

Analysis of Residual Pesticides in Foods using GC/MS (Part 2)

In Japan as for residual pesticides in food products, the official gazette designated additional 108 items in 1995, 30 in 1996, 23 in 1997 and 18 in 1998. Pesticides that had been designated before 1995 are summarized in the Databook of Residual Pesticides in Foods (C180-0091). Application News No. M192 introduces examples of EI (electron ionization) analyses for pesticides regulated after 1995 that can be analyzed by GC/MS.

This article introduces examples of analyzing these pesticides by the NCI (negative ion chemical ionization) method. The NCI method allows detection with higher sensitivity than the EI method for some compounds. Though the NCI method is especially effective for compounds containing chlorine, this article also shows examples of compounds not containing chlorine.

The EI and NCI mass spectra, as well as the SIM chromatograms at 10ppb for isofenphos, pyributicarb and fenvalerate are shown in Figs 1 to 3 respectively. Isofenphos and pyributicarb are pesticides that do not contain chlorine.

Detection of 10ppb fenvalerate is difficult with the EI method but the NCI method allows high sensitivity detection. Compounds not containing chlorine such as isofenphos and pyributicarb can also be detectable by the NCI method.

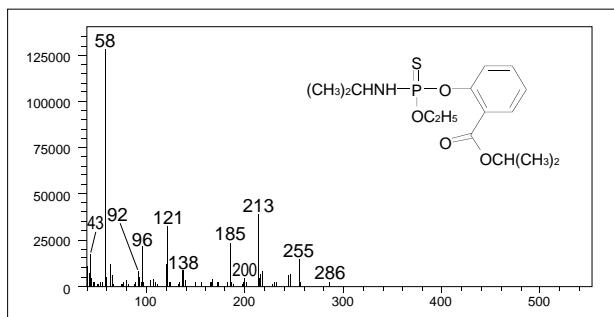


Fig.1-1 EI mass spectrum of isofenphos

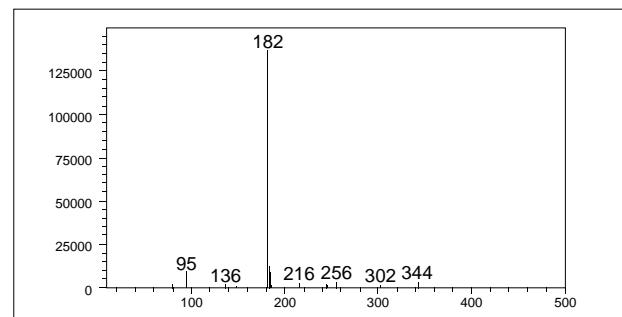


Fig.1-2 NCI mass spectrum of isofenphos

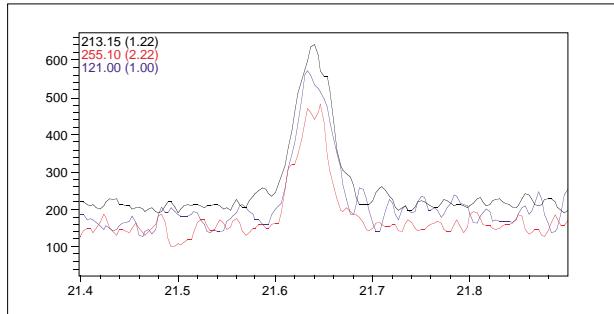


Fig.1-3 SIM chromatogram of isofenphos (EI method, 10ppb)

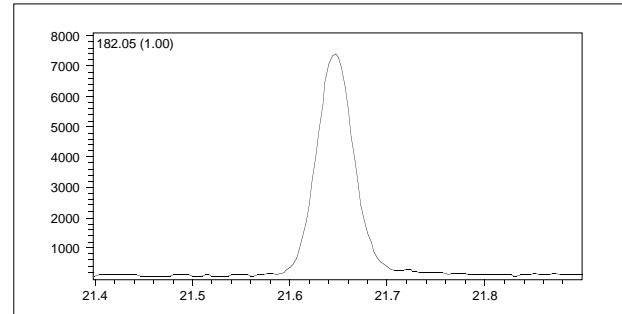


Fig.1-4 SIM chromatogram of isofenphos (NCI method, 10ppb)

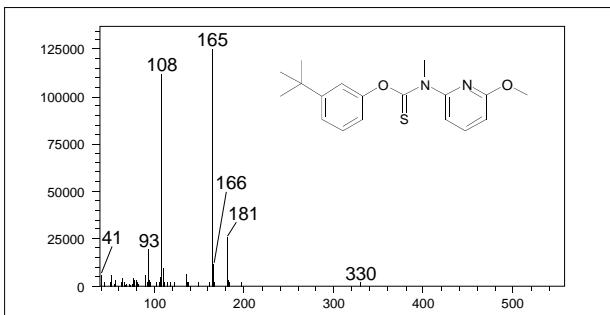


Fig.2-1 EI mass spectrum of Pyributicarb

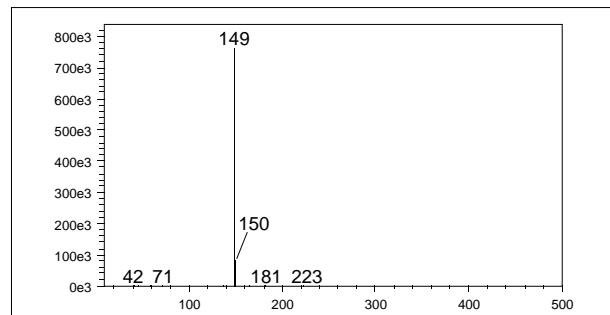


Fig.2-2 NCI mass spectrum of Pyributicarb

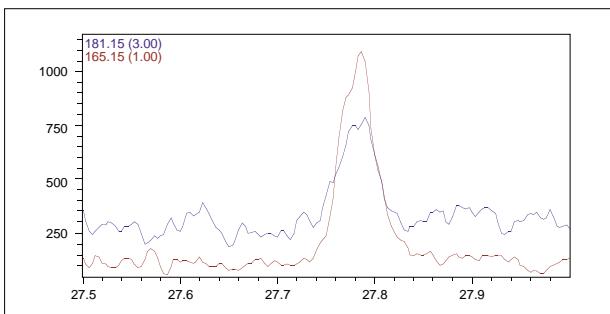


Fig.2-3 SIM chromatogram of Pyributicarb (EI method, 10ppb)

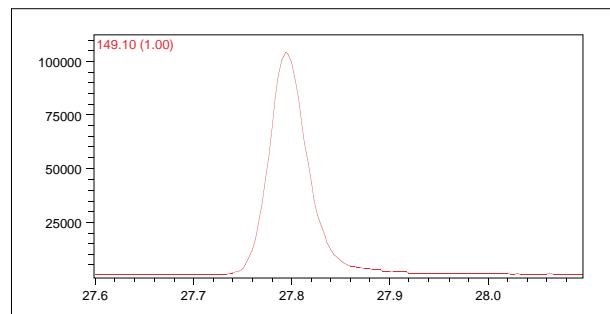


Fig.2-4 SIM chromatogram of Pyributicarb (NCI method, 10ppb)

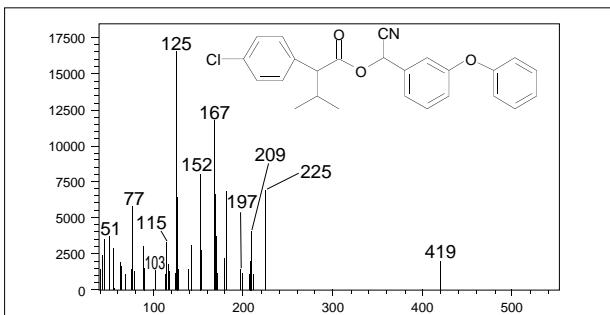


Fig.3-1 EI mass spectrum of Fenvalerate

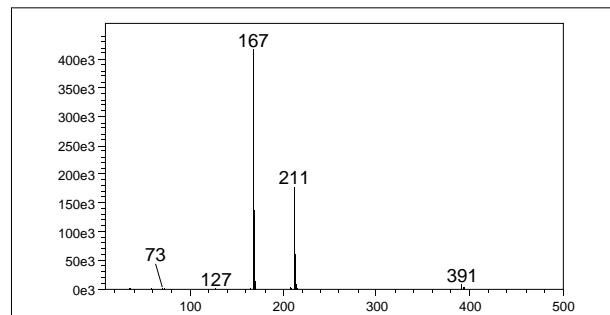


Fig.3-2 NCI mass spectrum of Fenvalerate

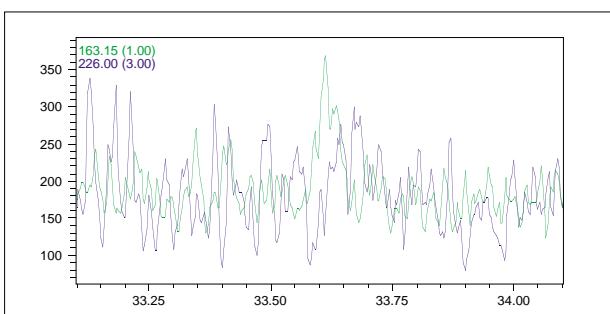


Fig.3-3 SIM chromatogram of Fenvalerate (EI method, 10ppb)

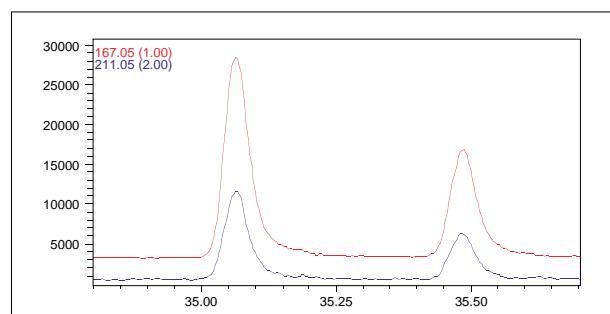


Fig.3-4 SIM chromatogram of Fenvalerate (NCI method, 10ppb)

Table 1 Analytical Conditions

GCMS	: Shimadzu GCMS-QP5050A	-MS-
-GC-		
Column	: DB-5 (30m × 0.25mm I.D. df=0.25μm)	Interface Temp. : 300°C
Column Temp.	: 50°C(1min)-20°C/min-100°C-5°C/min-300°C(1.5min)	Scan Range : EI m/z 35~550 NCI m/z 10~550
Injector Temp.	: 300°C	Ionization Method : NCI (iso-Butane)
Carrier Gas	: He 100kPa(2min)-3kPa/min-220kPa(3min) Splitless(2min)	



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Printed in Japan 3100-03304-10A-IK