Asian Journal of Chemistry; Vol. 27, No. 2 (2015), 781-782

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2015.18092

NOTE

HPLC Determination of Four Derivatives of Benzene

YUN WANG* and YAN-LI XI

School of Chemistry and Chemical Engineering, Lingnan Normal University, Zhanjiang 524048, P.R. China

*Corresponding author: Fax: +86 759 3183510; Tel: +86 759 3183176; E-mail: wangyun203@hotmail.com

Received: 28 May 2014; *Accepted*: 5 August 2014; *Published online*: 10 January 2015; AJC-16671

In the paper, the HPLC determination condition for four derivatives of benzene including 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'-fluoroacetophenone has been improved. The trifluoroacetic acid has been applied in the HPLC determination condition for the four compounds for the first time. The ODS-C_{18} (4.6 × 150 mm, 5 μm) column was used, The mobile phase was a mixture of acetonitrile-0.1 % trifluoroacetic acid aqueous solution (55:45 or 50:50). The wavelength of determination was 254 nm. The four compouds have been separated throughly with the HPLC determination condition. This method is accurate, simple and rapid and suitable to laboratory and industry.

Keywords: Trifluoroacetic acid, Four derivatives of benzene, HPLC.

The 1, 4-phthalaldehyde (Fig. 1), nitroxinil (Fig. 2), 1,2,4,5-tetrafluorobenzene (Fig. 3) and 2-chloro-4'-fluoroacetophenone (Fig. 4) which are the derivatives of benzene are very important fine chemical intermediates for medicines, agrichemicals and fine-chemicals[1-8]. In the laboratorial and industrial production, the estimate of the reaction time and the analysis of the four compounds are all important problems. The four compounds have been detected and quantified by different methods, *e.g.*, thin layer chromatography (TLC)[9-12], gas chromatography (GC)[13-16] and gas chromatography combined with mass spectrometry (GC-MS)[17]. But the high performance liquid chromatography (HPLC) method has been received attention rarely.

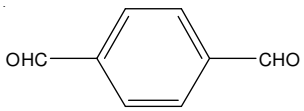

Fig. 1. Structure of 1,4-phthalaldehyde

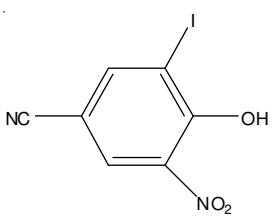

Fig. 2. Structure of nitroxinil

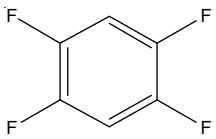

Fig. 3. Structure of 1,2,4,5-tetrafluorobenzene

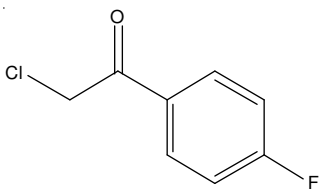

Fig. 4. Structure of 2-chloro-4'-fluoroacetophenone

Trifluoroacetic acid (TFA) is promising key intermediates because of easy transformation to many chemicals such as fine-chemicals, pharmaceuticals and agrichemicals[18,19]. It is also a good solvent for many organic chemicals[20,21], a good catalyst for the esterification reaction and condensation reaction[22,23]. Moreover, trifluoroacetic acid is usually used in the HPLC method to adjusting the pH value of the mobile phase[24-27]. The purpose of the present investigation is to apply the TFA in the HPLC method, improve the HPLC determination conditions for 1,4-phthalal-dehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'-fluoroacetophenone and to establish an accurate, sensitive and stable analytical method that is suitable to laboratory and industry.

High performance liquid chromatography system: The DIONEX P680 HPLC system was equipped with a P680A programmable pump, a UVD 170U detector and 7725 manual injector. The ODS-C_{18} (4.6 × 150 mm, 5 μm) column (Supelco, Shanghai, China) was used. The chromatographic system was controlled by using the Chameleon chromatography manager software (version 6.80).

The 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'-fluoroacetophenone were synthesized by ourselves. Acetonitrile and trifluoroacetic acid were chromatographic grade. Ultrapure water was used. All other chemicals were analytical reagent grade.

Preparation of standard solutions of four compounds: Standard solutions of 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'-fluoroacetophenone were prepared by dissolving an accurately weighed quantity (about 15 mg) of 1,4-phthalaldehyde, nitroxinil and 1,2,4,5-tetrafluorobenzene in acetonitrile-0.1 % trifluoroacetic acid aqueous solution (55: 45), respectively and 2-chloro-4'-fluoroacetophenone in the same mixed solution (50: 50) and adjusted to the l00 mL mark. These solutions were analyzed directly by HPLC.

High performance liquid chromatography system operation conditions: The mobile phase was the mixture of acetonitrile 0.1 % trifluoroacetic acid aqueous solution (55:45 or 50:50) with a flow rate of 1 mL/min. Samples were kept at room temperature before HPLC. It was filtered and degassed before use. Detection wavelength was 254 nm. Each sample was filtered by a 0.45 μm filter and injected 5 μL each time. All chromatographic analysis were performed isocratically and at room temperature.

The DIONEX P680 HPLC system was equipped with a UVD 170U detector, which can detect samples at four kinds detection wavelength simultaneously. The 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'- fluoroacetophenone were detected at 220, 230, 240 and 254 nm, respectively. The wavelength (nm) max. absorbances of the four samples are all 254 nm, so 254 nm was chose as detection wavelength.

The 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'-fluoroacetophenone were detected at a flow rate of 0.5, 1.0 and 1.5 mL/min, respectively. The retention time of four samples varies from 10 to 16 min. at a flow rate of 0.5 mL/min, the detection time is longer. The retention time of four samples is too short not to be separated thoroughly and the peaks overlap at a flow rate of 1.5 mL/min. So the flow-rate of 1 mL/min was chose for good separating effect.

At HPLC operation conditions described above, the retention time was 2.612, 4.022 and 6.025 min for the 1,4-phthalaldehyde, nitroxinil), 1,2,4,5-tetrafluorobenzene, respectively using a mixture of acetonitrile 0.1 % aqueous solution of trifluoroacetic acid (55: 45) as mobile phase. The retention time was 6.193 min for the 2-chloro-4'-fluoroacetophenone using a mixture of acetonitrile 0.1 % aqueous solution of trifluoroacetic acid (50: 50) as mobile phase. The content of four samples with area normalization method is 98.68, 99.54, 99.41 and 99.42 % for the 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluoro-benzene and 2-chloro-4'-fluoroacetophenone, respectively.

Conclusion

In the paper, the HPLC determination condition for four derivatives of benzene including 1,4-phthalaldehyde, nitroxinil, 1,2,4,5-tetrafluorobenzene and 2-chloro-4'- fluoroacetophenone has been improved. Trifluoroacetic acid has been applied in the HPLC determination conditions for the four compounds for the first time. The high performance liquid chromatography with UV detection method has been improved and validated for the four derivatives of benzene. The mobile phase is a mixture of acetonitrile 0.1 % aqueous solution of trifluoroacetic acid which only has three components to be made up easily. The detection wavelength was 254 nm. This method was accurate, sensitive, stable, reproducible and suitable to routine analysis.

REFERENCES

1. Z.H. Chen, C.J. Zheng, L.P. Sun and H.R. Piao, *Eur. J. Med. Chem*., **45**, 5739 (2010).
2. B.L. Li, P.H. Li, X.N. Fang, C.-X. Li, J.-L. Sun, L.-P. Mo and Z.-H. Zhang, *Tetrahedron*, **69**, 7011 (2013).
3. A.B.A. Boxall, Comparative and Veterinary Pharmacology, Handbook of Experimental Pharmacology, Vol. 199, pp. 291-314 (2010).
4. T. Grabowski, J.J. Jaroszewski and W. Piotrowski, *Toxicol. In Vitro*, **24**, 953 (2010).
5. A. Streitwieser, D.Z. Wang, M. Stratakis, A. Facchetti, R. Gareyev, A. Abbotto, J.A. Krom and K.V. Kilway, *Can. J. Chem*., **76**, 765 (1998).
6. J. Leroy, B. Schöllhorn, J.L. Syssa-Magalé, K. Boubekeur and P. Palvadeau, *J. Fluor. Chem*., **125**, 1379 (2004).
7. J.M. Cobb, N. Grimster, N. Khan, J.Y.Q. Lai, H.J. Payne (née Gold), L.J. Payne, T. Raynham and J. Taylor, *Tetrahedron*, **43**, 7557 (2002).
8. D. Zhu, B.A. Hyatt and L. Hua, *J. Mol. Catal. B*, **56**, 272 (2009).
9. M. Rostami, A.R. Khosropour, V. Mirkhani, I. Mohammadpoor-Baltork, M. Moghadam and S. Tangestaninejad, *Comptes Rendus Chim*., **14**, 869 (2011).
10. G. Milhaud, G. Keck and D. Courtot, *Vet. Res. Commun*., 107 (1983).
11. A.S. Kende, J.J. Wade, D. Ridge and A. Poland, *J. Org. Chem*., **39**, 931 (1974).
12. A.J. Heeger, F. Wudl and C.W. Dirk, US Patent 4, 626, 586 (1986).
13. M. Schlosser and E. Marzi, *Chem. Eur. J.*, **11**, 3449 (2005).
14. M. Stratakis, P.G. Wang and A. Streitwieser, *J. Org. Chem*., **61**, 3145 (1996).
15. A. Meudt, R. Wisdom, C. Boehm *et al*., US Patent 0206826 A1 (2008).
16. K. Takeba and M. Matsumoto, *Jpn. J. Public Health*, **39**, 75 (1992).
17. T. Hirano, T. Arai and M. Kinouchi, US Patent 0115695 A1 (2006).
18. K. Li, L.N. Foresee and J.A. Tunge, *J. Org. Chem*., **70**, 2881 (2005).
19. M.A. Deeg, D.R. Humphrey and S.H. Yang, *J. Biol. Chem*., **267**, 18573 (1992).
20. M. Hasegawa, A. Isogai, F. Onabe and M. Usuda, *J. Appl. Polym. Sci*., **45**, 1857 (1992).
21. A. Otaka, T. Koide, A. Shide and N. Fujii, *Tetrahedron Lett*., **32**, 1223 (1991).
22. E.J. Bourne, M. Stacey, J.C. Tatlow and J.M. Tedder, *J. Chem. Soc*., 2976 (1949).
23. A.S. Castanet, F. Colobert and P.E. Broutin, *Tetrahedron Lett*., **43**, 5047 (2002).
24. Z. Zhong, Q. Ji and J.A. Zhang, *J. Pharm. Biomed. Anal*., **51**, 947 (2010).
25. M. Ciulu, S. Solinas, I. Floris, A. Panzanelli, M.I. Pilo, P.C. Piu, N. Spano and G. Sanna, *Talanta*, **83**, 924 (2011).
26. G. Venkatasami and J.R. Sowa Jr., *Anal. Chim. Acta*, **665**, 227 (2010).
27. C. Guarino, F. Fuselli, A.L. Mantia and L. Longo, *Food Chem*., **127**, 1294 (2011).