Food and Agriculture Organization
of the United Nations

AGP:CP/336

FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

ACEPHATE (AGP:CP/336)

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

Rome, 1996

TABLE OF CONTENTS

DISCLAIMER [1]

FAO specifications are developed with the basic objective of promoting, as far as practicable, the manufacture, distribution and use of pesticides that meet basic quality requirements.

Compliance with the specifications does not constitute an endorsement or warranty of the fitness of a particular pesticide for a particular purpose, including its suitability for the control of any given pest, or its suitability for use in a particular area. Owing to the complexity of the problems involved, the suitability of pesticides for a particular purpose and the content of the labelling instructions must be decided at the national or provincial level.

Furthermore, pesticides which are manufactured to comply with these specifications are not exempted from any safety regulation or other legal or administrative provision applicable to their manufacture, sale, transportation, storage, handling, preparation and/or use.

FAO disclaims any and all liability for any injury, death, loss, damage or other prejudice of any kind that may arise as a result of, or in connection with, the manufacture, sale, transportation, storage, handling, preparation and/or use of pesticides which are found, or are claimed, to have been manufactured to comply with these specifications.

Additionally, FAO wishes to alert users to the fact that improper storage, handling, preparation and/or use of pesticides can result in either a lowering or complete loss of safety and/or efficacy.

FAO is not responsible, and does not accept any liability, for the testing of pesticides for compliance with the specifications, nor for any methods recommended and/or used for testing compliance. As a result, FAO does not in any way warrant or represent that any pesticide claimed to comply with a FAO specification actually does so.

[1] This disclaimer applies to all specifications published by FAO.

INTRODUCTION TO FAO SPECIFICATIONS DEVELOPED UNDER THE OLD PROCEDURE

Between 1975 and 2000, FAO published booklets of specifications for technical materials and related formulations of plant protection products. Revisions of, and additions to, already published specifications will be issued when necessary. However, all changes and revisions of FAO specifications are now subject to the new procedure described in the *Manual on the development and use of FAO and WHO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 173, Rome 2002 (*Revised First Edition* available only on the FAO home page of the Internet at: http://www.fao.org/pest-and-pesticide-management/en/)

FAO specifications developed under the old procedure are based on the requirements defined in the Fourth Edition of the *Manual on the development and use of FAO specifications for plant protection products*, Plant Production and Protection Paper No. 128, Rome 1995.

This manual contained detailed definitions and other essential background information on basic procedures and technical principles adopted by the group on Pesticide Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent, such as:

1. Categories of Specifications (Section 3.1 of the Manual)

   FAO Tentative Specifications (Code 'S/T', formerly 'TS') are those which have been recommended by FAO as preliminary specifications and which are based on minimum requirements. The methods of analysis cited are normally supplied by the manufacturer or may already have been published or be the subject of collaborative work.

   FAO Provisional Specifications [Code 'S/P', formerly ('S')] are those for which more evidence of the necessary parameters is available and where some collaborative study of the methods of analysis has been carried out.

   FAO (full) Specifications (Code 'S/F', formerly 'S').
   Specifications that have all necessary requirements together with CIPAC (full) methods, or other collaboratively studied (proven) methods.[2, 3]

   Wherever possible, standards for apparatus and common names for pesticides are those approved by the International Organization for Standardization (ISO).

2. Expression of active ingredient content (Section 4.2.5 of the Manual)

   - for solids, liquid technical materials, volatile liquids (of maximum boiling point 50°C) and viscous liquids (with minimum kinematic viscosity of 1×10^3 m^2/s at 20°C) the FAO Specification shall be based on expression of the content as g/kg;

- for all other liquids the active ingredient content of the product shall be declared in terms of g/kg *or* g/l at 20°C. If the customer requires both g/kg *and* g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.

3. Tolerance on content (Section 4.2.7 of the Manual)

A declared content of active ingredient must be included in all specifications, and one of the problems immediately arising is the level of tolerance acceptable about the nominal figure. The tolerance is influenced by (a) the reproducibility of the method of analysis, (b) the sampling error and (c) the manufacturing variance.

Allowable variations in analytical results (i.e. tolerances in content of active ingredient) with respect to specific pesticide consignments are intended to cover reasonable variations in the contents of active ingredients. For examples of such tolerances, see the table in Section 4.2.7 of the Manual.

4. Containers/packaging

FAO guidelines are in preparation.

Containers shall comply with pertinent national and international transport and safety regulations.

Technical materials, dustable powders and granules

Containers shall be suitable, clean, dry and as specified, and shall not adversely affect, or be affected by, the contents, but shall adequately protect them against external conditions.

Wettable powders

The product shall be packed in suitable, clean, dry containers as specified in the order. The container shall provide all necessary protection against compaction, atmospheric moisture, loss by vaporization and/or contamination to ensure that the product suffers no deterioration under normal transit and storage conditions.

The product shall be protected by an adequate moisture barrier. This may be a suitable bag of polyethylene or alternative means of giving equal or better protection.

Solutions and emulsifiable concentrates

Containers shall be lined, where necessary, with a suitable material, or the interior surfaces shall be treated to prevent corrosion and/or deterioration of the contents.

Additional information should be given in all specifications where particular pesticides present problems in packaging.

5. Biological information

Phytotoxicity

No test can be specified to cover the possible phytotoxicity of a formulation to all crops. When a crop is not mentioned in the instructions for use, purchasers should check with the supplier that the material is suitable, always provided that such a use is not restricted or legally forbidden.

Wetting of crops

The dilute spray should satisfactorily wet the leaves of the specified crops when used in accordance with the instructions. Test method MT 53.2, CIPAC F, p.162, may be useful.

---

[1] *Should national pesticide specifications developed from these approved FAO specifications deviate from them, the National Authority responsible for making such changes is requested to inform the FAO Plant Protection Service of the nature of, and the reasons for, the modifications.*

[2] *Methods of analysis and miscellaneous techniques referred to in these specifications have been developed and adopted by CIPAC (Collaborative International Pesticides Analytical Council Ltd.). See CIPAC Handbooks 1 (1970), 1A (1980), 1B (1983), 1C (1985), D (1988), E (1993), F (1995), G (1995), CIPAC Proceedings 1980 and 1981, obtainable from Black Bear Press Limited, King's Hedges Road, Cambridge CB4 2PQ, England. The page numbers of specific methods are given in parentheses in the specifications. Copies of methods not yet published can be obtained from the FAO Plant Protection Service.*

[3] *Information on standard waters for laboratory evaluation of pesticidal formulations will be found in CIPAC Monograph 1*, Standard Waters and an FAO Survey on Naturally Occurring Waters *(1972), Black Bear Press Limited, King's Hedges Road, Cambridge CB4 2PQ, England.*

SUBMISSION OF DRAFT SPECIFICATIONS TO FAO

Any organization, commercial firm or interested individual is encouraged to submit relevant specifications, or proposals for revision of existing specifications, for pesticide products for consideration and possible adoption by FAO. Correspondence should be addressed to the Pesticide Management Group, Plant Production and Protection Division, FAO, Viale delle Terme di Caracalla, 00153 Rome, Italy.

General guidelines on preparing draft specifications are given in the *Manual on the development and use of FAO and WHO Specifications for Plant Protection Products*, FAO Plant Production and Protection Paper No. 173, Rome 2002 (Revised First Edition available only on the FAO home page of the Internet at: http://www.fao.org/pest-and-pesticide-management/en/).

Specifications which are considered suitable for further processing are assigned priorities and circulated to appropriate organizations and specialists to comment. Comments, together with other relevant information, are then reviewed in detail by the Group on Specifications of the FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent. The drafts are converted into FAO Provisional Specifications, or full FAO Specifications.

AGP:CP/336

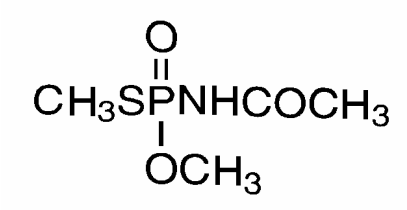
ACEPHATE

O,S-dimethyl acetylphosphoramidothioate

INFORMATION

COMMON NAME: acephate (ISO)

STRUCTURAL FORMULA:



EMPIRICAL FORMULA: $C_4H_{10}NO_3PS$

RMM: 183.2

CAS REGISTRY NUMBER: 30560-19-1

CIPAC CODE NUMBER: 338

EEC NUMBER: 015-079-00-7

CHEMICAL NAMES: (IUPAC)

O,*S*-dimethyl acetylphosphoramidothioate

N-[methoxy(methylthio)phosphinoyl]acetamide (CA)

ACEPHATE TECHNICAL
FAO Specification 338/TC/S/P (1995)

1. DESCRIPTION

The material shall consist of acephate together with related manufacturing impurities and shall be a white crystalline powder with a strong mercaptan-like odour. The material shall be free from visible extraneous matter and added modifying agents.

2. ACTIVE INGREDIENT

2.1 Identity test (CIPAC H, p.6)

An identity test is required if the identity of the active ingredient is in doubt.

Acephate (CIPAC H, p.6) [2]

The acephate content shall be declared (not less than 990 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 10 g/kg.

3 IMPURITIES

3.1 Methamidophos[3]
Maximum: 5.0 g/kg.

3.2 *O,O,S*-trimethyl phosphorothioate[3]
Maximum: 1.0 g/kg.

3.3 Acetamide[3]
Maximum: 1.0 g/kg.

3.4 Water (MT. 30, CIPAC F, p.91)
Maximum: 2 g/kg (Note 1).

---

[2] The analytical method for determination of the active ingredient is available from the Pesticide Management Group of the FAO Plant Protection Service, or can be downloaded here

[3] The analytical method for determination of the relevant impurity is available from the Pesticide Management Group of the FAO Plant Protection Service, or can be downloaded here.

4. PHYSICAL PROPERTIES

4.1 pH range (MT 75, CIPAC F, p.205)

pH range: 3.4 to 3.6.

4.2 Melting range (MT 2, CIPAC F, p.5)

Melting range: 88-90°C.

NOTES

1. Typically the water content is about 1.5 g/kg. However, as acephate technical is hygroscopic, care must be taken to package it in moisture-proof containers and store in a dry location.

ACEPHATE WATER SOLUBLE POWDERS

FAO specification 338/SP/S/P (1995)

1. DESCRIPTION

The material shall consist of a homogeneous mixture of technical acephate, complying with the requirements of FAO specification 338/TC/S/P (1995), together with any necessary formulants. It shall be in the form of a powder to be applied as a true solution of the active ingredient after dissolution in water but which may contain insoluble inert ingredients.

2. ACTIVE INGREDIENT

2.1 Identity test (CIPAC H, p.8)

An identity test is required if the identity of the active ingredient is in doubt.

2.2 Acephate (CIPAC H, p.8)[4]

The acephate content shall be declared (g/kg) and, when determined, the content obtained shall not differ from that declared by more that the following amounts.

Declared content	Permitted tolerance
Up to 500 g/kg	± 5% of the declared content
Above 500 g/kg	± 25 g/kg.

3. IMPURITIES

3.1 Methamidophos[5]

Maximum: 0.5% of the acephate content found under 2.2.

3.2 *O,O,S*-trimethyl phosphorothioate[5]

Maximum: 0.1% of the acephate content found under 2.2.

3.3 Acetamide[5]

Maximum: 0.1% of the acephate content found under 2.2.

---

[4] The analytical method for determination of the active ingredient is available from the Pesticide Management Group of the FAO Plant Protection Service, or can be downloaded here

[5] The analytical method for determination of the relevant impurity is available from the Pesticide Management Group of the FAO Plant Protection Service, or can be downloaded here.

3.4 Water content (MT 30, CIPAC F, p.91)

Maximum: 20 g/kg.

3.5 Material insoluble in water (MT10, CIPAC F, p.27)

Maximum: 220 g/kg.

4. PHYSICAL PROPERTIES

4.1 pH range (MT 75.2, CIPAC F, p.206)

pH range: 3.5 to 3.8.

4.2 Wet sieve test (MT 59.3, CIPAC F, p.179)

Maximum: 2% retained on a 75 µm test sieve.

4.3 Rate of dissolution and solution stability (CIPAC MT 60, CIPAC F, p.182)

After 5 minutes:	opaque suspension with slight settling of particles (< 1.0 ml) and no large particles observed.
After 18 hours:	4.5 ml of fine white particulate sediment and 95.5 ml of translucent supernatant containing very fine suspended particles.

4.4 Wetting of the material (MT 53.3.1, CIPAC F, p.164)

The product shall be completely wetted in less than 1 min without swirling.

5. STORAGE STABILITY

5.1 Stability at 54°C (MT 46.1.1, CIPAC F, p.149)

After storage at 54 ± 2°Cfor 14 days, the determined average active ingredient content must not be lower that 97% relative to the determined average content found before storage (Note 1) and the product shall continue to comply with 3.1, 4.1, 4.3 and 4.4.

NOTES

1. Samples of the product taken before and after the storage stability test should be analysed together after the test to reduce the analytical error.

ASSAY FOR ACEPHATE ACTIVE INGREDIENT (A.I.) IN ACEPHATE TECHNICAL

Arysta LifeScience Method: ACE-100 (Version 1.0)

This procedure is based on Tomen Method TM-ORT/TC[1], as validated by Analytical Development Corporation[2] (refer to ADC Report 1589-5).

1 STANDARDS AND REAGENTS

Acephate reference standard of known purity (store at nominal -20 ºC)

Diethylphthalate, 99%, Aldrich or equivalent

Dichloromethane (DCM), Fisher Optima or equivalent

2 EQUIPMENT

GC capillary column, fused silica, DB-17, 15 m x 0.53 mm, 1-µm film, J&W or equivalent

Analytical balance, capable of weighing to 0.0001-g accuracy

General laboratory glassware (including 20-mL glass vials)

Data collection and integration system

Gas chromatograph (e.g., HP 5890) equipped with a flame ionization (FID) detector, autoinjector, and a split/splitless injection port (with a glass capillary insert containing silanized glass wool)

3 SOLUTIONS

3.1 Internal Standard

Weigh 2.62 g of diethylphthalate and quantitatively transfer into a 1-L volumetric flask with DCM. Dilute to volume with DCM.

[1] "Determination of Acephate in Technical and Formulated Products", 05/22/2000.
[2] 4405 N. Chestnut Street, Suite D, Colorado Springs, CO 80907, 719-260-1711.

3.2 Acephate Standard

Prepare stock solutions in a fume hood.

Accurately (to the nearest 0.0001 g) weigh two 0.1-g aliquots of acephate standard (corrected for the percent purity, refer to **Section 4.3.1**) and quantitatively transfer each to glass vials with 20 mL of internal standard solution (using a volumetric pipet).

Shake the vials by hand to assure dissolution. Label the vials STD A and STD B, respectively. Transfer portions of the standard solutions to GC vials for analysis.

Note: It is recommended that the GC system be equilibrated prior to the injection run by injecting a standard solution twice.

To verify acceptability of the acephate quantitation standard preparation, inject STD A and STD B in triplicate (alternating STD A and STD B). For each injection, determine the peak response ratio (refer to **Section 4.3.2**) and then determine the mean peak response ratio for STD A and for STD B. The mean peak response ratios must agree to within 2% (corrected for the actual amount of standard weighed, refer to **Section 4.3.3**). One of the standard solutions is then designated for use in quantitation of acephate in the technical samples.

4 PROCEDURE

4.1 Sample Preparation

Accurately (to the nearest 0.0001 g) weigh duplicate 0.1-g aliquots of the technical sample. Quantitatively transfer the sample aliquots to glass vial with 20 mL of internal standard solution (using a volumetric pipet).

Shake the vial by hand several times to dissolve the sample. Transfer a portion of each sample solution to a GC vial for analysis.

4.2 Analysis by Gas Chromatography with FID Detection

Injection order: Inject three standards, duplicate sample extracts, a standard, duplicate sample extracts, a standard, etc., such that every two sample injections are bracketed by standard injections.

Note: Stated GC conditions may be modified to obtain acceptable chromatography.

Temperatures:

Column oven: 175 °C (isothermal, run time 5 min)
Injection port: 220 °C
Detector: 250 °C

Gas flows:

Carrier (helium): 10 mL/min
Split vent: 400 mL/min (split ratio 40:1)
Detector hydrogen: 30 mL/min
Detector air: ~400 mL/min
Makeup (helium): 22 mL/min
Split purge on at 0 min (split injection)

Injection volume: 1 µL

Approximate retention times: Acephate: 1.2 min
Internal standard: 1.5 min

For each injection, calculate the peak response ratio (acephate response/internal standard response). The ratios for the three standard injections preceding the first sample should agree to within 2%. For sample results to be deemed acceptable, the peak response ratio of the two standards bracketing the duplicate sample extracts must agree to within 2% and the peak response ratio of the duplicate sample extracts must agree to within 2%.

4.3 Calculations

4.3.1 Weight of standard used for calculations, corrected for purity =

$$\frac{\%\ \text{purity}}{100} \times \text{aliquot weighed (g)}$$

4.3.2 Peak response ratio =

$$\frac{\text{peak response* for acephate}}{\text{peak response* for internal std.}}$$

* µV (area or height)

4.3.3 Normalized mean response ratio =

$$\frac{\text{mean response ratio of standard}}{\text{concentration (mg/mL) of standard aliquot}}$$

[Note: This calculation is only required if the weights of the A and B reference standard aliquots are not equivalent.]

4.3.4 % Difference of STD A and STD B solutions =

$$100 \times \frac{(\text{mean* response ratio STD B} - \text{mean* response ratio STD A})}{\text{mean* response ratio STD A}}$$

*Normalized, if required, as described in **Section 4.3.3**.

4.3.5 % Response ratio difference for bracketing standard injections =

$$100 \times \frac{(\text{response ratio second injection} - \text{response ratio first injection})}{\text{response ratio first injection}}$$

4.3.6 Mean response ratio of bracketing standards =

$$\frac{(\text{response ratio first standard injection} + \text{response ratio second standard injection})}{2}$$

4.3.7 % Content (purity) of acephate a.i. found for sample extract solution =

$$100 \times \frac{[\text{response ratio of sample/sample wt. (g)}] \times \text{wt. (g) of standard}^{**}}{\text{mean response ratio of bracketing standard injections}}$$

Corrected for purity, as described in **Section 4.3.1 (expressed to 4 decimal places).

4.3.8 % Difference in the duplicate sample preparations =

$$100 \times \frac{(\%\text{ acephate found for the second replicate} - \%\text{ acephate found for the first replicate})}{\%\text{ acephate found for the first replicate}}$$

4.3.9 Mean percent acephate found =

$$\frac{(\%\text{ acephate found for first replicate} + \%\text{ acephate found for second replicate})}{2}$$

Note: The mean of the percent acephate found for the duplicate assays is reported.

4.4 Notes

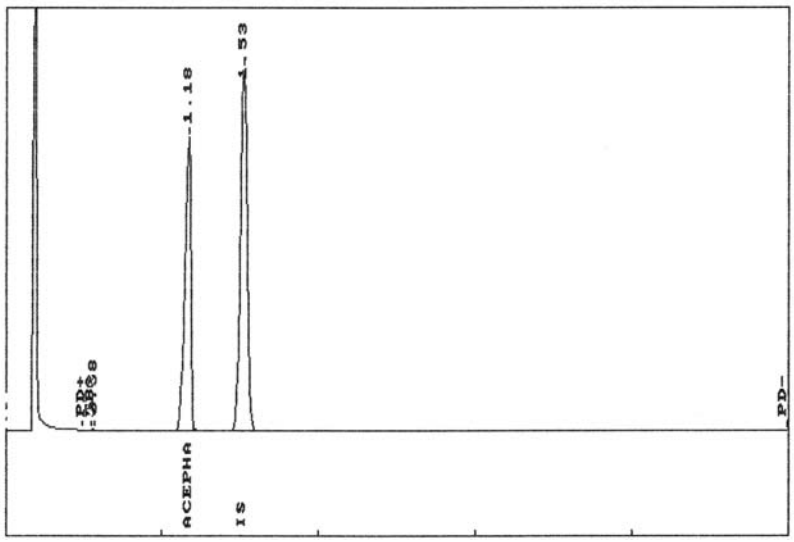
If the peak response ratios for bracketing standard injections do not agree within 2%, the injection port septum and the silanized glass wool in the injection port liner may need to be replaced. After remedial measures are taken, the samples and standards should be reinjected (fresh aliquots should be loaded if the vials have been on the autoinjector tray overnight or if significant solvent evaporation may have occurred).

If the results for the bracketing standards are acceptable, but the results for the duplicate sample extractions do not agree to within 2%, the run should be reinjected.

If the results are still unacceptable, new replicate extractions of the sample may need to be prepared.

Equivalent equipment and reagents may be substituted.

Representative Standard Chromatogram for Quantitation of Acephate A.I.
Column: DB-17, 15 m x 0.53 mm, 1-µm film



ASSAY FOR THREE FAO IMPURITIES (ACETAMIDE, RE-15283, AND METHAMIDOPHOS) IN ACEPHATE TECHNICAL

Arysta LifeScience Method: ACE-106 (Version 1.0)

This procedure is based on Chevron methods ORT-330[1] and ORT-390.[2]

1 STANDARDS AND REAGENTS

Reference standards of known purity (store at nominal -20 ºC)

Acetamide
RE-15283 (a.k.a. O,O,S-trimethylphosphorothioate)
Methamidophos

Dipropylphthalate, 99%, Aldrich or equivalent

Dichloromethane (DCM), Fisher Optima or equivalent

2 EQUIPMENT

GC capillary column, fused silica, DB-17, 15 m x 0.53 mm fused silica, 1-µm film thickness, J&W Scientific or equivalent)

Analytical balance, capable of weighing to 0.0001-g accuracy
General laboratory glassware

Data collection and integration system

Gas chromatograph (e.g., HP 5890) equipped with a flame ionization (FID) detector, autoinjector, and a split/splitless injection port (with a glass capillary insert containing silanized glass wool)

3 SOLUTIONS

3.1 Internal Standard

Dilute 0.6 mL of dipropylphthalate to 1,000 mL with DCM.

[1] "Acetamides and RE-15283", 06/09/1990.
[2] "RE-9171, DPA, Monitor, and DAPA in Orthene Technical", 01/17/1991.

3.2 Stock Impurity Standards

Prepare stock solutions in a fume hood.

Accurately (to the nearest 0.0001 g) weigh 0.0500 g of each impurity standard (corrected for the percent purity, refer to **Section 4.3.1**) and quantitatively transfer each to individual 50-mL volumetric flasks using DCM. Calculate the actual concentration (1 mg/mL, nominal).

3.3 Combined Impurity Standard Solution

Pipet 5 mL of each stock impurity standard solution (nominal 1 mg/mL each) into a 100-mL volumetric flask, add 3 mL of internal standard solution using a volumetric pipet, and dilute to volume with DCM. Calculate the actual concentration (50 µg/mL, nominal) of each impurity in the combined standard solution.

4 PROCEDURE

4.1 Sample Preparation

Accurately (to the nearest 0.0001 g) weigh 1 g of the technical sample. Quantitatively transfer the sample aliquot to a 100-mL volumetric flask using DCM. Add 3 mL of internal standard solution using a volumetric pipet, then dilute to volume with DCM.

Shake the flask by hand several times to dissolve the sample. Transfer a portion of the sample solution to a GC vial for analysis.

4.2 Analysis by Gas Chromatography with FID Detection

Injection order: three combined standards, two sample extracts, a combined standard, two sample extracts, a combined standard, etc., such that two sample injections are bracketed by injections of the combined standard.

Note: Stated GC conditions may be modified to obtain acceptable chromatography.

Temperatures:

Oven temperature ramp: 60 °C, hold 4 min, ramp at 10 °C per min to 100 °C, hold 4 min, ramp 10 °C/min to 200 °C, hold 4 min. Equilibration time: 2 min.
Inlet temperature: 150 °C
Detector temperature: 250 °C

Gas flows:

Carrier helium at 5 mL/min
Split vent helium at 40 mL/min. Purge time on: 0.5 min (splitless injection).
Hydrogen (detector gas) at 30 mL/min
Air (detector gas) at 400 mL/min
Makeup gas (helium) at 22 mL/min

Injection volume: 1 µL

Approximate retention times:		
	Acetamide	0.9 min
	RE-15283	7.1 min
	Methamidophos	10.9 min
	Internal Standard	19.5 min

Notes: To confirm elution order, inject the individual impurity stock solutions (this need not be repeated for every run). An acephate peak will be present (retention time ~16 min) in technical sample chromatograms; however, acephate is not quantitated in the assay, because the peak size saturates the detector.

For each injection, calculate the peak response ratio (impurity response/internal standard response). The ratios for the three combined standard injections preceding the first sample should agree to within 10%. For sample results to be deemed acceptable, the peak response ratio of the two combined standards bracketing the sample extracts must agree within 10%.

4.3 Calculations

4.3.1 Weight of standard used for calculations, corrected for purity =

$$\frac{\%\ \text{purity}}{100} \times \text{aliquot weighed (g)}$$

4.3.2 Concentration (mg/mL) of each stock standard solution =

$$\frac{\text{weight of standard corrected for purity (g)} \times 1{,}000\ \text{mg/g}}{\text{final volume (mL)}}$$

4.3.3 Concentration (µg/mL) of each analyte in the combined standard solution =

$$\frac{\text{aliquot of stock standard (mL)} \times \text{mg/mL concentration of stock} \times 1{,}000\ \mu\text{g/mg}}{\text{final volume (mL)}}$$

4.3.4 Peak response ratio =

$$\frac{\text{peak response* for impurity}}{\text{peak response* for internal std.}}$$

* µV (area or height)

4.3.5 % Response ratio difference for bracketing standard injections =

$$100 \times \frac{\text{(response ratio second injection - response ratio first injection)}}{\text{response ratio first injection}}$$

4.3.6 Mean response ratio of bracketing standards =

$$\frac{\text{(response ratio first standard injection + response ratio second standard injection)}}{2}$$

4.3.7 Concentration (µg/mL) found for sample extract solution =

$$\frac{\text{µg/mL conc. of std. x sample extract response ratio}}{\text{mean response ratio of bracketing standards}}$$

4.3.8 Percent impurity in technical sample =

$$100 \times \frac{\text{(µg/mL conc. found for sample extract x mL extract volume)/1,000,000 µg/g}}{\text{sample wt (g)}}$$

4.4 Notes

If the acetamide peak elutes too close to the solvent front, the GC flow and/or temperature setting should be adjusted so that the acetamide peak is more resolved from the solvent front.

If the peak response ratios for bracketing standard injections do not agree within 10%, the injection port septum and the silanized glass wool in the injection port liner may need to be replaced. After remedial measures are taken, the samples and standards should be reinjected (fresh aliquots should be loaded if the vials have been on the autoinjector tray overnight or if significant solvent evaporation may have occurred). If the results are still unacceptable, the samples may need to be re-extracted.

Equivalent equipment and reagents may be substituted.

Representative Standard Chromatogram for Quantitation of Three FAO Impurities
Column: DB-17, 15 m x 0.53 mm, 1-µm film