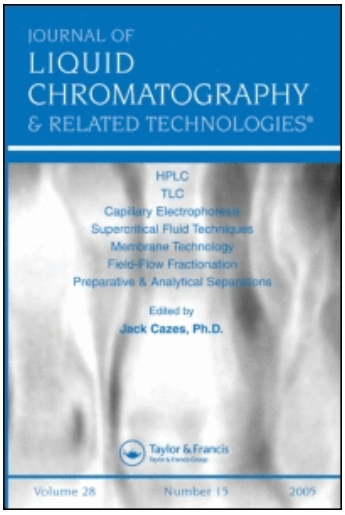

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

The Determination of Sodium Monofluoroacetate (Compound 1080) in Formulation and Technical Samples by High Pressure Liquid Chromatography

W. J. Allender[a]
[a] Chemistry Branch Biological and Chemical Research Institute PMB 10, Rydalmere, New South Wales, Australia

To cite this Article Allender, W. J.(1990) 'The Determination of Sodium Monofluoroacetate (Compound 1080) in Formulation and Technical Samples by High Pressure Liquid Chromatography', Journal of Liquid Chromatography & Related Technologies, 13: 17, 3465 — 3471
To link to this Article: DOI: 10.1080/01483919008049114
URL: http://dx.doi.org/10.1080/01483919008049114

JOURNAL OF LIQUID CHROMATOGRAPHY, 13(17), 3465-3471 (1990)

THE DETERMINATION OF SODIUM MONOFLUOROACETATE (COMPOUND 1080) IN FORMULATION AND TECHNICAL SAMPLES BY HIGH PRESSURE LIQUID CHROMATOGRAPHY

W. J. ALLENDER
Chemistry Branch
Biological and Chemical Research Institute
PMB 10, Rydalmere
New South Wales, 2115 Australia

ABSTRACT

A high pressure liquid chromatographic (HPLC) procedure was developed for the determination of sodium monofluoroacetate (Compound 1080). The procedure utilized an amine (NH_2) bonded column for the reverse phase determination of sodium monofluoroacetate in formulation and technical samples.

INTRODUCTION

Sodium monofluoroacetate, commonly called Compound 1080, is a water soluble, potent mammalian poision used in baits as a rodenticide [1] and for control of rabbits, dingoes, pigs and foxes in Australia [2-4]. Poisoning with 1080 is generally the recommended method for reducing large numbers of feral animals to manageable levels because it is cheap, easy to use and effective. However, due to a wide variation in toxicity between different animal species to the poison [2,5,6], it is important to ensure that the correct amount of sodium monofluoroacetate is added to the mixed - bait prior to a pest eradication programme. This is necessary in order to gain some degree of selectivity and reduce risk to non-target species [6].

Several approaches based on the formation of either volatile derivatives of the parent acid, monofluoroacetic acid (MFA) for gas-liquid chromatography (GLC) [7,8] or by ion-selective electrode determination, after hot aqueous alkaline defluoridation of the sodium salt [9], were developed and utilized within the laboratory for the determination of 1080 content in tissues and baits. Although these methods were adequate for many applications, a search was initiated to assess the potential of applying HPLC to the analysis of formulation and technical samples without the need for derivatisation [10]. A rapid and sensitive analytical procedure based on high pressure reverse phase chromatography was developed and is the subject of this paper. The procedure can be used to measure 1080 in aqueous formulations and technical material prepared by authorized control officers.

EXPERIMENTAL

Chromatographic conditions

The LC system consisted of a Model 6000A pump, a U6K injector, Model 450 variable UV wavelength detector, and a Model 730 data module (Waters Associates, Milford, Massachusetts). The chart speed of the module recorder was set at 0.5cm/min.

A 250 mm x 4 mm LiChrosorb-NH_2 column (E. Merck, Darmstadt, Germany) reverse-phase, particle size 10 μm) was used at a flow rate of 1.5mL/min. Detection was monitored at 210 nm, an sensitivity was set at 0.1 Aufs.

The mobile phase consisted of methanol (20%v/v) and distilled water (80% v/v) containing 0.05 M potassium hydrogen phosphate (KH_2PO_4), pH was adjusted to 4.0 with phosphoric acid. Prior to injection the column was stabilized with the eluant mixture at a flow rate of 0.7ml/min for approximately 1 hr.

Standard and Sample Preparation

Sodium monofluoroacetate ('1080') analytical grade (97.0%) was obtained from E. Merck (Darmstadt, Germany). Standard solution was prepared by accurately weighing about 200 mg of

Table 1. Determination for Sodium Monofluoroacetate (Compound 1080)

Sample No.	Wt % of '1080' by F^- Ion Selective Electrode Method	HPLC Wt % of '1080' 1*	2*	Average	Standard Deviation
1	93.8	90.98, 90.94	91.19, 90.95	91.01	0.10
2	87.0	86.22, 85.45	85.27, 84.98	85.48	0.46
3	97.8	95.07, 94.83	95.07, 94.83	94.89	0.10
4	96.3	95.45, 94.61	94.39, 94.06	94.63	0.51
5	3.15	3.19, 3.16	3.21, 3.17	3.18	0.02
6	3.60	3.57, 3.56	3.60, 3.51	3.56	0.03
7	2.48	2.51, 2.49	2.53, 2.50	2.50	0.02
8	2.67	2.68, 2.71	2.68, 2.81	2.72	0.05
9	2.84	2.82, 2.80	2.81, 2.81	2.81	0.01
10	3.24	3.21, 3.21	3.20, 3.22	3.21	0.01

* 1 and 2 duplicate samples with 2 injections

sodium monofluoroacetate into a 100mL of volumetric flask and diluting to the mark with distilled water containing 20% v/v of methanol. A calibration curve was constructed using six concentrations of the active constituent and calculated by external quantitation. All samples were prepared by either accurately weighing (technical grade powders) or pipetting (liquid formulations) appropriate sample amounts and diluting with water containing 20% v/v of methanol.

Peak Identification and Thin-Layer Chromatography

The general procedure for sample preparation was to collect the unknown fraction, evaporate it to dryness, and then dissolve the dried sample in distilled water (20-50 μL) for thin-layer chromatography (TLC). This solution was then spotted onto microcrystalline K2F cellulose (250 μ thickness) TLC plates (Whatman, Madstone, England) and developed in methanol/concentrated ammonia/pyridine/water (95:3:1:1). The plates were dried and sprayed with Nile Blue (Basic Blue 12) solution. The Nile Blue reagent was prepared based on Vickery *et. al.* [11] by dissolving 0.4 g of the compound in 100 mL of methanol, adding triethanolamine until the blue colour acquired a purple hue, and then adding a further 100 mL methanol.

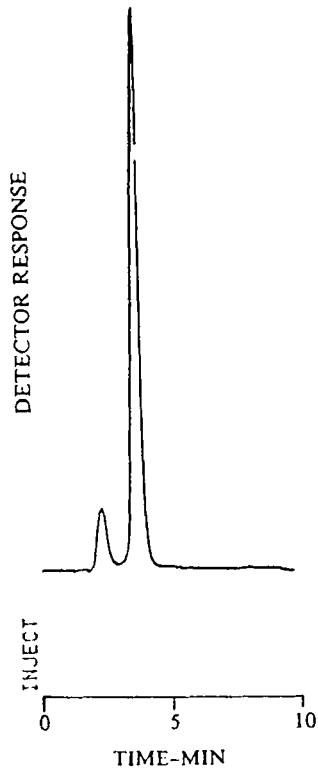

Figure 1. HPLC chromatogram of monofluoroacetic acid (MFA).

25cm x 4mm LiChrosorb - NH_2column eluted at 1.5 ml/min with MeOH-H_20 20:80 (containing 0.5M KH_2PO_4, pH 4.0) v/v. Injection: sodium monofluoroacetate (2 mg/ml) in 20 μl in mobile phase. Detection: UV 210 nm x 0.1 AUfsd.

Table 2. Thin Layer Chromatography of Fluorinated acids.

Compound	Retention Factor (Rf, x 100)
Monofluoroacetic acid	71
Difluoroacetic acid	85
Trifuoroacetic acid	95

Fluoride Ion Selective Electrode Assay

The procedure used for the fluoride ion selective assay is described elsewhere [9] and involves aqueous alkaline hydrolysis of the fluoroacetate; results are reported in Table 1.

Reagents

The reagents utilized were all analytical grade quality. Methanol was purchased from Mallinckrodt (Sydney, Australia) and was spectral grade. The sodium monofluoroacetate was used as received.

RESULTS AND DISCUSSION

The determination of the sodium salt of MFA presented a difficult HPLC problem due to its strong acidic nature (pKa = 2.58) [12], its insolubility in most solvents, and its low UV absorbance. After examining several columns, it was found that the LiChrosorb$^{(R)}$amine bonded column, when used under the chromatographic conditions described, gave reproducible determinations of fluoroacetate for diverse applications. No interference from the blue marker dye used in some formulations was observed. A sample chromatogram produced under the described conditions is given in Figure 1. Under the suggested conditions (pH = 4.0) the sodium salt of fluoroacetic acid ('1080') was chromatographed as the acid. The retention time of MFA at pH 4.0 was 3.70 minutes. The results of the chemical determinations using the fluoride ion selective electrode assay procedure, compared to HPLC determinations are shown in Table 1.

Peaks eluting from the HPLC column were isolated for subsequent identification and quantitation by other independent methods. Since peak identity and component concentrations based only on retention times can be deceptive, peaks were collected in an effort to identify the components under the suggested conditions. The fractions were compared to a known standard along with several other fluorinated acids. The retention factors of several fluorinated acids are given in Table 2.

The response of UV (210 nm) was linear from 0.5 to 5 mg/mL. The detection limit was 5 μg per injection at a signal-to-noise ratio of 2. It was found that after many analyses that an external standard method using a precision injection technique (Reodyne fixed loop, 20 μL) was precise and satisfactory for routine determinations of liquid and solid preparations.

REFERENCES

[1] Worthing, C.R. and Walker, S.B. (Eds) The Pesticide Manual - A World Compendium 7th Ed. British Crop Protection Council, London, 495, 1983.

[2] McIlroy, J.C. Aust. Wildl. Res. 8, 369, (1981).

[3] McIlroy, J.C. Aust. Wildl. Res. 8, 385, (1981).

[4] Mead, R.J., Oliver, A.J., and King, D.R. Aust. J. Biol. Sci. 32, 15, (1979).

[5] Buck, W.B., Osweiler, G.D. and Van Gelder, G.A. Clinical and Diagnostic Veterinary Toxicology, Kendall/Hunt, Dubuque, 1A 233, (1976).

[6] Waithman, J. Vertebrate Pest Control Manual AG bulletin 13, NSW Department of Agriculture 16, (1982).

[7] Okuno, I. and Meeker, D.L. J. Assoc. Off. Anal. Chem. 63 49, (1980).

[8] Allender, W.J. J. Analyt. Toxicol. 14 45, (1990).

[9] Livanos, G. and Milham, P.J. J. Assoc. Off. Anal. Chem. 67 10, (1984).

[10] Collins, D.M., Fawcett, J.P. and Rammell, C.G. Bull. Environm. Contam. Toxicol. 26 669, (1981).

[11] Vickery, M.L. and Ashu, J.T. Phytochem. 12 145, (1973).

[12] Rappoport, Z. Handbook of Tables for Organic Compound Identification 3rd Ed. The Chemical Rubber Co. Cleveland, Ohio. 429 (1967).