Liquid Chromatographic Method for the Determination of Methyl Anthranilate in Liquid Formulation and Residues on Formulated Rice Seed Bait

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Methyl anthranilate was extracted from formulated products and analyzed by reversed-phase high-performance liquid chromatography. Methyl anthranilate was quantified by UV absorbance at 220, 248, and 336 nm. Recovery data were determined by analyzing methyl anthranilate-fortified blank liquid formulation and rice seed. The mean recovery of methyl anthranilate in the microencapsulated formulation was 97.3 ± 2.4% for the range of 7 to 28% methyl anthranilate and 96.1 ± 6.1% for the range of 0.020 to 1.00% methyl anthranilate on rice seed.

Keywords: *Methyl anthranilate; high-performance liquid chromatography; microencapsulated formulation; rice*

INTRODUCTION

Methyl anthranilate (MA) is a methyl ester used as an additive in the food and cosmetic industry. It is commonly used in chewing gum and beverages as grape flavoring and odor and as a fragrance in perfumes.

Methyl anthranilate is also an effective feeding deterrent to many species of birds (Kare, 1961; Mason et al., 1991; Avery et al., 1995b) and rodents (Nolte et al., 1993). Because of its efficacy to inhibit feeding by birds and because MA is generally regarded as safe by the Food and Drug Administration, it is being used as an active ingredient in bird repellents for various bird management uses. Bird depredation to newly planted rice seed is a major problem for producers in the southeastern United States, with annual losses estimated at $4 million/year in Louisiana (Wilson et al., 1989) and $4–5 million/year in Texas (Decker et al., 1990).

To determine the effectiveness of MA as a bird deterrent on rice seed, microencapsulated MA formulation was mixed with an adhesive and applied to rice seed by Grow Tec Ltd. (Nisku, Alberta, Canada). The MA-fortified rice seed field test was completed by the Denver Wildlife Research Center. To support efficacy and field residue studies, analytical methods were developed to confirm the content of MA in the formulation and to determine MA residues on rice seed over the period of the field test.

Several methods exist for the determination of MA, including steam distillation followed by fluorescence analysis (Mattick et al., 1963; Casimir et al., 1976; Moyer and Mattick, 1976; Moyer et al., 1977; Reynolds et al., 1982; Liu and Gallander, 1985; Fischer et al., 1990), spectrophotometry (Ekanayake et al., 1990), gas chromatography (Brunelle et al., 1965; Neudoerffer et al., 1965; Stevens et al., 1965; Stern et al., 1967; Nelson et al., 1976; Nelson et al., 1977; Tomlinson and Boison, 1987), and liquid chromatography with UV (Schmitt et al., 1986; Viñas et al., 1992; Clark et al., 1993) or (Williams and Slavin, 1977; Viñas et al., 1993) fluorescence detection. Because of the large number of samples to be assayed, a method requiring minimum analysis time and a high degree of accuracy and precision was needed. Optimally, the analytical method must extract both the microencapsulated MA suspended in an aqueous solution and the microencapsulated MA bound to rice seed with an adhesive.

MATERIALS AND METHODS

Samples. The samples consist of a proprietary microencapsulated MA formulation (ReJeX-iT AG-36, formulated by PMC Specialties Group Inc., Cincinnatti, OH) and rice seeds coated with a mixture of ReJeX-iT AG-36 and an adhesive (Grow-Tec Ltd., Nisku, Alberta, Canada).

Apparatus. The high-performance liquid chromatography (HPLC) system consisted of a Hewlett-Packard 1050 liquid chromatograph (Palo Alto, CA) operated at ambient temperature. A Hewlett-Packard 1050 UV–vis multiple wavelength detector and/or a Hewlett-Packard 1050 variable wavelength detector were used at wavelengths of 220, 248, and 336 nm to detect MA. Aliquots of 10 μL were injected automatically by the pneumatically controlled injector valve. The MA was separated on a 25 × 0.46-cm i.d. stainless steel analytical column packed with 5-μm Alltech Econosil C-18 (Deerfield, IL). To prolong column lifetime, a 1.5 × 0.46-cm i.d. Keystone Octyl-H (Bellefonte, PA) guard column was used. The samples were chromatographed with a acetonitrile:water (70:30) mobile phase at 1.00 mL/min. The MA peak was identified by comparison with the UV spectra (Figure 1) and retention time of a standard. A Hewlett-Packard 386 Vectra computer work station with an Epson printer was used to collect, process, store, and print the chromatographic data.

Reagents. Methanol and acetonitrile (Fisher Scientific, Denver, CO) were liquid chromatography grade. Deionized water was purified with a Milli-Q water purification system (Millipore, Bedford, MA). The solvents were deaerated by the Hewlett-Packard 1050 series online degasser. Concentrated sulfuric acid (Fisher Scientific, Denver, CO) was diluted with methanol to prepare a 1% (v/v) sulfuric acid solution.

Methyl anthranilate with purities of 99.9% and 98.0% was obtained from Aldrich (Milwaukee, WI) and PMC Specialties (Cincinnati, OH), respectively. Concentrated stock solutions of MA were prepared from the commercial products, without further purification, by dissolving 100 mg in 25 mL of methanol. Working solutions were prepared weekly by dilution with 1% sulfuric acid in methanol. All standard solutions were stored in the dark at 5 °C.

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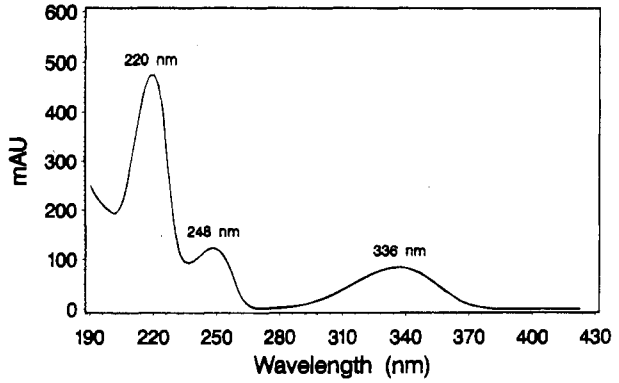


Figure 1. Ultraviolet–visible absorption spectra of MA in acetonitrile/water (70:30).

Table 1. Fortification of Control Samples

A. Liquid Formulation

level (%)	MA (g)	liquid formulation (g)	total (g)	% MA (w/w)
7	0.116	1.450	1.566	7.4
14	0.232	1.350	1.582	14.7
28	0.464	1.150	1.614	28.7

B. Rice Seed

level (%)	stock solution (mg/mL)	aliquot (mL)	rice seed (g)	% MA (w/w)
0.02	4.057	0.0500	1.00	0.0203
0.20	124.6	0.0150	1.00	0.187
0.40	124.6	0.0300	1.00	0.374
1.00	124.6	0.0800	1.00	0.997

Calibration Curve. Seven MA working solutions (1.09–218 μg/mL) were prepared and analyzed by HPLC in duplicate at 220, 248, and 336 nm. A plot was constructed of MA chromatographic peak response (*y*-axis) versus MA concentration (*x*-axis) for each wavelength. Linear regressions were performed on the data.

Determination of MA in Microencapsulated Formulation. The aqueous suspension of microencapsulated MA samples were analyzed in triplicate. Aliquots (1.50–1.60 g) of the MA formulation were accurately weighed into graduated 15-mL screw-capped centrifuge tubes. The samples were diluted to 15.0 mL by the addition of 1% sulfuric acid in methanol. The sample tubes were vortexed, manually shaken for 10 s, and sonicated for 15 min. This step was repeated twice more to ensure the complete rupturing of microcapsules. The sample tubes were centrifuged for 5 min at ≈2500 rpm to separate the encapsulating agent from the MA extract. Aliquots (50 μL) of the MA-containing supernatant were diluted to 10.0 mL with methanol and analyzed by HPLC. The concentration of MA was quantified from the calibration curve.

Fortified control samples were prepared at 7, 14, and 21% MA. To determine recovery efficiencies, MA-fortified control formulation samples were prepared by mixing weighed portions of technical grade MA and blank liquid formulation in the proportions specified in Table 1. The fortified samples were analyzed with the same procedure as just described.

Determination of MA Residue on Fortified Rice Seed. For avian repellent efficacy studies, the MA-fortified rice seed was applied to four flooded test fields located in southwestern Louisiana by aerial seeding. To simplify sampling of fortified and control rice seed for residue analysis, ≈50 g of MA-fortified seed was placed in small sacks of porous cloth and placed randomly in the flooded rice fields. To evaluate the effects of sunlight and hydration/hydrolysis on the stability of MA in the fortified rice seed, fortified rice seed was placed in dry petri dishes located adjacent to one of the fields. The control field with rice seed containing no MA was adjacent to the test fields with at least a 45 m buffer separating the treated and control fields. The 50-g sacks of rice seed and rice seed from the petri dishes were collected for residue analyses from the treated and control fields on posttreatment days 0, 3, 6, and 9. The rice fields were drained after day 3. The rice seed samples were placed in plastic bags, sealed, and refrigerated at 4 °C within 1 h of collection. The samples were shipped overnight in coolers with icepacks at the end of the field study. Upon receipt, the samples were stored in a refrigerator at 4 °C until analyzed. Whenever possible, samples were assayed in triplicate. Subsamples of ≈1.0 g of rice seed were weighed into 25-mL glass screw-capped test tubes. A 10.0-mL aliquot of 1% sulfuric acid in methanol was added to each sample, and the tubes were capped. The samples were vortexed, shaken, and sonicated with the same procedure as described for the microencapsulated formulation. The sample tubes were centrifuged for 5 min at ≈2500 rpm to separate the encapsulating agent and rice seed from the MA extract. Aliquots of 100 μL were diluted to 10.0 mL, and MA concentrations were quantified by HPLC.

To determine recovery efficiencies, MA-fortified control rice seed samples were prepared by fortifying 1.0 g of adhesive-coated control rice seed with an aliquot of a concentrated MA stock solution in methanol, as specified in Table 1. Fortified control samples were prepared at 1.0, 0.40, 0.20, and 0.020% MA. The fortified samples were analyzed with the same procedure as previously described.

Storage Stability of MA-Fortified Rice Seed. From the time of collection to the time of analysis, the samples were stored in sealed plastic bags in the dark at 4 °C. To confirm that these conditions were suitable for storage with negligible loss of MA, a storage stability experiment was performed. Control rice was fortified at 0.020 and 1.00% MA and stored under the conditions stated above. The fortified samples were analyzed after 0, 15, 30, and 45 days.

RESULTS AND DISCUSSION

Instrument Limits of Detection. The UV spectra of MA in acetonitrile:water (70:30) is shown in Figure 1. Three major absorbance maxima were observed at 220, 248, and 336 nm. Each of these wavelengths were acceptable for analysis of MA by HPLC with a UV/vis detector. Using the criterion of three times the peak-to-peak noise of the baseline, the instrument limit of detection for the multiple wavelength detector for all three analytical wavelengths was estimated. At 220, 248, and 336 nm, the instrument limits of detection were 0.05, 0.15, and 0.35 μg/mL, respectively. Of these three wavelengths, MA showed the greatest absorbance at 220 nm. However, for some samples, coeluting compounds interfered with the MA analysis at 220 nm. At 336 nm, no interferences were observed, but there was inadequate sensitivity. Thus, the 248 nm wavelength was selected as a good compromise with respect to sensitivity and chromatographic interferences.

Improved detection limits of 0.006, 0.018, and 0.035 μg/mL were obtained with a variable wavelength detector set at 220, 248, and 336 nm, respectively. Variable wavelength detectors often produce lower detection limits compared with multiple wavelength detectors because the associated noise is typically lower when a single wavelength is being monitored rather than several. A more sensitive detection limit of 0.002 μg/mL was obtained with a fluorescence detector (Primus et al., 1995), which is superior to the previously reported HPLC/fluorescence detection limit of 0.03 μg/mL (10 μL injection; Williams and Slavin, 1977). For this study, the sensitivity afforded by the multiple wavelength detector at 248 nm was sufficient. However, for field residue studies of a longer duration, the sensitivity required for the associated lower residue levels would require a variable wavelength or fluorescence detector.

Linear Regression Data. The regression statistics for the analysis wavelengths are shown in Table 2. The

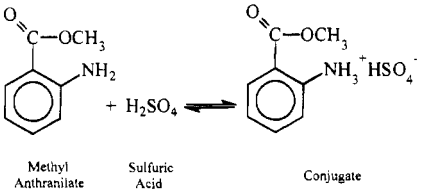
Table 2. Linear Regression Data for Calibration Curves (1.09–218 μg/mL)

MA Concn (μg/mL) versus Peak Area					
λ (nm)	slope	y-intercept	r^2	mean RF[a]	CV (%)
220	106.2	30.1	0.9999	0.00934	0.82
248	27.71	−0.96	1.0000	0.0366	3.4
336	18.78	−1.19	1.0000	0.0543	5.2

Log (MA Concn) versus Log (Peak Area)		
λ (nm)	r^2	slope
220	1.0000	0.9994
248	0.9998	1.008
336	0.9995	1.010

[a] Response factor, concentration of standard solution injected into liquid chromatogram divided by the chromatographic response.

Scheme 1. Formation of a Stable Quaternary Amine Conjugate with Sulfuric Acid



correlation coefficients of peak response versus concentration were all ≥0.9999 and the log–log data demonstrates that peak response was proportional to concentration over the range of the calibration curves for each wavelength.

Methyl Anthranilate Stability. Methyl anthranilate is easily lost and subject to degradation because it is volatile, photoreactive, and susceptible to microbial degradation (Clark et al., 1993). Methyl anthranilate is soluble in water, but the solubility is greater in methanol and biodegradation problems can be eliminated with the use of methanol. The addition of a small amount of sulfuric acid to the extraction solvent favors the formation of a stable quaternary amine conjugate (Scheme 1). All samples were extracted with this solution during the sample preparation procedure.

Sample Extraction. Simple shaking of the microencapsulated liquid formulation and the fortified rice seed with the extraction solvent produced inadequate extraction. A more efficient process was needed to rupture the microcapsules and assure complete extraction of MA. To improve the efficiency of the extraction, the samples were sonicated for 15 min. This step was needed twice for most samples, but a third sonication was added because of occasional agglomeration problems with the liquid formulation. All rice seed samples required three sonication for the extraction solution to penetrate the adhesive used to bind the microcapsules to the rice seed.

During method development, several samples were ground, extracted, and analyzed for MA. The MA concentration of the ground samples was compared with unground samples to access the potential difference in extraction efficiency of the method between whole and ground rice seed. The mean quantities of MA in the unground and ground samples were 0.938 ± 0.42 and 0.969 ± 0.031, respectively. A t test performed on the mean MA concentration of unground and ground samples showed no difference ($p = 0.37$) between the mean MA concentrations. Therefore, the grinding step was not incorporated in the MA residue on formulated rice seed method.

Table 3. Validation Results for the Determination of MA in the Microencapsualted Formulation

	fortification levels and % recovery		
sample	7%	14%	21%
1	101	95.8	94.3
2	100	98.2	94.5
3	97.4	97.0	98.0
4	99.1	96.2	98.7
5	100	95.0	98.5
6	94.8	94.4	99.8
7	101	94.8	94.1
mean	99.0	95.9	96.8
SD	2.2	1.3	2.4
CV	2.2	1.4	2.5

Table 4. Validation Results for the Determination of MA Residues in Fortified Rice Seed

	fortification levels and % recovery			
sample	0.020%	0.20%	0.40%	1.0%
1	84.1	91.8	95.6	94.0
2	104	90.2	89.5	93.3
3	109	96.5	99.7	91.4
4	92.3	97.5	100	104
5	97.0	103	97.5	93.2
6	80.9	101	99.6	93.0
7	103	96.5	99.6	93.0
mean	95.8	96.6	97.4	94.6
SD	10.5	4.6	3.8	4.2
CV	11.0	4.8	3.9	4.4

Microencapsulated Liquid Formulation Method. The method was validated from 7 to 28% MA, to cover the range of formulation concentrations (8–23% MA). The levels validated were 7, 14, and 28% MA. The recoveries of MA from fortified control formulation samples obtained by the HPLC method are given in Table 3. Mean recoveries ranged from 95.9 to 99.0%, with coefficients of variation (CV) of ≤2.5%.

Residue on Rice Seed. The recoveries of MA from fortified control rice seed samples obtained by the HPLC method are listed in Table 4. The mean recoveries range from 94.6 to 97.4% MA with a CV of ≤11.0%. With the multiple wavelength detector at 248 nm, 0.02% fortified control samples were used to determine the method limit of detection of 1.2 μg/g. The initial mean quantity of MA residue on the rice seed determined prior to application was 0.806 ± 0.019%.

The residue results from the field test are shown in Figure 3. By inspection, the MA residue data from the four field and open air samples seem very similar over the study period. The 9-day field test data were analyzed with SAS PROC GLM (SAS Institute, Cary, NC). Comparing the 9-day field test data by an analysis of variance (ANOVA), there was no significant day by field interaction ($p = 0.12$), indicating similar MA residue degradation for the open air and field samples over time. The mean MA residues on rice seed decreased significantly each sampling day for all test sites. This result would indicate that leaching, hydrolysis, and/or UV degradation of microencapsulated MA were not significant, as the rice seed in the petri dishes (open air samples) were dry and the field samples were submerged for 3 days. The rate of degradation was more rapid over the first 3 days, with ≈80% being lost. In contrast, over the last 6 days, the rate of loss was slowed. This result suggests that more than one mechanism is responsible for the degradation of MA. Future experiments investigating the effects of temperature, humidity, and microbial activity on MA stability are

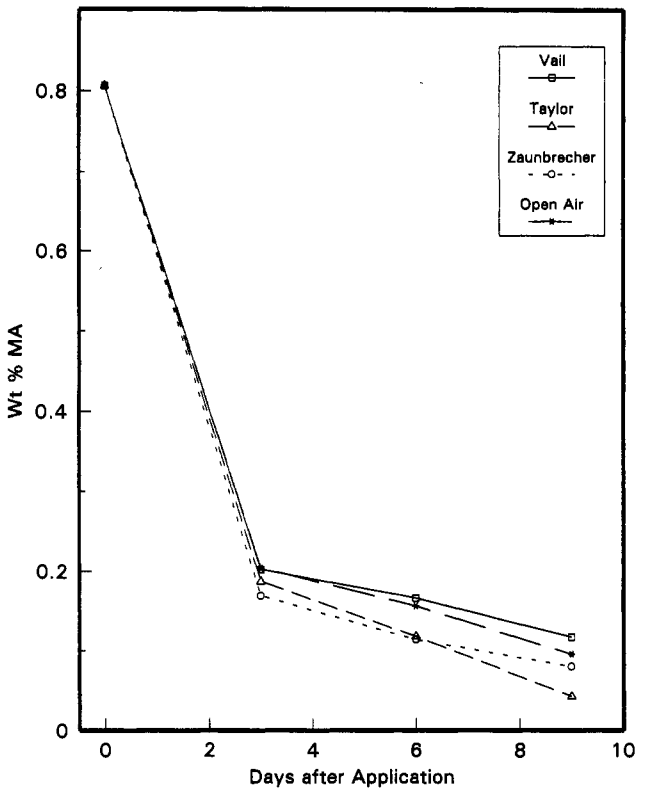


Figure 2. MA residue on fortified rice seed from four test sites over a 9-day period.

needed to understand the mechanisms contributing to this degradation pattern.

Storage Stability. The sample storage stability results showed a 4% loss of MA from the 1.0% fortified samples and a 5% loss of MA from the 0.020% fortified samples after 30 days. The rice seed samples were analyzed 10–31 days after collection. The minor loss of MA from the storage stability experiment was considered negligible, and the residue samples were not corrected for the loss. Data from other storage stability experiments show freezing of MA formulation and MA fortified blueberries have <1% loss of analyte after 70 days at less than −10 °C (Avery et al., 1995a).

Conclusion. This method for the determination of MA in both the microencapsulated formulation and the fortified rice seed was simple and efficient. The mean recoveries for the determination of MA in the microencapsulated formulation were 97.3 ± 2.4% for the range of 7 to 28% MA and 96.1 ± 6.1% for the range of 0.020 to 1.00% MA on rice seed. This methodology can be utilized for additional studies to determine factors contributing to MA instability, leading to the improved effectiveness of MA as an avian repellent.

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