

tube through them and then pressing them closed. They are adjustable for different thickness of tubing by removing the screw and screwing the jaw out or in on the brass connecting rod.

*Component list for collector control*

- 2 counters, 115 V a.c., SPDT, manual reset, Veeder Root BX-150703;
- 1 relay, 115 V a.c., SPDT, 2-3 V.A., Advance Electric GHP/1C;
- 1 relay, 28 V, 1 sec thermal delay, G-V HM-01;
- 1 wirewound adjustable resistance, 25 W, 750  $\Omega$  set for 600  $\Omega$  to supply delay relay coil from 115 V a.c. line;
- 2 non-locking pushbutton switches, DPDT, 3 A;
- 1 pilot light, 115 V a.c.;
- 2 AB sockets for chassis mounting, 6-contact, Cinch-Jones S-306;
- 2 CCT polarized plugs with cable clamps, 6-contact, Cinch-Jones P-306;
- 1 AB socket for chassis mounting, 2-contact, Cinch-Jones S-302;
- 1 CCT plug with cable clamp, 2-contact, Cinch-Jones P-302;
- 3 standard 2-pole female sockets with retaining rings, Amphenol.

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## Gas chromatography of hydroxyethyl derivatives

Hydroxyethyl derivatives are of diverse and widening interest in industry, agriculture, medicine and metabolic studies. The literature is fairly abundant with investigations concerning the role of C-hydroxylation, as well as N-hydroxylation, in the metabolic pathways for pesticidal carbamates<sup>1-4</sup> and 2-chlorotriazines<sup>5-12</sup>. N-(2-Hydroxyethyl) ethyleneimine has been proposed as a modifier for nitrogen plastics and other resins, and as an intermediate in the preparation of their polymers<sup>13</sup>.  $\beta$ -Hydroxyethyl hydrazine has been used as a growth regulator by pineapple growers to induce flowering and control the date of harvest<sup>14, 15</sup>. A nitroimidazole derivative, 1-(2-hydroxyethyl)-2-methyl-5-nitroimidazole (metronidazole) is employed extensively as a specific agent against human trichomoniasis<sup>16</sup>. The technique of  $\beta$ -hydroxyethylation has been found useful for the characterization and enhancement of water solubility for complex molecules such as rutoside<sup>17</sup>. A number of *p*-acetylbenzenesulfonylureas utilized in diabetes therapy have been shown to be rapidly absorbed and metabolized in man by reduction to their corresponding *p*- $\alpha$ -hydroxyethyl derivatives<sup>18</sup>.

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TABLE I  
GAS CHROMATOGRAPHY OF HYDROXYETHYL DERIVATIVES  
R—CH<sub>2</sub>CH<sub>2</sub>OH

Compound No.	R	Mol. wt.	Name	<i>t<sub>r</sub></i> <sup>a</sup>	
				10% <sup>b</sup> C20M	4% <sup>c</sup> C20M
1	H <sub>2</sub> N—	61	Ethanolamine	0.15	0.9, 2.75
2	CH <sub>3</sub> NH—	75	2-(Methylamino) ethanol		2.10
3	H <sub>2</sub> NNH—	76	β-Hydroxyethylhydrazine	0.30	2.15 <sup>d</sup>
4	HS—	78	2-Mercaptoethanol		3.65
5	Cl—	80	2-Chloroethanol		1.45
6		88	N-(2-Hydroxyethyl) formamide	6.7	
7	(CH <sub>3</sub> ) <sub>2</sub> N—	89	2-(Dimethylamino) ethanol		0.60
8	(CH <sub>3</sub> ) <sub>2</sub> CHNH—	103	2-(Isopropylamino) ethanol		2.90
9		103	N-(2-Hydroxyethyl) acetamide	5.6	
10		104	2-Hydroxyethyl acetate		5.7, 9.4
11		112	N-(β-Hydroxyethyl) pyrazole		3.50 <sup>d</sup>
12		115	N-(β-Hydroxyethyl) pyrrolidine		5.6
13		122	Phenethyl alcohol		2.8 <sup>d</sup>
14		123	2-(Pyridine) ethanol	1.7	
15		129	N-(β-Hydroxyethyl) piperidine		4.1
16		130	2-Hydroxyethyl methacrylate		1.70 <sup>d</sup>
17		130	1-Piperazine ethanol	1.55	4.25 <sup>d</sup>
18		131	N-(β-Hydroxyethyl) morpholine		1.95 <sup>d</sup>
19		143	N-(2-Hydroxyethyl) cyclohexylamine	1.25	
20		120	N,N'-Bis(β-hydroxyethyl) hydrazine		2.20 <sup>d</sup>

<sup>a</sup> Retention time in minutes from solvent front.

<sup>b</sup> 10% Carbowax 20M on 60–80 mesh Chromosorb W; 8 ft. by 0.125 in. O.D. stainless steel column; column temperature 220°; 50 p.s.i.g. helium; 150 mA filament current; detector temperature 250°; hot wire detector.

<sup>c</sup> 4% Carbowax 20M terminated with terephthalic acid on 60–80 mesh HMDS-pretreated Chromosorb W; 6 ft. by 0.25 in. O.D. glass coil column; column temperature 73° and nitrogen carrier flow 63 ml/min except for those designated footnote d; hydrogen 45 ml/min, air 300 ml/min; detector temperature 200°; hydrogen flame detector.

<sup>d</sup> *Ibid* to footnote c, except: column temperature 110° and nitrogen carrier 81 ml/min.

Pyrolysis-gas chromatography has been employed as an analytical technique for the determination of the hydroxyethyl group in hydroxyethyl starch *via* measurement of the liberated acetaldehyde<sup>10</sup>. The present study reports the chromatographic behavior of twenty 2-hydroxyethyl derivatives of diverse structure, including the growth regulator  $\beta$ -hydroxyethyl hydrazine and discusses the faculty of silylation in elution enhancement of the derivatives.

### Experimental

The hydroxyethyl compounds employed in this study were obtained from various commercial sources. Gas chromatography was carried out on a 10% Carbowax 20M column housed in an F & M Model 720 dual column oven containing a hot wire detector and coupled to an F & M Model 500 gas chromatograph; and on a 4% Carbowax 20M column housed in an F & M Model 1609 gas chromatograph containing a hydrogen flame detector. Specific analytical operating conditions are given in the footnotes to Table I.

### Results and discussion

The chromatographic results for the hydroxyethyl derivatives are presented in Table I. The derivatives required the use of more than one column temperature. The low temperatures employed for the chromatography of most of the derivatives (73° and 110° on 4% Carbowax 20M), were not sufficient for several of the more strongly retained compounds (*viz.* compounds 6 and 9). Hence, one may qualitatively observe the varying degrees of interaction of the 2-hydroxyethyl substituents with

TABLE II

INFLUENCE OF SILYLATION ON CHROMATOGRAPHIC ELUTION OF HYDROXYETHYL DERIVATIVES

Compound No.	R	Name	$t_r^{\circ a}$		% <sup>b</sup> Elution enhancement
			$RCH_2-CH_2OH$	$RCH_2-CH_2OSi(CH_3)_3$	
16		2-Hydroxyethyl methacrylate	1.70	0.60	65
18		N-( $\beta$ -Hydroxyethyl) morpholine	1.95	0.90	54
3	H <sub>2</sub> NNH—	$\beta$ -Hydroxyethylhydrazine	2.15	0.60	72
20		N,N'-Bis( $\beta$ -hydroxyethyl)-hydrazine	2.20	0.60, 1.90	73, 14
13		Phenethyl alcohol	2.80	0.80	71
11		N-( $\beta$ -Hydroxyethyl) pyrazole	3.50	0.55, 0.90	84, 74
17		1-Piperazine ethanol	4.25	2.00	53

<sup>a</sup> For column and conditions see footnote d, Table I.

<sup>b</sup> Calculated by:  $100 \frac{[t_r^{\circ}(RCH_2CH_2OSi(CH_3)_3)]}{t_r^{\circ}(RCH_2CH_2OH)} 100$

the polar polyglycol stationary phase. The carbonyl derivatives (compounds 6, 9, 10 and 16) illustrate to some degree the influence of dipole-dipole type interaction and its influence on retention. Hydrogen bonding, on the other hand, is also of considerable importance to solute retention, as may be seen for example, between compounds 2 and 7, or between compounds 17 and 15 or 17 and 18. The relatively large retentions observed for compounds 6 and 9 may well be attributed to hydrogen bonding *via* the tautomerized enol form of the solute.

The influence of hydrogen bonding *via* the hydroxyl proton on the chromatographic behavior of hydroxyethyl derivatives may be more strikingly observed by examination of the elution results on 4% Carbowax 20M at 110° between several hydroxyethyl compounds and their trimethylsilyl derivatives given in Table II. Trimethylsilylation was achieved by reaction with hexamethyldisilazane in a pyridine medium, initiated with trimethylchlorosilane. Elution enhancement *via* preparation of the silyl ether derivatives was generally in the order of 50–80%. The two peaks observed during chromatography of the silylated N,N'-bis( $\beta$ -hydroxyethyl)hydrazine (compound 20) was believed to be due to a mixture of the mono- and disilylated derivatives.

In view of the important role of hydroxylation in the metabolic pathway of herbicides, other classes of alcoholic, as well as phenolic derivatives are presently under investigation.

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