# Determination of Daminozide Residues in Apple Pulp Using HPLC−DAD-UV

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This paper reports an HPLC−UV method to determine daminozide residues in apple pulps adopting the recently introduced EU limit of 0.01 mg/kg for baby food preparation (Commission Directive 1999/39/CE). The method is based on alkaline hydrolysis of daminozide to *N′*,*N′*-dimethylhydrazine (UDMH), which is recovered by distillation and subsequently derivatizated with salicyl aldehyde to salicyl aldehyde-*N*,*N*-dimethylhydrazone under strongly basic conditions. The resulting solution was cleaned up with Extrelut 20 NT and dichloromethane as eluent, then analyzed by HPLC with a C18 column and a mobile phase programmed from 50:50 AcCN/$H_2O$ to 100% AcCN. The salicyl aldehyde-*N*,*N*-dimethylhydrazone was selectively detected through two diagnostic UV absorption maxima at 295 and 325 nm, which have strong molar absorbivities. Recoveries of daminozide at 0.01 mg/kg were above 80%. The limits of detection (LODs) of salicyl aldehyde-*N*,*N*-dimethylhydrazone expressed as daminozide concentration were 100 pg/$\mu$L at 295 nm and 150 pg/$\mu$L at 325 nm, and the limits of quantitation (LOQs) of daminozide were 0.0013 mg/kg at 295 nm and 0.0022 mg/kg at 325 nm.

**Keywords:** *Daminozide; N′,N′-dimethylhydrazine (UDMH); salicyl aldehyde-N,N-dimethyl hydrazone; HPLC−UV; apple pulps; 0.01 mg/kg*

## INTRODUCTION

Daminozide (Alar, *N*-dimethylaminosuccinamic acid) **(1)** is a plant growth regulator used mainly with apples (although registered for many other crops) to control flower development, to reduce premature fruit fall, and to improve color development, size, and storage capabilities. Daminozide has been found to be a possible carcinogen in laboratory animals (*1*) and is known to decompose to *N′*,*N′*-dimethylhydrazine (unsymmetrical-dimethylhydrazine, UDMH) **(2)** in apple homogenates submitted to heat treatments (*2*); UDMH residues, too, have been recognized as potential carcinogens when present in foods (*3*, *4*). There is particular concern over potential UDMH residues in apple-derived baby foods because the intake/body weight ratio in children is greater than in adults.

Several methods are available to determine daminozide residues in apples and related semi-worked products. The early colorimetric methods are not compatible with the residue limits required today (below 0.05 ppm), therefore daminozide residues are now generally determined either by GC with different detection systems or by LC in combination with MS. As daminozide cannot be directly analyzed because of it is high polarity and thermolability, GC methods generally involve daminozide hydrolysis to UDMH with strong alkali, UDMH recovery by distillation, its derivatization to a derivative suitable for a selective detector (nitrogen−phosphorus detector (NPD), electron capture detector (ECD), or mass spectrometry (MS)), and then analysis by GC. Newsome (*2*) used pentafluorobenzoyl chloride as derivatizing agent and GC/ECD, achieving a limit of detection (LOD) of 0.01 ppm (although with a very high relative standard deviation % (RSD%)), and a limit of quantitation (LOQ) of 0.1 ppm; Rutschmann and Buser (*5*) investigated Swiss apple juice concentrates using the same reagent but using GC/MS. Allen (*6*) used salicyl aldehyde (SA) and GC/NPD in sweet cherries; Conditt and Baumgardner (*7*) used the same reagent but GC/MS to analyze apple juices and sauces and peanut butter with sensitivity in a range between 0.1 and 0.5 ppm, although an LOD of 0.01 ppm was obtained but with an RSD of 82%. Wright (*8*) derivatized UDMH with 2-nitrobenzaldehyde and GC/ECD for apples and peaches with a sensitivity of 0.1 ppm; this method was also used by Saxton et al. (*9*) to analyze apples, their juices and sauce, cherries, canned sour cherries, and grapes. Steinbrecher et al. (*10*) modified and improved Wright's method, reaching an LOD of 0.05 ppm. Brinkman et al. (*11*) analyzed 2-nitrobenzaldehyde-*N*,*N*-dimethylhydrazone by GC/NPD, and Majumdar et al. (*12*) used GC/MS; the latter achieving the lowest LOD for daminozide residues in apples (0.4 ppb). Liu et al. (*13*) determined daminozide residues in apples after extraction and esterification to its methyl ester by GC/CIMS.

To the best of the authors' knowledge, HPLC has been less frequently applied to analyze daminozide residues. Kim et al. (*14*) used anion exchange chromatography combined with particle beam-positive-CIMS to analyze intact daminozide in apple juice, with a detection limit of 0.025 ppm. Very recently, Mol et al. (*15*) analyzed intact daminozide in apples and leaves by LC/APCI−MS−MS with an LOD of 0.08 ppm. The lack of chromophoric groups makes the direct determination of daminozide by HPLC−UV very difficult, if not impos-

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10.1021/jf0103673 CCC: $20.00 
Published on Web 07/17/2001

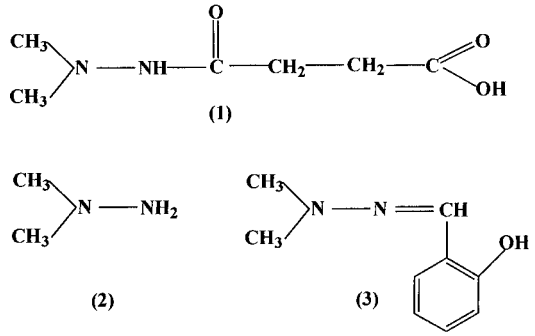


**Figure 1.** Structures of the compounds involved in this study. **1** daminozide; **2** *N′N′*-dimethylhydrazine, UDMH; **3** salicyl aldehyde-*N*,*N*-dimethylhydrazone.

sible. This drawback can be overcome by derivatizing UDMH with a reagent containing a chromophoric group, thus affording analysis of total daminozide residues by HPLC including those degraded to UDMH by technological treatment.

This paper reports an HPLC−UV method to determine daminozide residues in apple pulps based on alkaline hydrolysis to UDMH, recovery by distillation, and subsequent derivatization with salicyl aldehyde to SA-*N*,*N*-dimethylhydrazone **(3)** derivative (Figure 1). This method has been developed within a project aiming to determine pesticide residues in fruits (*16*−*19*), vegetables, and their semi-finished products adopting the recently introduced EU limit of 0.01 mg/kg for baby food preparation (Commission Directive 1999/39/CE).

## MATERIALS AND METHODS

**Materials and Reagents.** *Apple Pulp Samples.* Apple pulp samples were supplied by Allione SpA (Tarantasca − Cuneo, Italy)). Control apple pulp was prepared from untreated plots of the same apple cultivars. Six different lots of commercially available apple pulp samples of different origins were also analyzed.

*Solvents and Chemicals*. Acetonitrile and dichloromethane were HPLC grade or gradient grade from Riedel-de Haen (Seelze, Germany). Analytical grade sodium hydroxide and hydrochloric acid were from Sigma Aldrich (Milano, Italy) and Extrelut 20 NT was from Merck (Darmstadt, Germany). 99% Salicyl aldehyde (SA) was from Riedel-de Haen (Milan, Italy).

*Standards*. Pure standard sample of daminozide was from Dr. Ehrenstorfer GmbH (Augsburg, Germany), and 98% UDMH was from Merck (Darmstadt, Germany).

**Hazard Warnings.** (1) The distillation system must be perfectly tight to prevent leakage of carcinogenic UDMH vapors. (2) Some samples may foam. The foam is strongly caustic because of the high amount of sodium hydroxide. Foam can be avoided by adding 5 g of paraffin wax to the distillation flask.

**Sample Preparation.** *Hydrolysis of Daminozide and Recovery of UDMH*. The method reported is based on that described by Conditt and Baumgartner (*7*) and modified in the authors' laboratory. Sodium hydroxide solution (50%; 160 mL) was carefully added to 50 g of apple pulp in a 1-L distillation flask under constant stirring, and a further 30 g of sodium hydroxide pellets was added to the sample to ensure complete daminozide hydrolysis. The distillation flask was then connected with the distillation apparatus, as was the 10-mL receiving flask containing 20 $\mu$L of SA. Distillation apparatus and conditions were those described by Conditt and Baumgartner (*7*), but avoiding the treatments with $CaCl_2$, $TiCl_3$, and paraffin, since they have been shown to have no influence on the distillation process (*11*). The sample was digested for 30 min at room temperature under rapid stirring to hydrolyze daminozide, and then the resulting UDMH (bp 62 °C) was rapidly distilled. A 10-mL portion of aqueous distillate of UDMH was collected in 15−30 min.

*UDMH Derivatization*. The receiving flask with 10 mL of distillate was then transferred to a thermostatic bath at 50 °C for 25 min to form SA-dimethylhydrazone. Sodium hydroxide (50%; 1.5 mL)was added to the distillate to keep it strongly basic so that unreacted SA was transformed to the corresponding alcohol and acid. SA elimination was monitored by GC, under the same conditions adopted for GC/MS analysis (see below). The SA-dimethylhydrazone is stable and may be stored in the dark at −4 °C in a refrigerator.

*Distillate Cleanup*. The resulting solution was quantitatively transferred in a Extrelut 20 NT polypropylene cartridge, and after 10 min was eluted with 50 mL of dichoromethane under atmospheric pressure. The eluate was evaporated to dryness under vacuum, redissolved in 500 $\mu$L of acetonitrile/$H_2O$, and submitted to HPLC analysis.

**HPLC Analysis.** *HPLC Unit and Data System*. The HPLC was a Hewlett Packard HP 1090 unit equipped with a DAD UV detector and an HP Chem Station data system for quali-quantitative elaboration.

*Column*. The column used was a Microsorb-MV 100 Å C18 4.6 mm i.d. × 250 mm × 5 $\mu$m (Varian, Turin, Italy) provided with a guard column: 4 × 4 mm × 5 $\mu$m LiChrospher RP-18 (Agilent Technologies, Milan, Italy).

*Operative Conditions*. The injection volume was 20 $\mu$L. Before injection, the sample was filtered through disposable syringe filters with 0.2 $\mu$m × 13 mm PTFE membranes (Waters, Milford, CT). The oven temperature was 35 °C. Detection wavelengths were 295 and 325 nm. Mobile phase was AcCN/$H_2O$ − AcCN and the flow rate was 1 mL/min. The mobile phase program was from 50:50 AcCN/$H_2O$ to 100% AcCN in 5 min, then hold for 25 min at 100% AcCN. Before re-injection, the HPLC system was stabilized for at least 10 min.

SA-dimethylhydrazone was identified by comparing its retention time and UV spectrum with those from a standard solution of an authentic sample, and its residues were quantified using the calibration curve obtained as reported below.

*SA-Dimethylhydrazone Standard*. A standard of the derivative was prepared : 500 $\mu$L of a 37.5 $\mu$g/mL UDMH solution in HCl 0.1 M corresponding to 10 $\mu$g/mL of daminozide was transferred to a 20-mL flask containing 10 mL of distilled water, 20 $\mu$L of SA, and 1.5 mL of 50% sodium hydroxide. The resulting solution was left to react and cleaned up as reported above. The authenticity of SA-dimethylhydrazone was confirmed by GC−MS. The resulting AcCN solution was used as a standard for quantitative determination.

SA-dimethylhydrazone stability was controlled by storing three standard solutions in a freezer at −4 °C and analyzing them every 12 h.

**Quantitative Analysis.** The standard solution of SA-dimethylhydrazone obtained as reported above was diluted with suitable amounts of AcCN to obtain concentrations corresponding respectively to 0.05, 0.03, 0.02, 0.01, and 0.005 mg/kg of daminozide residues, and these were analyzed by HPLC. A calibration curve was made by analyzing the resulting standard solutions by HPLC−UV at 295 nm; the correlation coefficient was above 0.997.

**GC−MS Analysis.** GC/EI-MS analyses were carried out on a Hewlett-Packard 5988 A GC/MS system provided with a Hewlett-Packard 5890 GC unit. An OV-1701 open tubular column (25 m length, 0.25 mm i.d., 0.3 $\mu$m df) (MEGA, Legnano, Milan, Italy) was used. Chromatographic conditions were injector temperature, 250 °C; temperature, 200 °C isotherm; transfer line temperature, 260 °C; carrier gas, helium, flow rate, 2 mL/min. The injection system was split, with a split ratio of 1:20. Diagnostic ions of SA-dimethylhydrazone were at *m*/*z* 164 (corresponding to the molecular ion), 121, and 120 as reported by Conditt and Baumgardner (*7*). Figure 2 reports mass and UV spectra of SA-*N*,*N*-dimethylhydrazone.

**Recovery.** *Daminozide Recovery*. Aliquots of 250, 150, 100, 50, and 25 $\mu$L of a 10 $\mu$g/mL standard solution corresponding to 0.05, 0.03, 0.02, 0.01, and 0.005 mg/kg of daminozide

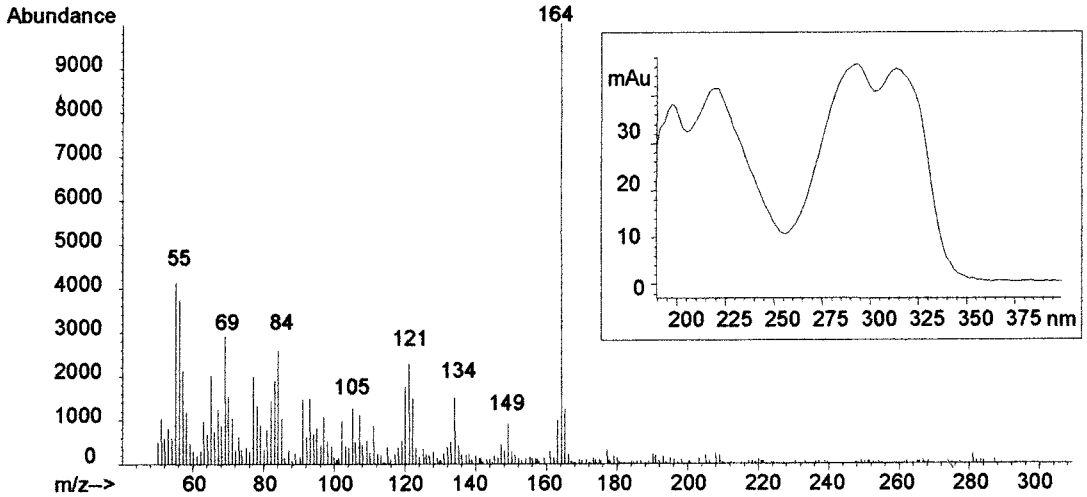


**Figure 2.** Mass and UV spectra of SA-*N*,*N*-dimethylhydrazone.

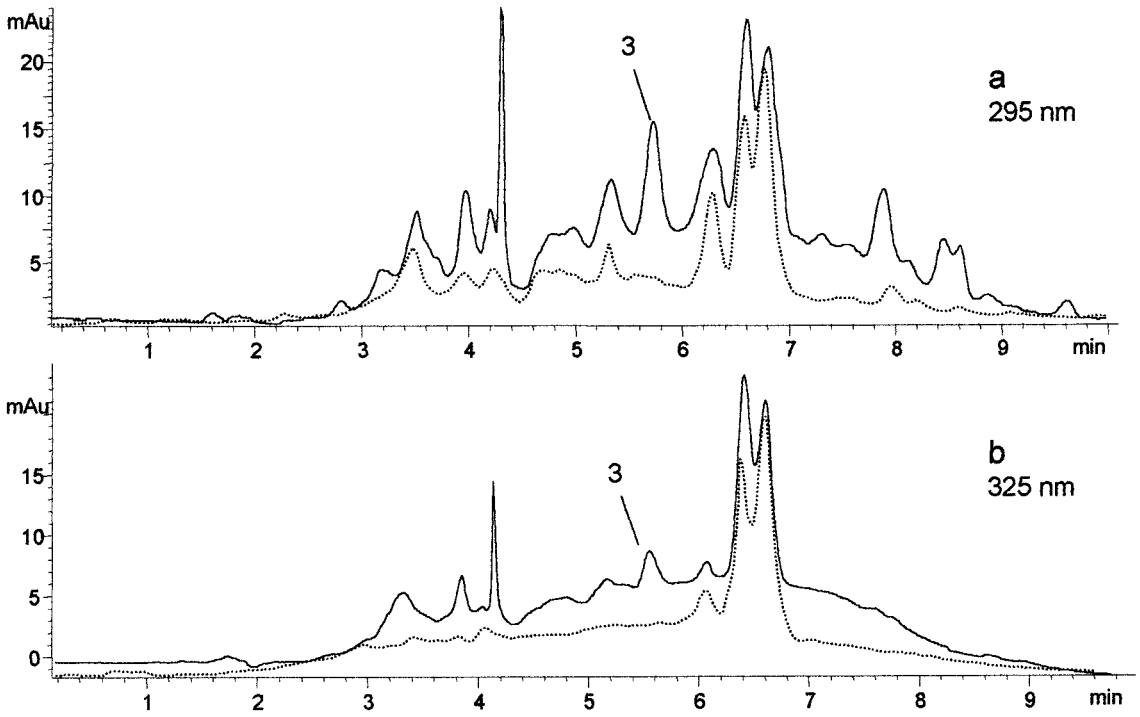


**Figure 3.** HPLC−UV patterns of an apple pulp sample fortified with 0.01 mg/kg of daminozide (—) and of an apple pulp blank (···) carried out at 295 (a) and 325 (b) nm.

residue, respectively, were added to 50 g of pulp prepared with daminozide-untreated apples. The resulting samples were homogenized for 1 min and then submitted to digestion, distillation, derivatization, cleanup, and HPLC−UV analysis as described above. Each recovery test was repeated six times. Recovery of daminozide calculated as SA-dimethylhydrazone was determined through the calibration curve obtained as reported above.

*UDMH Recovery.* UDMH distillation recovery was evaluated by spiking a daminozide-free apple pulp sample with known amounts of UDMH corresponding to 0.1 and 0.01 mg/kg of daminozide residue and submitting it to distillation, derivatization, cleanup, and HPLC−UV analysis as described above.

**LOD and LOQ Determination.** LOD was measured by analyzing a standard solution of SA-dimethylhydrazone under the HPLC conditions reported above in amounts sufficient to obtain a signal-to-noise ratio of three to one. LOQs were determined through the IUPAC method (*20*), and calculated by analyzing untreated apple-pulp samples submitted to digestion, distillation, derivatization, and cleanup, six times.

## RESULTS AND DISCUSSION

UDMH residues can be determined by HPLC−UV only after being derivatized with a reagent containing a chromophoric group, making UV-adsorbing the resulting derivative. Salicyl aldehyde was chosen as the derivatizing reagent not only because the corresponding SA-dimethylhydrazone shows a characteristic UV spectrum with two diagnostic and specific absorption maxima at 295 and 325 nm, but also because of their strong molar absorbivities ($\epsilon$), affording the very low detection limits required. The UV spectrum of SA-dimethylhydrazone is reported in Figure 2. Moreover, the combination of two diagnostic UV absorptions of similar intensity with peak purity control are useful to detect possible matrix interference when pulps prepared with apples of different origins are analyzed. The formation of SA-dimethylhydrazone was studied in depth through a series of experiments with different UDMH amounts

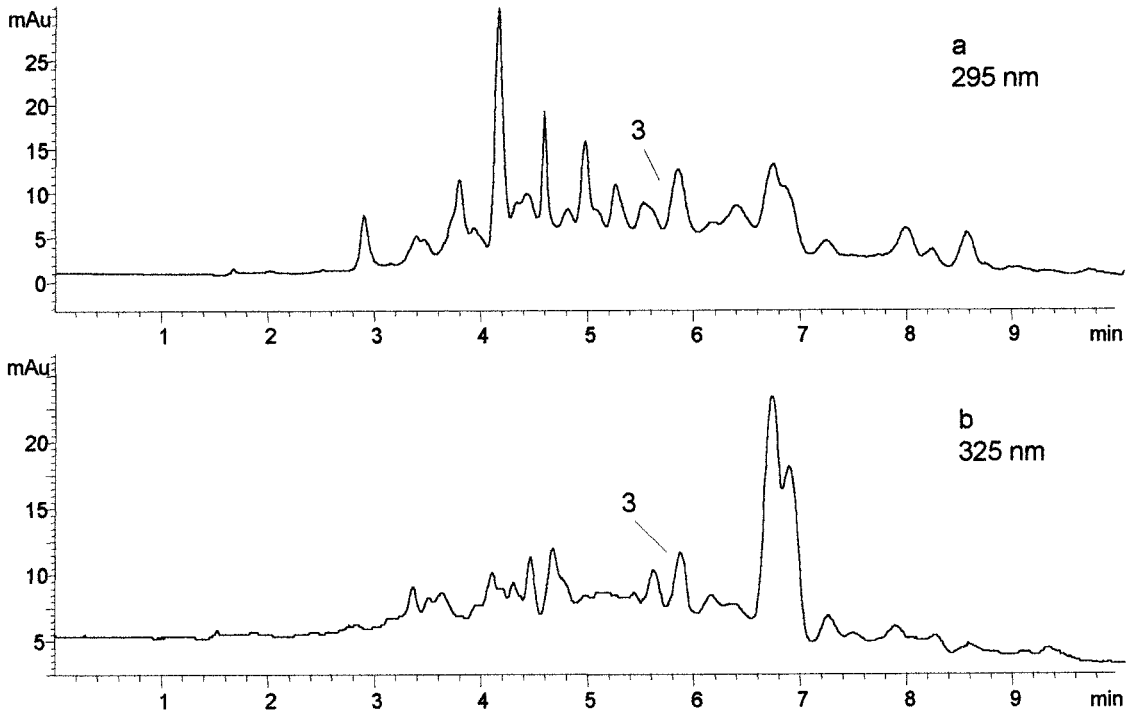


**Figure 4.** HPLC−UV patterns of a commercially available apple pulp sample containing 0.015 mg/kg of daminozide residues, detected at 295 (a) and 325 (b) nm.

(1−1000 ng) and under different reaction conditions. These experiments indicated that SA-dimethylhydrazone was quantitatively formed in 15 min. Moreover, SA-dimethylhydrazone is stable over time (decomposition below 5% over 24 h) making correct quantitative determination time-independent, provided that it is stored in the dark at −4 °C. SA-dimethylhydrazone stability was confirmed by repeated analyses on standard samples over time whose results are in agreement with those reported by Wright (*8*), Conditt and Baumgardner (*7*), and Brinkmann et al (*11*). SA-dimethylhydrazone is also stable in strongly basic solution, making it possible to eliminate the SA excess at very high pH values without affecting the SA-dimethylhydrazone quantitative determination. Last but not least, SA is nontoxic (Safety data sheet, Riedl-de Haen, St. Louis, MO; product n*o*. 60270).

Figure 3 reports HPLC−UV patterns of an apple pulp sample fortified with 0.01 mg/kg of daminozide and of an apple pulp blank carried out at (a) 295 nm and (b) 325 nm. All samples were submitted to the cleanup procedure described above.

**Sample Preparation and Recovery.** In this article, quantitative data are expressed as daminozide, even when UDMH or SA-dimethylhydrazone are involved, and have been obtained through the calibration curve at 295 nm made as described above. The correlation coefficient for that curve in question was 0.997. Sample preparation procedures were strongly conditioned by the low limit fixed for daminozide residues (0.01 mg/kg). At this limit even trace matrix components interfered with SA-dimethylhydrazone (and, as a consequence, with daminozide) residue quantitation; this made necessary a cleanup step with Extrelut NT 20 to eliminate matrix interference. UDMH distillation recovery was quantitative (better than 95% for all samples) although relatively higher variations were found for the highest fortification levels, as already reported by Newsome (*2*). These data are in agreement with those reported by Majumdar et al. (*12*).

**Table 1. Recovery %, Standard Deviation, Relative Standard Deviation %, and Reproducibility from an Apple Pulp Spiked with Different Amounts of Daminozide (determined as SA-dimethylhydrazone)**

| daminozide recovery | | | | |
|---|---|---|---|---|
| spiked amount mg/kg | *n* | recovery % | SD. | RSD% |
| 0.05 | 6 | 83.3 | 10.1 | 5.1 |
| 0.04 | 6 | 79.4 | 4.9 | 3.4 |
| 0.03 | 6 | 90.1 | 8.2 | 7.8 |
| 0.02 | 6 | 82.9 | 4.4 | 6.6 |
| 0.01 | 6 | 84.2 | 3.5 | 3.2 |
| 0.005 | 6 | 71.2 | 7.8 | 5.5 |
| | | reproducibility | | |
| 0.1 | 6 | 86.6 | 7.8 | 6.7 |
| 0.5 | 6 | 83.4 | 5.9 | 5.6 |

With the sample preparation procedure reported, recoveries of daminozide at 0.01 mg/kg were above 80%. Repeatability of the method was determined through recovery experiments at different fortification levels (0.05, 0.04, 0.03, 0.02, 0.01, and 0.005 mg/kg). Six recovery experiments were run for each fortification level, and each solution resulting from the cleanup was analyzed three times. Recovery (%), standard deviation (SD), and relative standard deviation % (RSD%) were determined. These results are reported in Table 1.

Reproducibility was evaluated by controlling recoveries of daminozide (as SA-dimethylhydrazone) at different fortification levels (0.5 and 0.1 mg/kg) every two weeks over a three month period, for a total of six determinations. Results are reported in Table 1. By combining all the recovery results at each fortification level, RSD% values were within the chosen range in all cases (up to 10%). GC/MS was used as a confirmation procedure for the present method. This procedure is in agreement with the *Guidance Document on Residue*

*Analytical Methods* (8064/VI/97-rev 4) by the European Commission.

The limits of detection (LODs) were determined by analyzing SA-dimethylhydrazone standard solutions of increasing dilutions up to a signal-to-noise (S/N) ratio of 3. LODs expressed as daminozide concentration were 100 pg/$\mu$L at 295 nm and 150 pg/$\mu$L at 325 nm. The limits of quantitation (LOQs) for routine analysis of daminozide were 0.0013 mg/kg at 295 nm and 0.0022 mg/kg at 325 nm.

Although the use of daminozide on apples has probably been discontinued all over the world in recent years as a consequence of the 1989 outcry, its determination on semifinished products is still required, in particular nowadays, as industry tends to extend semi-finished production over time, using crops from different countries and suppliers. Results of analyses on six real-world samples found daminozide residues in only two lots investigated: at the 0.02 and 0.015 mg/kg levels. Figure 4 reports the HPLC−UV patterns of a commercially available apple pulp sample containing 0.015 mg/kg of SA-dimethylhydrazone residues, detected at 295 and 325 nm.

## CONCLUSION

The method described here enables daminozide and UDMH residues to be determined as SA-dimethylhydrazone by HPLC−UV in apple pulp. The method is specific and selective because of diagnostic and specific absorption maxima in the UV spectrum (295 and 325 nm) of the UDMH derivative; sensitive because it affords the very low detection limits required by the EU limits for baby food preparation (0.01 mg/kg) partly because of the strong UV molar absorbivities of the SA-dimethylhydrazone; and reliable because it gives a high recovery with a very good repeatability and reproducibilibty. The method makes it possible to apply HPLC to daminozide residue determination also in those laboratories where LC−MS facilities are not available.

## ACKNOWLEDGMENT

The authors are indebted to Allione − Industria Alimentare SpA − Tarantasca (Cuneo, Italy) for the financial support to the laboratory. C. Cordero is grateful to the University of Turin for the financial support to the project "*Sviluppo di tecnologie veloci per l'analisi di residui di fitofarmaci in derrate e semilavorati alimentari*".

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**Registry No.** as supplied by author: UDMH, 56-14-7; daminozide, 1596-84-5; salicyl aldehyde, 90-02-8; salicyl aldehyde-*N*,*N*-dimethylhydrazone, 13405-65-7.

Received for review March 19, 2001. Revised manuscript received May 23, 2001. Accepted May 24, 2001.

JF0103673