were studied.

Schiff bases derived from 2-vinylalkoxyethylamine and carbonyl compounds are promising nitrogen-containing monomers and starting materials in the synthesis of biologically active substances [1, 2]. However, such Schiff bases were prepared only from ketones and aromatic aldehydes. Presumably, this is explained by the relatively low stability of aliphatic Schiff bases [3]. For example, Shostakovskii *et al.* [4] reported that the condensation product of 2-vinylalkoxyethylamine (**Ia**) and acetaldehyde instantaneously undergoes complete tarring on attempted vacuum distillation. These data contradict our results: We have synthesized Schiff base **IIIa** by acid-catalyzed isomerization of vinyl ether **Ia** [5].

The present communication reports on the results of our study on the condensation of aminoalkyl vinyl ethers with aliphatic aldehydes and the stability of the resulting Schiff bases. All vinyl ethers **Ia–Ie**, regardless of their structure, vigorously reacted with acetaldehyde in the cold to give expected Schiff bases **IIIa–IIIe** in 60–74% yield (Scheme 1). After drying over anhydrous magnesium sulfate, Schiff bases **IIIa–IIIe** can be stored for 4–5 days at room temperature under nitrogen without appreciable decomposition. Later on, rapidly progressing tarring occurs. Distillation of the tarry materials obtained after storage of Schiff bases **IIIa** and **IIId** for a month gave compounds **IVa** and **IVb**, respectively, in 7–9% yield.

Scheme 1.

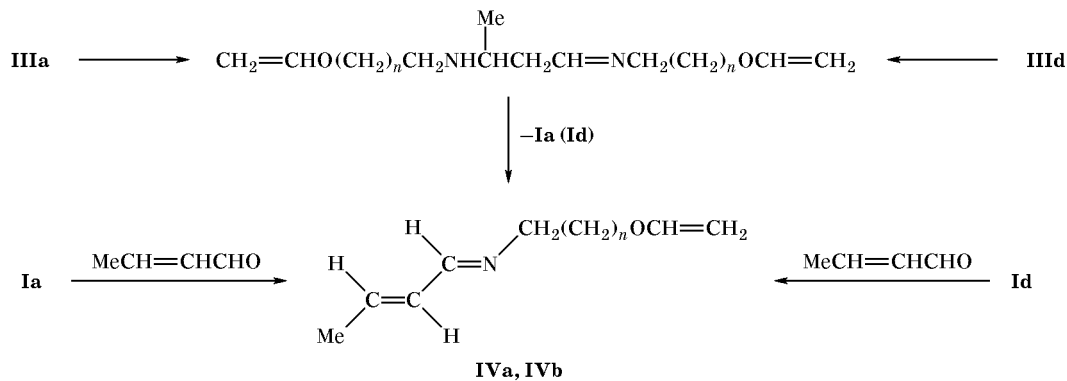


**I**, R<sup>1</sup> = R<sup>2</sup> = H, n = 0 (**a**); R<sup>1</sup> = Me, R<sup>2</sup> = H, n = 0 (**b**); R<sup>1</sup> = H, R<sup>2</sup> = Me, n = 0 (**c**); R<sup>1</sup> = R<sup>2</sup> = H, n = 1 (**d**); R<sup>1</sup> = R<sup>2</sup> = H, n = 2 (**e**); **II**, R<sup>3</sup> = H (**a**), Me (**b**), *i*-Pr (**c**), *i*-Bu (**d**); **III**, R<sup>1</sup> = R<sup>2</sup> = H, R<sup>3</sup> = Me, n = 0 (**a**); R<sup>1</sup> = R<sup>3</sup> = Me, R<sup>2</sup> = H, n = 0 (**b**); R<sup>2</sup> = R<sup>3</sup> = Me, R<sup>1</sup> = H, n = 0 (**c**); R<sup>1</sup> = R<sup>2</sup> = H, R<sup>3</sup> = Me, n = 1 (**d**); R<sup>1</sup> = R<sup>2</sup> = H, R<sup>3</sup> = Me, n = 2 (**e**); R<sup>1</sup> = R<sup>2</sup> = H, R<sup>3</sup> = *i*-Pr, n = 0 (**f**); R<sup>1</sup> = R<sup>2</sup> = H, R<sup>3</sup> = *i*-Pr, n = 1 (**g**); R<sup>1</sup> = R<sup>2</sup> = H, R<sup>3</sup> = *i*-Bu, n = 0 (**h**); R<sup>1</sup> = R<sup>3</sup> = H, R<sup>2</sup> = Me, n = 0 (**i**).

The formation of Schiff bases **IVa** and **IVb** indicates that, as with aliphatic Schiff bases, the tarring process involves aldol-like condensation of compounds **IIIa** and **IIId**. The structure of products **IVa** and **IVb** was proved by the IR and <sup>1</sup>H NMR spectra and by independent syntheses from amines **Ia** and **Id** and crotonaldehyde (Scheme 2). The stability of Schiff bases **IVa** and **IVb** is almost the same as that of **IIIa** and **IIId**. Compounds **IIIf–IIIh** obtained from isobutyraldehyde and isovaleraldehyde are much more stable. Their decomposition becomes appreciable only after storage for 1 to 1.5 month.

Like primary aliphatic amines, 2-aminoethyl vinyl ether (**Ia**) is known to undergo condensation with formaldehyde, affording hexahydro-1,3,5-triazine derivative [2]. We effected condensation of formaldehyde with 3-vinylalkoxypropylamine (**Id**) and 1,1-dimethyl-2-vinylalkoxyethylamine (**Ic**) (Scheme 3). In the reaction with **Id** we obtained stable hexahydro-1,3,5-triazine **Va**. Under the same conditions, amine **Ic** gave rise to Schiff base **IIIi**, and the latter underwent fairly

Scheme 2.



IV,  $n = 1$  (a),  $2$  (b).

slow trimerization to hexahydrotriazine **Vb** at room temperature. According to the  $^1\text{H}$  NMR data, namely to the intensities of the  $\text{H}_2\text{C}=\text{N}$  and  $\text{NCH}_2\text{N}$  proton signals, the molar ratio of Schiff base **IIIi** and trimer **Vb** was 1:0.22 in 12 h. After storage for 3 months at room temperature, the equilibrium mixture contained 8 mol % of Schiff base **IIIi**. Distillation of trimer **Vb** even under reduced pressure (2 mm) leads to its complete transformation into Schiff base **IIIi**. Thus the trimerization of **IIIi** to hexahydrotriazine **Vb** is a readily reversible process. The observed difference in the stabilities of perhydrotriazines **Va** and **Vb** is likely to arise from steric factor, i.e., from the presence in molecule **Va** of two geminal methyl groups.

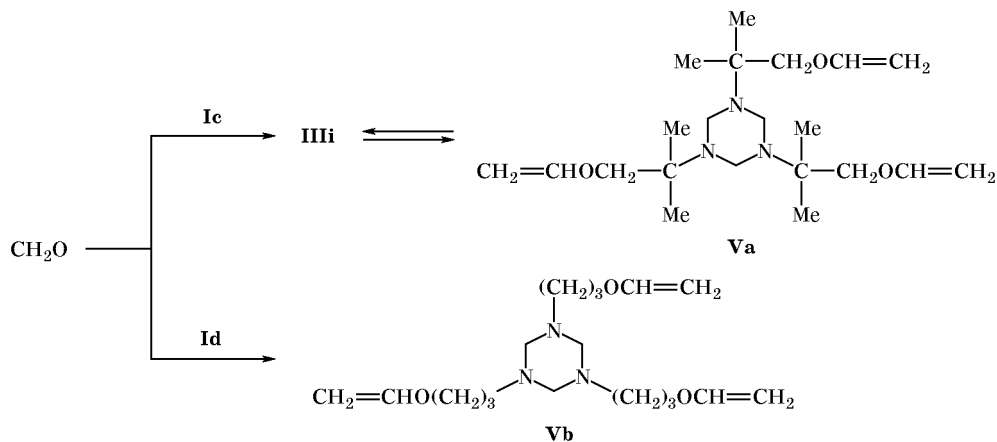
In the  $^1\text{H}$  NMR spectra of compounds **IIIb–IIIe**, the  $\text{N}=\text{CH}$  signal appears as a quartet at  $\delta$  7.7–7.73 ppm; the corresponding signal of Schiff bases **IIIg** and **IIIh** is a doublet at  $\delta$  7.51–7.56 ppm, and compound **IIIh** displays a triplet at  $\delta$  7.67 ppm from the same proton. The coupling constant  $^3J$  is 4.9 to 5.1 Hz. The presence of only one signal from the

$\text{CH}=\text{N}$  proton in the spectra of **IIIb–IIIh** indicates that these compounds have *E* configuration of the double  $\text{C}=\text{N}$  bond, which is typical of aliphatic Schiff bases [6].

Unlike compounds **IIIb–IIIe**, the  $\text{N}=\text{CH}$  signal in the spectrum of **IIIa** has a greater multiplicity. However, this is the result of long-range coupling which is often observed for Schiff bases [6] rather than of the presence of *Z* isomer. Therefore, the  $\text{N}=\text{CH}$  signal of **IIIa** appears as a triplet of quartets with  $^3J(\text{CHCH}_3) = 4.8 \pm 0.2$  and  $^4J(\text{CH}=\text{NCH}_2) = 1.2 \pm 0.2$  Hz. Protons of the methyl group give rise to a doublet of triplets with  $^3J = 4.8 \pm 0.2$  and  $^5J = 1.1 \pm 0.2$  Hz. An analogous long-range coupling is observed in the spectra of Schiff bases **IVa** and **IVb**. Schiff bases **IIIb** and **IIIf–IIIh** display no such coupling, though some broadening of the respective signal occurs in the spectra of **IIIb**, **IIIg**, and **IIIh**.

Protons of the  $\text{N}=\text{CH}_2$  group in Schiff base **IIIi**, instead of the expected doublet of doublets, give rise to a signal which can be treated as a doublet

Scheme 3.



**Table 1.** Yields, boiling points, densities, refractive indices, and elemental analyses of compounds **IIIa–IIIi**, **IVa**, **IVb**, **Va**, and **Vb**

Comp. no.	Yield, %	bp, °C ( <i>p</i> , mm)	$d_4^{20}$	$n_D^{20}$	Found, %			Formula	Calculated, %		
					C	H	N		C	H	N
<b>IIIa</b>	60	65–67 (63) <sup>a</sup>	0.8780	1.4402	63.34	9.59	12.23	C <sub>6</sub> H <sub>11</sub> NO	63.68	9.80	12.38
<b>IIIb</b>	63	75–78 (60)	0.8627	1.4378	66.35	10.18	10.75	C <sub>7</sub> H <sub>13</sub> NO	66.11	10.30	11.01
<b>IIIc</b>	69	64–67 (48)	0.8639	1.4418	67.84	10.84	9.81	C <sub>8</sub> H <sub>15</sub> NO	68.04	10.71	9.92
<b>III d</b>	74	74–77 (55)	0.8770	1.4424	66.43	10.21	11.42	C <sub>7</sub> H <sub>13</sub> NO	66.11	10.30	11.01
<b>III e</b>	70	82–84 (30)	0.8738	1.4480	67.79	10.70	9.75	C <sub>8</sub> H <sub>15</sub> NO	68.04	10.71	9.92
<b>III f</b>	87	77–80 (50)	0.8566	1.4402	68.12	10.59	9.80	C <sub>8</sub> H <sub>15</sub> NO	68.04	10.71	9.92
<b>III g</b>	84	70–73 (20)	0.8552	1.4415	69.55	10.92	9.18	C <sub>9</sub> H <sub>17</sub> NO	69.63	11.04	9.02
<b>III h</b>	84	91–93 (50)	0.8595	1.4440	69.51	11.08	9.14	C <sub>9</sub> H <sub>17</sub> NO	69.63	11.04	9.02
<b>III i</b>	93	57–59 (50)	0.8936	1.4485	66.00	10.14	10.9	C <sub>7</sub> H <sub>13</sub> NO	66.11	10.30	11.01
<b>IVa</b>	61, <sup>b</sup> 7 <sup>c</sup>	95–97 (25)	0.9073	1.4830	69.24	9.31	10.14	C <sub>8</sub> H <sub>13</sub> NO	69.12	9.42	10.08
<b>IVb</b>	63, <sup>b</sup> 9 <sup>c</sup>	103–105 (26)	0.8908	1.4812	70.27	9.48	9.21	C <sub>9</sub> H <sub>15</sub> NO	70.55	9.87	9.14
<b>Va</b>	94	207–210 (10)	1.0050	1.4930	63.78	9.79	12.51	C <sub>18</sub> H <sub>33</sub> N <sub>3</sub> O <sub>3</sub>	63.68	9.80	12.38
<b>Vb<sup>d</sup></b>	93	28–30 (2)	1.0047	1.4835	66.00	10.13	10.96	C <sub>21</sub> H <sub>39</sub> N <sub>3</sub> O <sub>3</sub>	66.11	10.30	11.01

<sup>a</sup> Published data [5]: bp 74–75°C (100 mm),  $d_4^{20} = 0.8784$ ,  $n_D^{20} = 1.4405$ .

<sup>b</sup> From amino alcohol vinyl ether and crotonaldehyde.

<sup>c</sup> From condensation products of Schiff base **III**.

<sup>d</sup> Given are the yield and boiling point of **IIIi** and  $d_4^{20}$ ,  $n_D^{20}$ , and analytical data of trimer **Vb** containing 8 mol % of **IIIi**.

**Table 2.** IR and <sup>1</sup>H NMR spectra of compounds **IIIa–IIIi**, **IVa**, **IVb**, **Va**, and **Vb**

Comp. no.	IR spectrum, $\nu$ , cm <sup>-1</sup>	<sup>1</sup> H NMR spectrum, $\delta$ , ppm ( <i>J</i> , Hz)
<b>IIIa</b>	1600, 1620 (C=C); 1660 (C=N); 3100 (=C–H)	1.91 d.t (3H, CH <sub>3</sub> , <sup>3</sup> <i>J</i> = 4.8, <sup>5</sup> <i>J</i> = 1.1), 3.54 m (2H, NCH <sub>2</sub> ), 3.75 m (2H, OCH <sub>2</sub> ), 3.87 d.d (1H, <i>cis</i> -CH=C, <sup>2</sup> <i>J</i> = 2.2, <sup>3</sup> <i>J</i> <sub><i>cis</i></sub> = 6.9), 4.08 d.d (1H, <i>trans</i> -CH=C, <sup>2</sup> <i>J</i> = 2.2, <sup>3</sup> <i>J</i> <sub><i>trans</i></sub> = 14.0), 6.32 d.d (1H, OCH=C, <sup>3</sup> <i>J</i> <sub><i>cis</i></sub> = 6.9, <sup>3</sup> <i>J</i> <sub><i>trans</i></sub> = 14.0), 7.72 t.q (1H, N=CH, <sup>3</sup> <i>J</i> = 4.8, <sup>4</sup> <i>J</i> = 1.2)
<b>IIIb</b>	1600, 1615 (C=N); 1660 (C=N); 3105 (=C–H)	1.24 d (3H, OCHCH <sub>3</sub> ), 1.97 d (3H, N=CHCH <sub>3</sub> ), 3.48 m (2H, NCH <sub>2</sub> ), 3.92–4.27 m (3H, OCH, <i>cis</i> -CH=C, <i>trans</i> -CH=C), 6.30 d.d (1H, OCH=C, <sup>3</sup> <i>J</i> <sub><i>cis</i></sub> = 6.8, <sup>3</sup> <i>J</i> <sub><i>trans</i></sub> = 14.1), 7.73 q (1H, N=CH)
<b>IIIc</b>	1605, 1620 (C=C); 1655 (C=N); 3050, 3110 (=C–H)	1.17 s (6H, 2CH <sub>3</sub> ), 1.96 d (3H, CH <sub>3</sub> ), 3.55 s (2H, CH <sub>2</sub> O), 3.92 d.d (1H, <i>cis</i> -HC=C), 4.14 d.d (1H, <i>trans</i> -CH=C), 6.44 d.d (1H, OCH=C), 7.73 q (1H, N=CH)
<b>III d</b>	1605, 1620 (C=C); 1660 (C=N); 3030, 3105 (=C–H)	1.95 m (5H, CH <sub>3</sub> , CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ), 3.45 t (2H, NCH <sub>2</sub> ), 3.70 t (2H, OCH <sub>2</sub> ), 3.96 d.d (1H, <i>cis</i> -CH=C), 4.16 d.d (1H, <i>trans</i> -CH=C), 6.44 d.d (1H, OCH=C), 7.72 q (1H, N=CH)
<b>III e</b>	1605, 1620 (C=C); 1650 (C=N); 3040, 3110 (=C–H)	1.67 m (4H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> ), 1.94 d (3H, CH <sub>3</sub> ), 3.38 t (2H, NCH <sub>2</sub> ), 3.68 t (2H, OCH <sub>2</sub> ), 3.95 d.d (1H, <i>cis</i> -CH=C), 4.14 d.d (1H, <i>trans</i> -CH=C), 6.44 d.d (1H, OCH=C), 7.70 q (1H, N=CH)
<b>III f</b>	1605, 1620 (C=C); 1650 (C=N); 3050, 3110 (=C–H)	1.04 d (6H, 2CH <sub>3</sub> ), 2.38 m [1H, CH(CH <sub>3</sub> ) <sub>2</sub> ], 3.56 t (2H, NCH <sub>2</sub> ), 3.83 t (2H, OCH <sub>2</sub> ), 3.91 d.d (1H, <i>cis</i> -CH=C), 4.11 d.d (1H, <i>trans</i> -CH=C), 6.39 d.d (1H, OCH=C), 7.56 d (1H, N=CH, <sup>3</sup> <i>J</i> = 5.1)

Table 2. (Contd.)

Comp. no.	IR spectrum, $\nu$ , $\text{cm}^{-1}$	$^1\text{H}$ NMR spectrum, $\delta$ , ppm ( $J$ , Hz)
<b>IIIg</b>	1610, 1625 (C=C); 1655 (C=N); 3110 (=C-H)	1.04 d (6H, $2\text{CH}_3$ , $^3J = 6.8$ ), 1.93 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 2.47 m [1H, $\text{CH}(\text{CH}_3)_2$ ], 3.44 t (2H, $\text{NCH}_2$ , $^3J = 6.5$ ), 3.68 t (2H, $\text{OCH}_2$ , $^3J = 6.2$ ), 3.94 d.d (1H, <i>cis</i> -CH=C, $^2J = 2.0$ , $^3J_{\text{cis}} = 6.8$ ), 4.14 d.d (1H, <i>trans</i> -CH=C, $^2J = 2.0$ , $^3J_{\text{trans}} = 14.3$ ), 6.43 d.d (1H, $\text{OCH}=\text{C}$ , $^3J_{\text{cis}} = 6.8$ , $^3J_{\text{trans}} = 14.3$ ), 7.51 d (1H, $\text{N}=\text{CH}$ , $^3J = 5.0$ )
<b>IIIh</b>	1600, 1620 (C=C); 1640 (C=N); 3080 (=C-H)	0.93 d (6H, $2\text{CH}_3$ ), 1.42 m [1H, $\text{CH}(\text{CH}_3)_2$ ], 2.14 m (2H, $\text{CHCH}_2\text{CH}$ ), 3.60 m (2H, $\text{NCH}_2$ ), 3.85 t (2H, $\text{OCH}_2$ ), 3.94 d.d (1H, <i>cis</i> -CH=C), 4.14 d.d (1H, <i>trans</i> -CH=C), 6.41 d.d (1H, $\text{OCH}=\text{C}$ ), 7.67 t (1H, $\text{N}=\text{CH}$ )
<b>IIIi</b>	1600, 1620 (C=C); 1650 (C=N); 3105 (=C-H)	1.20 s (6H, $2\text{CH}_3$ ), 3.59 s (2H, $\text{OCH}_2$ ), 3.95 d.d (1H, <i>cis</i> -CH=C, $^2J = 1.8$ , $^3J_{\text{cis}} = 6.7$ ), 4.13 d.d (1H, <i>trans</i> -CH=C, $^2J = 1.8$ , $^3J_{\text{trans}} = 14.3$ ), 6.46 d.d (1H, $\text{OCH}=\text{C}$ , $^3J_{\text{cis}} = 6.7$ , $^3J_{\text{trans}} = 14.3$ ), 7.39 s (1H, $\text{N}=\text{CH}$ ), 7.41 s (1H, $\text{N}=\text{CH}$ )
<b>IVa</b>	1605, 1620 (C=C); 1660 (C=N); 3050, 3105 (=C-H)	1.82 d (3H, $\text{CH}_3$ ), 3.55–3.71 m (4H, $\text{NCH}_2\text{CH}_2\text{O}$ ), 3.89 d.d (1H, <i>cis</i> -CH=CO, $^2J = 2.5$ , $^3J_{\text{cis}} = 7.0$ ), 4.09 d.d (1H, <i>trans</i> -CH=CO, $^2J = 2.5$ , $^3J_{\text{trans}} = 14.1$ ), 6.10 m (2H, $\text{CH}=\text{CH}$ ), 6.35 d.d (1H, $\text{OCH}=\text{C}$ , $^3J_{\text{cis}} = 7.0$ , $^3J_{\text{trans}} = 14.1$ ), 7.77 m (1H, $\text{N}=\text{CH}$ )
<b>IVb</b>	1605, 1615, 1620 (C=C); 1660 (C=N); 3050, 3105 (=C-H)	1.85 d (3H, $\text{CH}_3$ ), 1.92 m (2H, $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 3.50 t (2H, $\text{NCH}_2$ ), 3.71 t (2H, $\text{OCH}_2$ ), 3.95 d.d (1H, <i>cis</i> -CH=CO), 4.15 d.d (1H, <i>trans</i> -CH=CO), 6.17 m (2H, $\text{CH}=\text{CH}$ ), 6.43 d.d (1H, $\text{OCH}=\text{C}$ ), 7.80 m (1H, $\text{N}=\text{CH}$ )
<b>Va</b>	1610 (C=C); 3050, 3070; 3110 (=C-H)	1.77 m (6H, $3\text{CH}_2\text{CH}_2\text{CH}_2$ ), 2.51 t (6H, $3\text{NCH}_2\text{CH}_2$ ), 3.31 s (6H, $3\text{NCH}_2\text{N}$ ), 3.70 t (6H, $3\text{OCH}_2$ ), 3.94 d.d (3H, $3\text{cis-CH}=\text{C}$ ), 4.13 d.d (3H, $3\text{trans-CH}=\text{C}$ ), 6.43 d.d (3H, $3\text{OCH}=\text{C}$ )
<b>Vb</b>	1605 (C=C); 3105 (=C-H)	1.14 s (18H, $6\text{CH}_3$ ), 3.62 s (6H, $3\text{NCH}_2\text{N}$ ), 3.73 s (6H, $3\text{OCH}_2$ ), 3.94 d.d (3H, $3\text{cis-CH}=\text{C}$ ), 4.15 d.d (3H, $3\text{trans-CH}=\text{C}$ ), 6.47 d.d (3H, $3\text{OCH}=\text{C}$ )

( $\delta$  7.40 ppm,  $^2J = 1.8$  Hz) or two singlets ( $\delta$  7.39 and 7.41 ppm). The first case is possible if the chemical shifts of the *syn* and *anti* protons are similar, and the second, if the coupling constant  $^2J$  is smaller than 0.1 Hz.

On the whole, our results show that vinyl ethers derived from amino alcohols react with aldehydes in a way typical of common primary aliphatic amines and the products obtained thereby are also typical aliphatic Schiff bases.

## EXPERIMENTAL

The IR spectra were recorded on a Specord 75IR spectrophotometer from samples prepared as thin films. The  $^1\text{H}$  NMR spectra were obtained on a Jeol FX 90Q instrument (90 MHz) at 30°C using HMDS as internal reference. The purity of the products was checked by GLC on an LKhM-80 chromatograph equipped with a thermal conductivity detector; carrier gas helium; steel column, 3000 × 3 mm, packed with 3% of OV-17 on Inerton Super (0.160–0.200 mm); oven temperature programming from 50 to 160°C at a rate of 4 deg/min.

Initial aminoalkyl vinyl ethers were prepared by vinylation of the corresponding amino alcohols with acetylene, following a procedure analogous to that reported in [7].

**Schiff bases IIIa–IIIh, IVa, and IVb (general procedure).** A solution of 0.1 mol of amine **Ia–Ie** in 100 ml of diethyl ether was cooled to 10°C, and a solution of 0.1 mol of the corresponding aldehyde in 100 ml of diethyl ether, cooled to 10°C, was added under stirring. Anhydrous magnesium sulfate was then added, and the mixture was kept for 8 h at room temperature and distilled.

***N*-Methylene-1,1-dimethyl-2-vinyloxyethylamine (IIIi).** A mixture of 5.76 g (0.05 mol) of vinyl ether **Ib**, 1.65 g (0.055 mol) of paraformaldehyde, and 50 ml of benzene was heated under reflux in a flask equipped with a Dean–Stark trap until water no longer separated. Vacuum distillation gave 5.92 g (93%) of Schiff base **IIIi**.

**1,3,5-Tris(3-vinyloxypropyl)hexahydro-1,3,5-triazine (Va).** A mixture of 5.06 g (0.05 mol) of vinyl ether **Id**, 1.65 g (0.055 mol) of paraformaldehyde, and 50 ml of benzene was heated under reflux in a flask

equipped with a Dean–Stark trap until water no longer separated. Vacuum distillation gave 5.33 g (94%) of compound **Va**.

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