



REVIEW

# A review on sample preparation and chromatographic determination of acephate and methamidophos in different samples



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**Abstract** Acephate and its metabolite methamidophos are common organophosphorus insecticide used for crop protection. High uses of acephate and methamidophos have induced health issues and environmental pollution. Their undesired presence in the environment is creating ecotoxicology and may harm human health. It is therefore essential to detect the presence of acephate and methamidophos even in trace level. In this review, we have tried to accommodate successful methods of detection of acephate and methamidophos in the different biological media. Their recovery and residue analysis in different media such as vegetables, human and animal tissues have also included. The most common method for their determination is based on chromatographic separation and identification. Among different chromatographic methods, LC and GC coupled with different detectors have used. But, they both need extensive pretreatment and cleanup procedure, before undergoing chromatographic separation and identification. LC coupled with mass spectrometry (LCMS) is sometime able to detect acephate and methamidophos in ppm level.

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## 1. Introduction

Acephate (*O,S*-Dimethyl acetylphosphoramidothioate) (Table 1) is an organophosphate insecticide, introduced by Chevron Chemical in 1971 (Magee, 1973) and first time registered for use by the United States Environmental Protection Agency in 1973. It has been observed that the production of acephate increased quickly from last 5 years, surprisingly in India, 10% increase in production of technical grade was observed within one year (Standing Committee on Chemicals & Fertilizers, 2012-13).

It is an insecticide registered for use on food crops, agricultural seed and non-bearing plants, institutions and commercial buildings including public health facilities, sod, golf course turf, ant mounds, and horticultural nursery plants.

In soil, plants and insects approximate half-life period of acephate is 3–6 days, although in some soils the half-life may be increased to more than 13–60 days due to variation of properties (physical, chemical and biological) of soils (Antonious and Snyder, 1994; Bouchard and Lavy, 1982; Chuanjiang et al., 2010; Yen et al., 2000). Acidic nature of soil is responsible for long life span of acephate in soils ((Antonious and Snyder, 1994; Bouchard and Lavy, 1982; Chuanjiang et al., 2010). It has been observed that, after the decomposition acephate generally converts into highly toxic methamidophos (*O,S*-dimethyl phosphoramidothioate) (Table 1) and methamidophos is also an efficient organophosphorus insecticide (Yen et al., 2000; Chuckwudebe, 1984). Methamidophos is the major metabolite of acephate. It is toxic, not only for insects but also in various components of the environment such as nontarget animals, plants, waters, and soils (Wang et al., 2013) (see Tables 2–4).

Physicochemical properties of acephate and methamidophos are signifying that both are hydrophilic and having low soil sorption with an order acephate > methamidophos (Roberts & Hutson, 1999; Tomlin, 2006). Consequently, their runoff through water medium can produce potential water

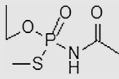
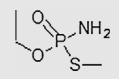
contamination (Roberts & Hutson, 1999; Tomlin, 2006). Acephate leads to contamination of groundwater much more readily than methamidophos under normal environmental conditions. Studies highlight that wet soils are more conducive for the contamination of the aquatic environment with acephate and its metabolites (Chai et al., 2009; Wang et al., 2013).

## 2. Mode of action and toxicity

Acephate and methamidophos inhibit acetylcholinesterase enzyme (AChE) in nervous system tissues, although, acephate itself is a weak acetylcholinesterase inhibitor as compared to its decomposed product methamidophos (Wilson et al., 1990; Spassova et al., 2000). The toxicity of acephate and methamidophos varies with application of enantiomeric compound of them (Wang et al., 2013). Both acephate and its metabolite methamidophos have the stereogenic phosphorus atom. The enantioselective bioactivity of enantiomers of acephate and methamidophos has been observed during past years. The R-(+)-enantiomers of acephate and methamidophos were found to be more potent to houseflies than the optical antipodes and racemates, whereas the S-(–)-enantiomers were more toxic for German cockroaches soils (Miyazaki et al., 1988; Wang et al., 2006, 2013). Another experiment showed that (–)-methamidophos was about 8.0–12.4 times more potent to the bovine erythrocytes and *Electrophorus electricus* than its (+)-form, but the (+)-enantiomer was 7.0 times more toxic to *D. magna* in 48 h tests (Lin et al., 2006). It was also concluded that D-(+)-methamidophos should be responsible for the organophosphate-induced delayed poly-neuropathy when racemic methamidophos is given to hens (Lotti et al., 1995).

Acephate and methamidophos are extremely toxic to aquatic invertebrates, birds and mammals (Tomlin, 2006; Vyas et al., 1996; Mahajna et al., 1997). The most likely route of exposure to acephate and methamidophos for the public is

**Table 1** Chemical structure and physicochemical properties of acephate and methamidophos (Roberts & Hutson, 1999; Tomlin, 2006).

Name	Chemical structure	Molecular weight (g/mol)	Solubility g/L	Log $K_{OW}$	Henry's law constant (atm m <sup>3</sup> /mol)
Acephate		183.16	790 in water at 25 °C	0.13 at 25 °C	$5.1 \times 10^{-13}$ at 25 °C
Methamidophos		141.13	200 in water at 25 °C	–0.65 at 25 °C	$8.64 \times 10^{-10}$ at 25 °C

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