

Analysis of Dicyandiamide in Fertilizers

The dicyandiamide contained in calcium cyanamide is used as a type of nitrification suppressant to inhibit ammonia from changing into nitric acid. In accordance with the partial revision to the matters specifying official standards for general fertilizers based on the Fertilizers Regulation Act (The Ministry of Agriculture, Forestry and Fisheries of Japan; Notification No. 1985; dated August 8, 2012), the content of dicyandiamide-nitrogen in calcium cyanamide is to be 20.0 % or less of the total nitrogen content.

This article introduces an example of the analysis of dicyandiamide in fertilizer, with reference to the fertilizer test method (2012) under the supervision of the Japan's Food and Agricultural Materials Inspection Center (FAMIC).

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■ Analysis of Standard Solution

Fig. 1 shows the results of the analysis of a standard solution of dicyandiamide (10 mg/L, dissolved in methanol) using an amino column. The analytical conditions are shown in Table 1.

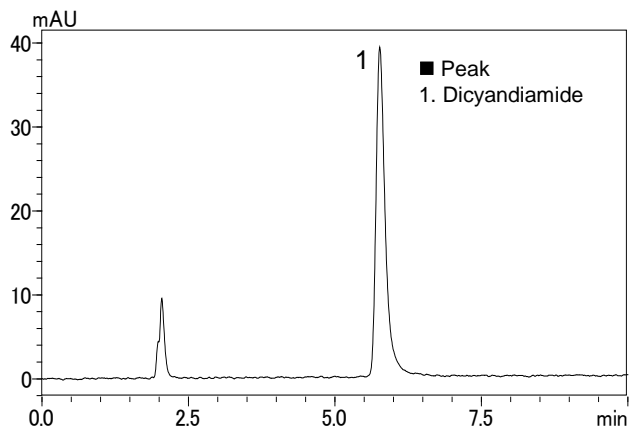


Fig. 1: Chromatogram of Dicyandiamide

Table 1: Analytical Conditions

Instrument:	Prominence HPLC system
Column:	Unison UK-Amino (Imtakt Corp., 150 mmL. × 4.6 mmI.D., 3 μm)
Mobile phase:	Acetonitrile / Methanol = 6/1 (v/v)
Flow rate:	1.0 mL/min
Column temp.:	40 °C
Detection:	SPD-20AV at 214 nm
Injection volume:	5 μL

■ Linearity and Repeatability

Fig. 2 shows the calibration curve for dicyandiamide in the range of 1 mg/L to 50 mg/L. The contribution ratio (R^2) was 0.999 or higher, indicating that favorable linearity was obtained. Calculating the detection limit and quantitation limit based on the absolute calibration curve for dicyandiamide in Fig. 2 yields 0.15 mg/L and 0.46 mg/L, respectively. Table 2 shows the relative standard deviation ($n=6$) for the peak area for each component when the dicyandiamide standard solution (1 mg/L) was analyzed. Favorable results were obtained, with a relative standard deviation of 0.48 %.

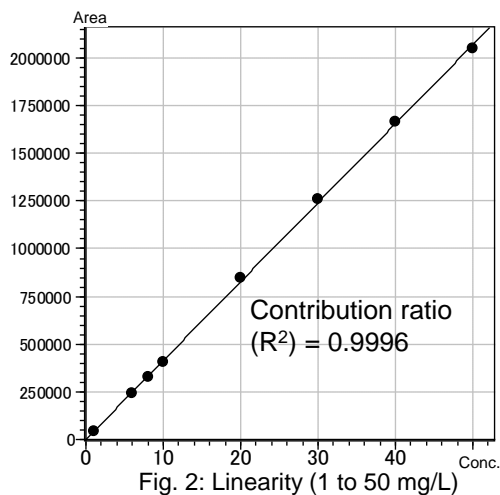


Fig. 2: Linearity (1 to 50 mg/L)

Table 2: Repeatability

	Standard solution 1 mg/L
1st	41511
2nd	41543
3rd	41107
4th	41561
5th	41143
6th	41482
%RSD	0.479

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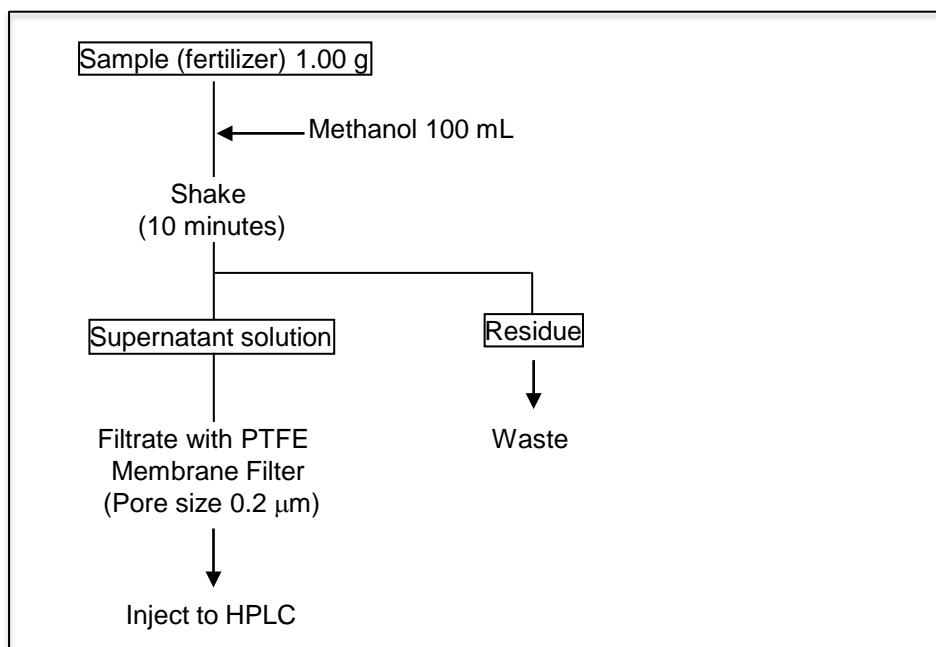


Fig. 3: Sample Preparation

Fig. 3 shows the sample preparation process described in the fertilizer test method. Fertilizer certified reference materials A and B* were treated in accordance with the sample preparation procedure described in Fig. 3. The final solutions from the sample preparation were then spiked with dicyandiamide with the standard addition technique so that the concentrations of dicyandiamide reached 10 mg/L. Figures 4 & 5 show the chromatograms obtained. The analysis conditions are the same as in Table 1. (In accordance with the sample preparation procedure in Fig. 3, if 100 % of the dicyandiamide was recovered, its content would be calculated as equivalent to a 0.1 % mass fraction with respect to the fertilizer.)

*Fertilizer certified reference material A (FAMIC-A-10) is a high-analysis compound fertilizer containing urea. Fertilizer certified reference material B (FAMIC-B-10) is a general-compound fertilizer not containing urea. Both are commercially available from FAMIC.

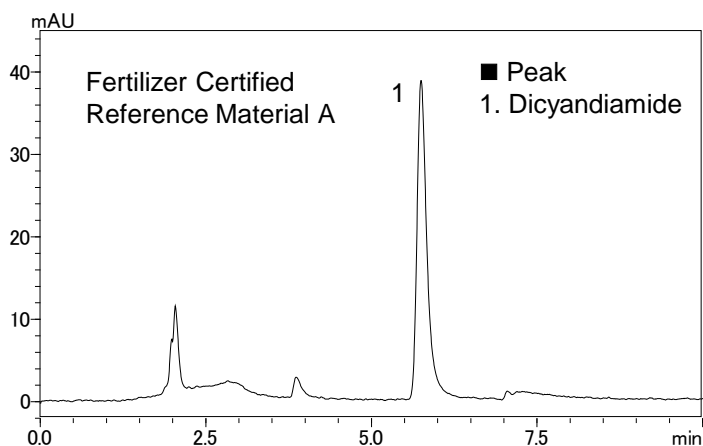


Fig. 4: Chromatogram of Fertilizer Certified Reference Material A (spiked with 10 mg/L dicyandiamide)

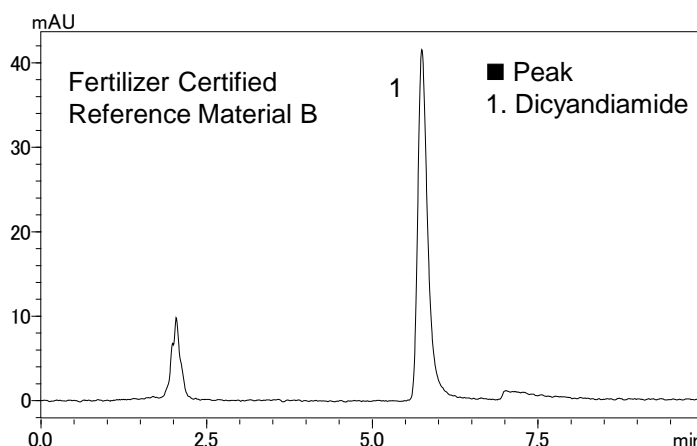


Fig. 5: Chromatogram of Fertilizer Certified Reference Material B (spiked with 10 mg/L dicyandiamide)