ANALYSIS OF RESIDUAL AGRICULTURAL CHEMICALS USING FTD, FPD, AND ECD (NO. 4)

The Japanese Ministry of Health and Welfare revised the standard analysis methods for foods, additives, and other related substances on 27 October 1992 under the auspices of the Food Sanitation Act. It established standards and methods for measurement of residual levels of 55 agricultural chemicals in 132 agricultural products. This act was appended on 4 March 1993 for 19 additional agricultural chemicals in 74 agricultural products.

These measurement methods include gas chromatography (GC), high performance liquid chromatography (HPLC), atomic absorption spectroscopy (AA), and gas chromatograph/mass spectrometry (GC/MS). Examples of analysis with the capillary column in GC have already been described in the previously published Application News G153 and G166. The following note describes the analyses for Glufosinate, Triclamide, and Inabenfid (Figure 1), components that belong to the set of 19 newly listed agricultural chemicals. Sample pretreatment is required before analysis for these components can be done.

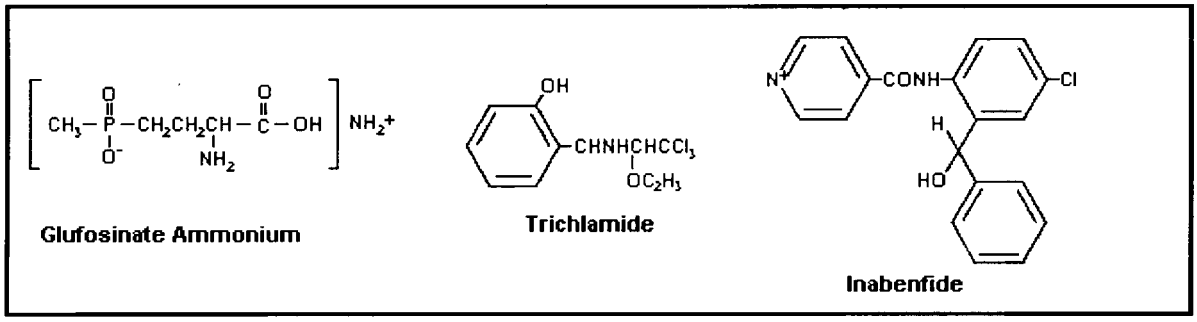

Figure 1 - Structures of Glufosinate, Triclamide, and Inabenfid

Analysis of Glufosinate and 3-Methyl Phosphine Propionic Acid by FTD

According to the instructions of the Ministry of Health and Welfare, Glufosinate and 3-methyl phosphine propionic acid must be measured by GC after methyl esterification. The concentration of 3-methyl phosphine propionic acid is added to find the final value for concentration of Glufosinate.

That method was followed here. Methyl esterification was performed on Glufosinate and 3-methyl phosphine propionic acid under acetic acid and ortho trimethyl acetate. Separation was performed using a capillary column, and flame thermionic detector (FTD) results are shown in Figure 2. Analysis of Glufosinate by HPLC after treatment by the post-column derivative method is described in Application News L231.

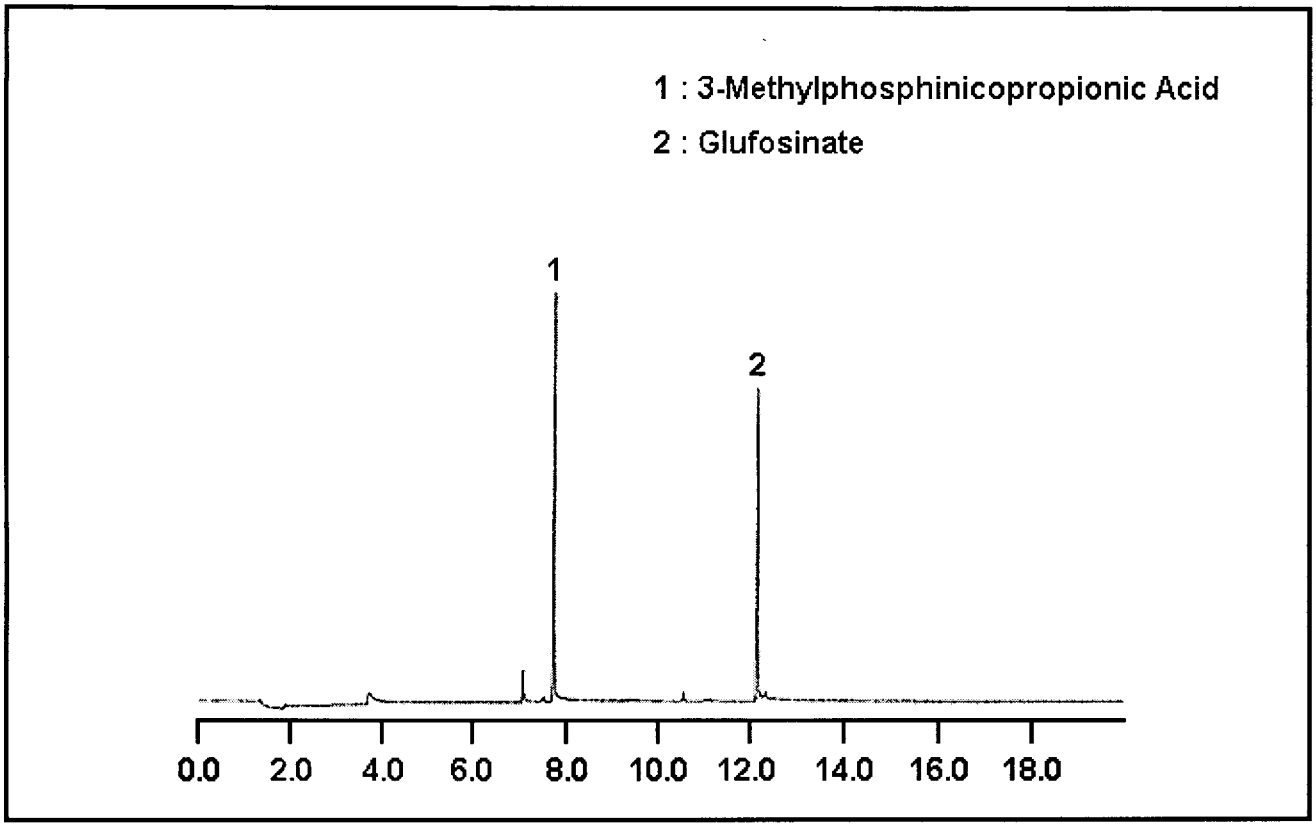

Figure 2 – Chromatogram of Glufosinate and 3-Methylphosphinicopropionic Acid with FTD

Instrument	: GC-17AAFwFt
Column	: CBJ5-S30-025 (0.32mmX30m, df=0.25μm)
Col. Temp.	: 60°C (1min) —$^{15°C/min}$→ 280°C (5min)
Detector	: FTD
Det. Temp.	: 300°C
Inj. Temp.	: 250°C
Inj. Method	: Splitless (1min)
Carrier Gas	: He, 100kPa
Make up Gas	: He, 40ml/min

Table 1 – Analytical conditions for Figure 2

Analysis of Glufosinate and 3-Methyl Phosphine Propionic Acid by FPD

Since both Glufosinate and 3-methyl phosphine propionic acid include phosphorus and nitrogen, they can be detected by either FTD or flame photometric (FPD). As in Figure 2, methyl esterification was performed, followed by separation using a capillary column. FPD results are shown in Figure 3.

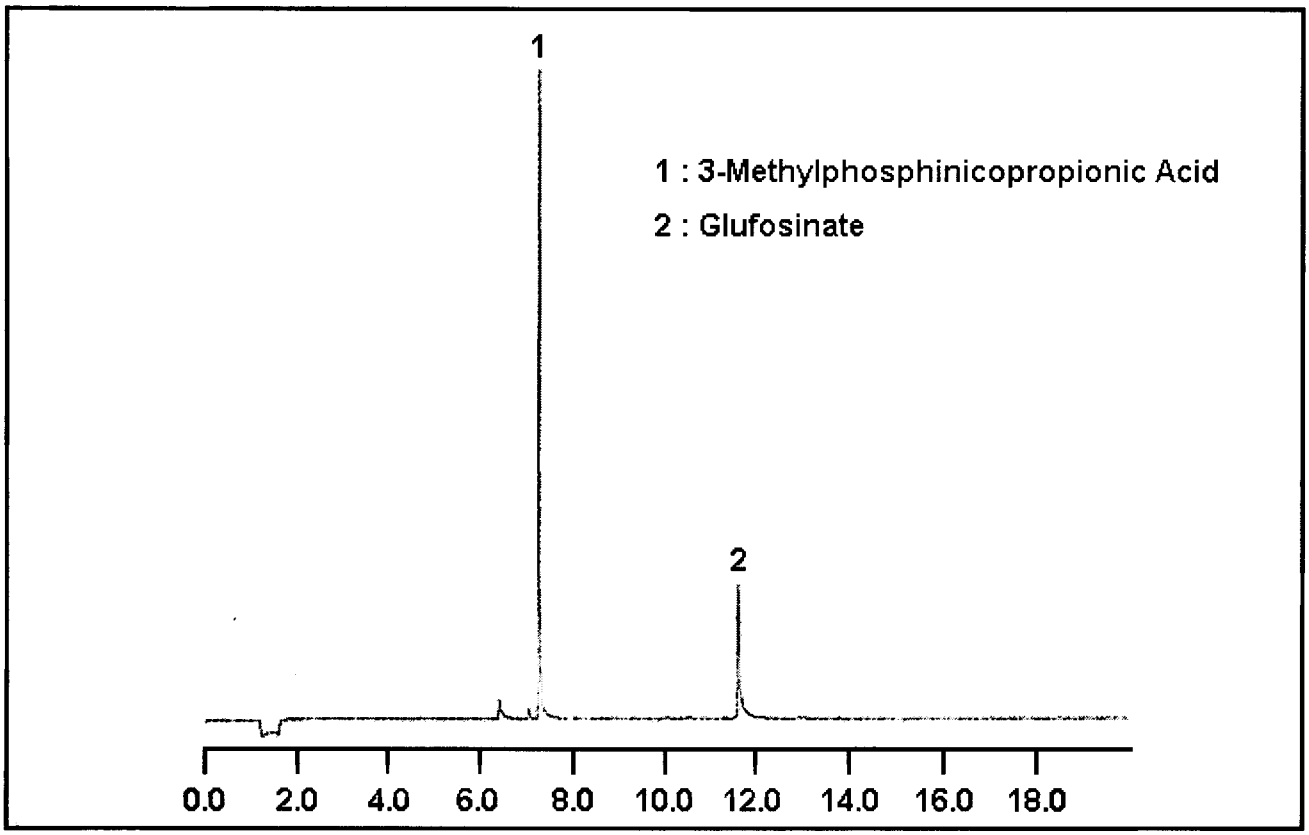

Figure 3 – Chromatogram of Glufosinate and 3-Methylphosphinicopropionic Acid with FPD

Instrument	: GC-17AAFwEp
Column	: CBJ1-S30-025 (0.32mmX30m, df=0.25μm)
Col. Temp.	: 60°C (1min) —$^{15°C/min}$→ 280°C (5min)
Detector	: FPD (P mode)
Det. Temp.	: 300°C
Inj. Temp.	: 250°C
Inj. Method	: Splitless (1min)
Carrier Gas	: He, 100kPa
Make up Gas	: He, 40ml/min

Table 2 - Analytical conditions for Figure 3

Analysis of Triclamide and Inabenfid by ECD

In the method mandated by the Ministry of Health and Welfare, Triclamide is acetylized with acetic anhydride and Inabenfid is chloroacetylized with chloroacetic anhydride. Both Triclamide and Inabenfid were separated using a packed column and detected by an electron capture detector (ECD).

An analysis of a sample injected directly into the capillary column without derivativization is shown in Figure 4.

Since the capillary column is made of inert fused silica, the instrument can provide sharp peaks even from structures that include OH radicals. The calibration curves are linear over a wide range, this indicates that the instrument will detect these molecules even at low concentrations without adsorption (Figure 5).

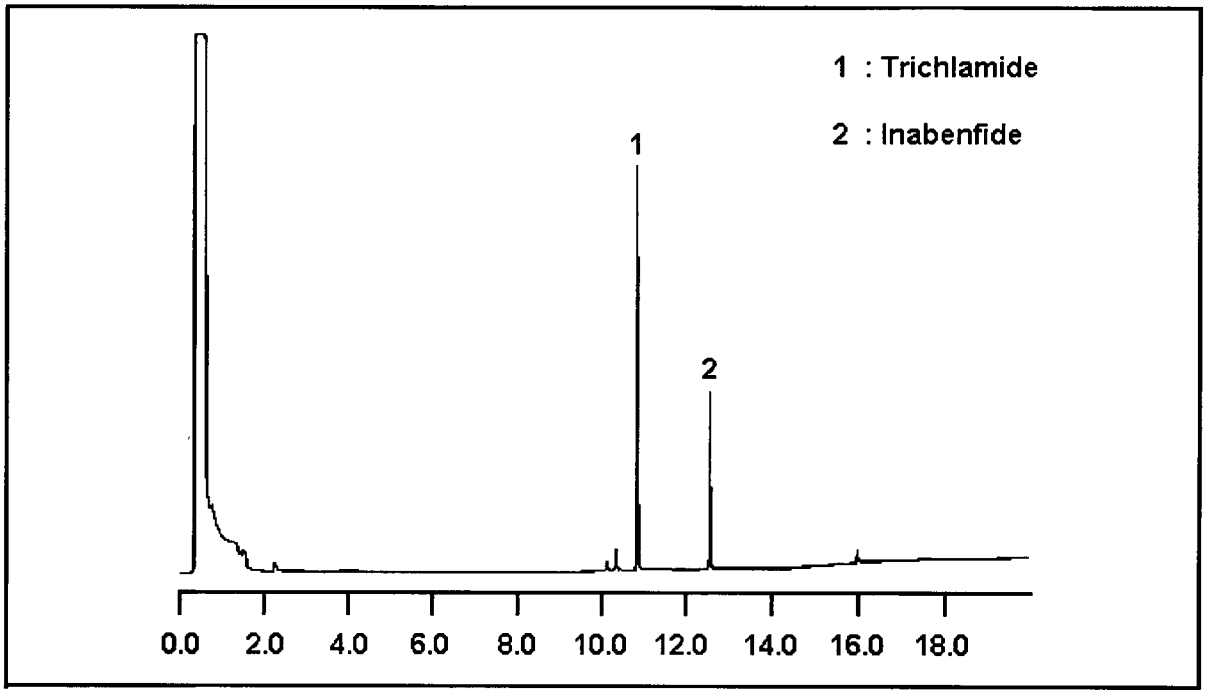

Figure 4 – Chromatogram of Triclamide and Inabenfid with ECD

Instrument	: GC-17AAFwE
Column	: CBJ1-S30-025 (0.32mmX30m, df=0.25μm)
Col. Temp.	: 60°C (1min) —$^{15°C/min}$→ 280°C (5min)
Detector	: ECD
Det. Temp.	: 300°C
Inj. Temp.	: 250°C
Inj. Method	: Splitless (1min)
Carrier Gas	: He, 100kPa
Make up Gas	: N_2, 40ml/min

Table 3 - Analytical conditions for Figure 4

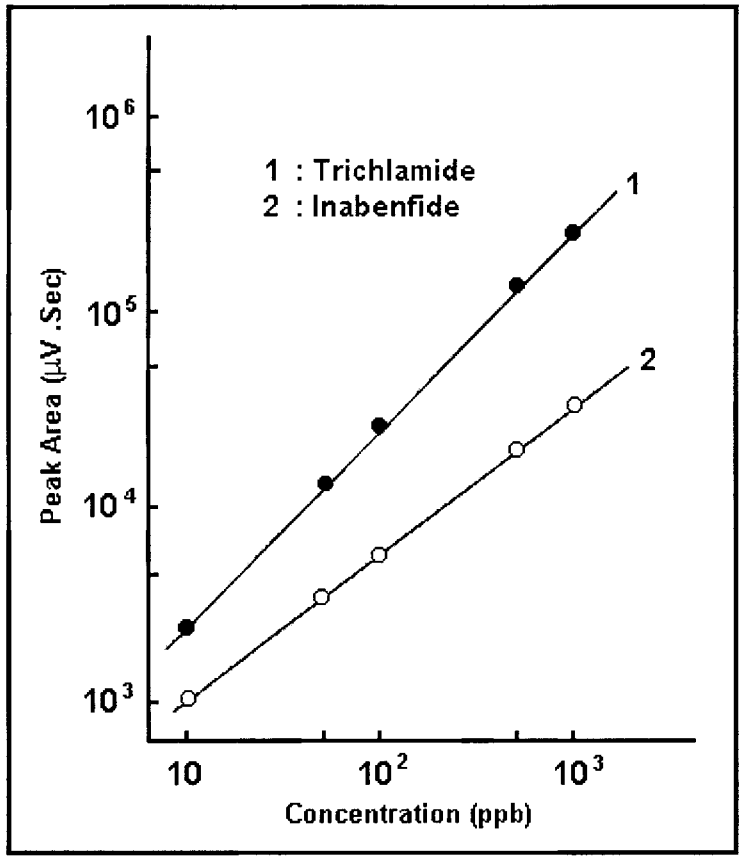

Figure 5 – Calibration Curves for Triclamide and Inabenfid