

NOTE

High Performance Liquid Chromatography Determination of Formitol

N. FOROUGHIFAR, A. MOBINKHALEDI*, M. TAJBAKHSHT† and H. NAEIMI‡

Department of Chemistry, Arak University, Dr. Beheshti Ave. Arak, Iran

E-mail: akbar_mobini@yahoo.com

Formitol, industrial or synthetic polyols, is prepared commercially by the catalytic hydrogenation of formose solution. High performance liquid chromatography was used for separation and identification of polyols, which are available in synthesized formitol. Polyols such as sorbitol (retention time = 13.01 min), glycerol (retention time = 15.44 min), ethylene glycol (retention time = 17.18 min) were the major polyhydroxy compounds in formitol which were analyzed by HPLC technique.

Key Words: HPLC, Determination, Formitol.

Formose solution is a mixture of linear and possibly branched sugars, which comes from formaldehyde and carbohydrate in alkaline solution¹. Hydrogenation of formose solution with sodium borohydride²⁻⁵ or catalyst^{6,7} produces polyhydroxylated compound which is called formitol. Formitol can be used as crosslinkers in the preparation of polyurethane resins, swelling agents and anti-freezers. Several workers have reported the separation and identification of polyhydroxylated compounds by paper chromatography⁸, thin layer chromatography⁹, paper electrophoresis¹⁰ and gas chromatography¹¹. This work describes the use of high performance liquid chromatography for separation and determination of polyols made from catalytic hydrogenation of formose.

HPLC model Shimadzu LC-5A equipped with two pumps and valve 3IL-LA; Column Oven Unit: CTO-2A; Detector: RID-2A; Recorder: Varian-440; Column: Shimpack-SCR-101 N 300 mm × 7.9 mm; Ultrasonic bath: Kreey-Pul 250

Run Conditions

Column temperature: 60°C; rate of flow: 0.5 mL/min; mobile phase: water and solvent: water.

Procedure: To undertake the analyses of synthesized polyols 10 µL of sample was injected on to HPLC. Also in similar experiment 10 µL of reference standard solution was injected onto HPLC under the same conditions. Then

†Department of Chemistry, Mazandaran University, Babolsar, Iran.

‡Department of Chemistry, Kashan University, Kashan, Iran.

comparison of retention times and peak areas of standard and unknown solution chromatograms led to identification of polyols.

The retention times and peak areas of polyols in the reference solution chromatogram were compared to those of unknown solution chromatogram. The concentration of each polyol in solution was calculated using:

$$C_x = F_x \times A_x$$

where C_x is concentration of sample in mg/L and A_x is peak area of polyol x.

For determination of concentration factor (or dilution factor), 10 μ L of sample was injected to HPLC and the factor calculated as follows:

$$F_x = C_{S_x} / A_{S_x}$$

The retention times and concentration factors for some hydroxy and polyhydroxy compounds (polyols) in reference standard solution which obtained from this equation are listed in Table-1.

Figs. 1 and 2 represent HPLC chromatograms of the standard and unknown polyol solutions respectively. These chromatograms were recorded under the same conditions.

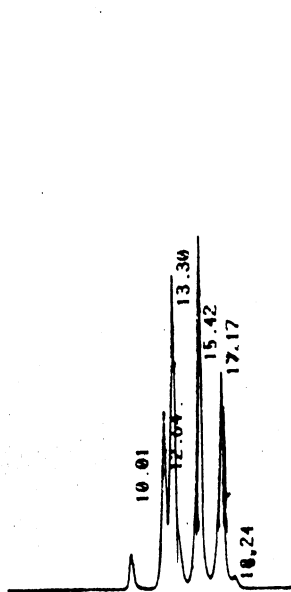


Fig. 1. HPLC chromatogram of standard polyols solution

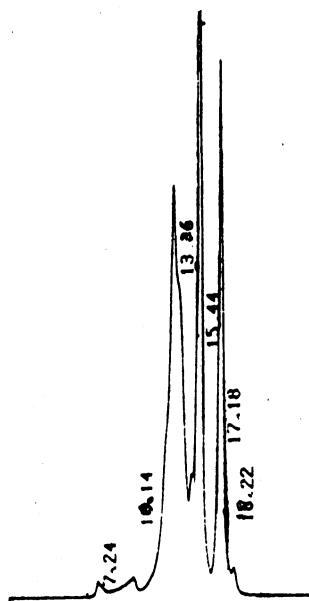


Fig. 2. HPLC chromatogram of unknown polyols solution

As we compare two chromatograms, it shows that formitols are mainly contain polyols such as sorbitol (retention time = 13.30 min), glycerol (retention time = 15.44 min), and ethylene glycol (retention time = 17.18 min). The peak with low retention-time (retention time = 7.24 min) is probably due to existence of some disaccharides in which only one ring hydrogenated.

TABLE-1

Hydroxy and Polyols	F _x	Retention time (min)
Lactol	1.60	10.01
Mannitol	0.20	12.62
Sorbitol	0.31	13.30
Glycerol	0.31	15.40
Ethylene glycol	0.34	17.16
Methanol	2.01	18.20

REFERENCES

1. A. Butlerow, *Compt. Rend.*, **53**, 145 (1861).
2. A.H. Weiss, R.F. Socha, V.A. Likholobov and M.M. Sakharov, *Chemtech.*, **644** (1980).
3. A.H. Weiss, R.B. Lapierre and J. Shapira, *J. Catal.*, **8**, 332 (1970).
4. A.H. Weiss, H. Tambawala, R.D. Partridge and J. Shapira, *J. Dechema Monogr.*, **68**, 239 (1971).
5. T. Mizuno, Report of the Faculty of Agriculture, Shizuoka University, **24**, 49 (1974).
6. H.P. Muller and K. Wagner, U.S. Patent, 4 156 636 (1979).
7. J.R. Skinner, U.S. Patent, 3 260 759 (1966).
8. C.F. Smulin. L. Hartmann and R.S. Stetzler, *J. Am. Oil. Chem. Soc.*, **35**, 179 (1958).
9. G.W. Hay. B.A. Lewis and F. Smith, *J. Chromatogr.*, **11**, 479 (1963).
10. E.M. Less and H. Weigel, *J. Chromatogr.*, **16**, 36 (1964).
11. L. Domm, D. Decleck and H. Verachtert, *J. Chromatogr.*, **42**, 349 (1969).

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Contact:

PROFESSOR SERGIO PINZAUTI

E-mail: info@pba2004.com