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FAO SPECIFICATIONS FOR PLANT PROTECTION PRODUCTS

CAPTAN

N-(trichloromethylthio)cyclohex-4-ene-1,2-dicarboximide

Group on Pesticide Specifications

FAO Panel of Experts on Pesticide Specifications, Registration Requirements and
Application Standards

Technical Secretary: Dr. A.V. Adam
Plant Protection Service
Plant Production and Protection Division

FAO

Via delle Terme di Caracalla
00100 Rome, Italy - Telex: 610181 FAO I

CONTENTS

DISCLAIMER	3
INTRODUCTION TO FAO SPECIFICATIONS DEVELOPED UNDER THE OLD PROCEDURE.....	4
SUBMISSION OF DRAFT SPECIFICATIONS TO FAO	7
INFORMATION.....	8
CAPTAN TECHNICAL	9
CAPTAN WETTABLE POWDERS	10
CAPTAN DUSTABLE POWDERS.....	12
APPENDIX 1	14

DISCLAIMER¹

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. Classes of Specifications.

FAO (full) specifications (Code "S"). Specifications that have all necessary requirements together with CIPAC (full) methods, or other collaboratively studied (proven) methods. 2/ and 3/.

FAO Provisional specifications [Code (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 Tentative specifications (Code "ts") are those which have been recommended by FAO as preliminary specifications 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.

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

2. Expression of Active Ingredient Content

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

- 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 buyer 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.

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 above the nominal figures. 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 content of active ingredient. For examples of such permitted tolerances, see the table on page 20 of the Manual.

4. Containers/Packaging.

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

- Technical material, dustable powders and granules

Containers shall be suitable, clean, dry and as specified, and shall not adversely affect, or be affected by, the product/material, but shall adequately protect it 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, oxidation, 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 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 possible phytotoxicity of 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 1, p. 965 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 or 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) and ID (1988), 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 brackets in the specifications. A copy of a method 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 Pesticides Control Officer, Plant Production and Protection Division, FAO, Via delle Terme di Caracalla, 00100, Rome, Italy.

FAO has published a *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.

INFORMATION

COMMON NAME: CAPTAN (ISO)

EMPIRICAL FORMULA: C₉H₈Cl₃NO₂S

RMM: 300.6

CAS REGISTRY NUMBER: 133-06-2

CIPAC CODE NUMBER: 40

CHEMICAL NAME:

N-(trichloromethylthio)cyclohex-4-ene-1,2-dicarboximide (IUPAC)
1,2,3,6-tetrahydro-N-(trichloromethylthio)phthalamide
3a,4,7,7a-tetrahydro-N-(trichloromethanesulphenyl)phthalimide
3a,4,7,7a-tetrahydro-2-[(trichloromethyl)]-1H-isoindole-1,3(2H)-dione
(CA)

CAPTAN TECHNICAL
FAO Specification 40/TC/S (1990)

.1 DESCRIPTION

The material shall consist of captan together with related manufacturing impurities and shall be an off-white to tan coloured powder free from visible extraneous matter and added modifying agents.

.2 ACTIVE INGREDIENTS

.2.1 Identity test (CIPAC 1C, 40/TC/M.3/2 p. 2013 and 40/TC/M.4/2 p. 2015)

Where the identity of the active ingredient is in doubt, then it shall comply with at least one additional test.

.2.2 Captan (CIPAC 1C, 40/TC/M.3/3 p. 2013 (Referee method) or -/M.4/3 p. 2015)

The captan content shall be declared (not less than 910 g/kg) and, when determined, the content obtained shall not differ from that declared by more than ± 30 g.

.3 IMPURITIES

.3.1 Perchlormethylmercaptan *

Maximum: 10 g/kg

.3.2 Loss on drying (MT 17.4.1, CIPAC 1, p. 874)

Maximum: 15 g/kg

.4 PHYSICAL PROPERTIES

.4.1 pH of 1% aqueous dispersion (MT 75, CIPAC 1A, p. 1589)

Range: 7.0 to 8.5

* The analytical method for determination of the relevant impurities is available from the Pesticide Management Group of the FAO Plant Protection Service or can be [downloaded here](#).

CAPTAN WETTABLE POWDERS
FAO Specification 40/WP/S) (1990)

.1 DESCRIPTION

The material shall consist of a homogenous mixture of technical captan [complying with the requirements of FAO specification 40/TC/S (1990)] together with filler(s) and any other necessary formulants. It shall be in the form of a fine powder free from visible extraneous matter and hard lumps.

.2 ACTIVE INGREDIENTS

.2.1 Identity tests (CIPAC 1C, 40/WP/M.3/2 p. 2017 and 40/WP/M.4/2 p. 2017)

Where the identity of the active ingredient is in doubt, then the isolated active ingredient shall comply with at least on additional test.

.2.2 Captan (CIPAC 1C, 40/WP/M.3/3 p. 2017 (referee method) or -/M.4/3 p. 2017)

The captan content shall be declared (g/kg) and when determined the content obtained shall not differ from that declared by more than the following amounts:

<u>Declared content</u>	<u>Permitted tolerance</u>
up to 400 g/kg	+/- 5% of the declared content
above 400 g/kg	+/- 20 g

.3 IMPURITIES

.3.1 Perchlormethylmercaptan 1/

Maximum: 1% of the captan content found under .2.2

1/ 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 of 1% aqueous dispersion (MT 75, CIPAC 1A, p.1589)

Minimum: 6.5

.4.2 Wet sieve test (MT 59.3, CIPAC 1, p. 981)

Maximum: 2% retained on a 75 µm test sieve

.4.3 Suspensibility (see Appendix 1)

A minimum of 60% of the captan content found under .2.2 shall be in suspension after 30 min. in CIPAC Standard Water C. (Notes 1 and 2)

Alternatively, if the buyer, requires other CIPAC Standard Waters to be used, then this shall be specified when ordering.

.4.4 Persistent foam (MT 47, CIPAC 1, p. 954) (Note 3)

Maximum: 25 ml after 1 minute

.4.5 Wetting of the product (MT 53.3.1, CIPAC 1, p. 967)

The product shall be completely wetted in 1 minute without swirling.

.5 STORAGE STABILITY

.5.1 Stability at 54 C (MT 46.1.1, CIPAC 1, p.951)

After storage at 54 +/- 2 C for 14 days, the product shall continue to comply with .2.2, .3.1, .4.1, .4.2, .4.3 and .4.5.

Note 1 The product should be tested at the highest and lowest rates of use recommended by the supplier, provided this does not exceed the conditions given in the method.

Note 2 This test will normally only be carried out after the heat stability test 5.1.

Note 3 The amount of sample to be used in the test should be specified.

CAPTAN DUSTABLE POWDERS
FAO Specification 40/DP/S) (1990)

.1 DESCRIPTION

The material shall consist of a homogenous mixture of technical captan [complying with the requirements of FAO specification 40/TC/S (1990)] together with carriers and any other necessary formulants. It shall be in the form of a fine, free-flowing powder free from visible extraneous matter and hard lumps.

.2 ACTIVE INGREDIENTS

.2.1 Identity tests (CIPAC 1C, 40/DP/M.3/2 p. 2018 and 40/DP/M.4/2 p. 2018)

Where the identity of the active ingredient is in doubt, then the isolated active ingredient shall comply with at least one additional test.

.2.2 Captan (CIPAC 1C, 40/DP/M.3/3 p. 2018 (Referee method) or -/M.4/3 p. 2018)

The captan content shall be declared (g/kg) and, when determined, the content obtained shall not differ from that declared by more than +/- 10%.

.3 IMPURITIES

.3.1 Perchlormethylmercaptan 1/

Maximum: 1% of the captan content found under .2.2

.4 PHYSICAL PROPERTIES

.4.1 pH of 1% aqueous dispersion (MT 75, CIPAC 1A, p.1589)

Minimum: 6.5

.4.2 Dry sieve test (CIPAC 1, 40/2/M.1/1.4, p. 178)

Maximum: 5% retained on a 75 µm test sieve. Not more than (0.005 x X) % of the amount of sample used for the determination shall be present as captan in the residue on the sieve, where X is the captan content (g/kg) found under .2.2 (Note 1)

.4.3 Flowability (MT 44, CIPAC 1A, p. 1567)

If required, maximum flow number: 12

In the absence of proven methodology, this clause is for information only.

.5 STORAGE STABILITY

.5.1 Stability at 54 C (MT 46.1.1, CIPAC 1, p.953)

After storage at 54 +/- 2 C for 14 days, the product shall continue to comply with .2.2, .3.1, .4.1, and .4.2.

Note 1 If the product has a found content of 50 g/kg of captan and 20 g of sample is used in the test, then the amount of captan in the residue on the sieve should not exceed 0.05 g, i.e.,

$$\frac{(0.005 \times 50) \times 20}{100} \quad \text{g}$$

1/ 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](#).

DETERMINATION OF SUSPENSIBILITY

Determine the suspensibility by MT 15.1 (CIPAC 1, p.861), using the appropriate CIPAC Standard Water.

Use amounts of sample sufficient to give concentrations in the 250 ml suspensions equivalent to the highest and lowest rates of use recommended for the product (Note 1). Carry out the determination on the suspension at a temperature of 30 +/- 2 C.

Determine the captan content in the bottom 25 ml of suspension as follows:

Transfer the bottom 25 ml of suspension quantitatively into a 250 ml separating funnel using 25 ml of distilled water. Add 50 ml of chloroform, shake for 2 minutes to dissolve the captan. Allow the two layers to separate and run off the bottom chloroform layer through a filter paper into a 250 ml round bottom flask. Repeat with two further 50 ml portions of chloroform. Evaporate the solvent using a rotary evaporator and eliminate the traces of solvent using a light current of dry air.

Determine the captan content of the residue either by method 40/WP/M.3/3, CIPAC 1C, p.2017 or method 40/WP/M.4/3, p.2017.

Note 1 The method is not suitable for high concentrations as recommended for aerial or low volume applications, i.e., above 1% captan in the diluted spray.



ANALYTICAL METHOD

FOLPAN PROJECT

<i>High Performance Liquid Chromatographic Method</i>		
Subject: Determination of Perchloromethyl Mercaptan (PCMM) in organic solutions by HPLC.	<u>Method No:</u>	
	<u>Version:</u>	<u>02</u>
	<u>Effective Date:</u>	<u>25.09.2006</u>
	<u>Number of Copies</u>	<u>4</u>

Author:	Signature:	Date:
Mr. Siritsov Alexander		01 February 07

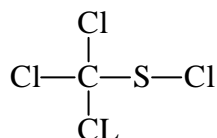
Checked and Approved by:	Signature:	Date:
Dr. Wilfried Mayer		01 February 07

A prerequisite for the use of this analytical method is that the person carrying it out has appropriate training in chemical analysis, is familiar with the safe handling of chemicals and analytical apparatus and has been informed about general laboratory guidelines relating to safety precautions and accident prevention and strictly observes these.

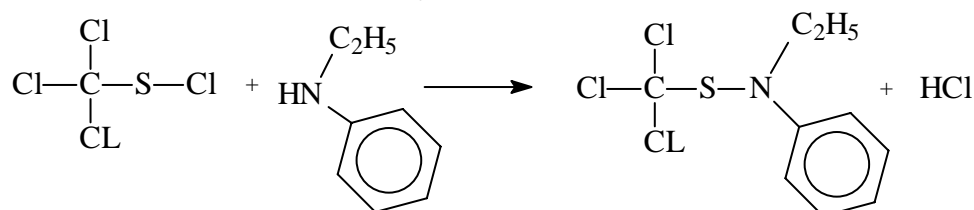
Scope:

This method is applicable for the determination of PCMM as its Ethylaniline Derivative in organic solutions and reaction mixtures.

The Perchloromethyl Mercaptan (PCMM) structure appears below:



The reaction of PCMM Ethylaniline Derivatization is as follows:



Summary of Method:

A HPLC Chromatographic System employing a column Merck LiChrospher RP-18e Column was used to separate and determine the PCMM derivate concentration. The Internal Standard Method was used for quantification purposes and a data acquisition system was used to calculate peak areas. Detection was achieved using a PDA detector.

Linearity:

The Linearity of the PCMM Derivative was tested by injecting a five (5) point calibration curve covering the range of 61.3-184.7mg/50ml of PCMM technical and constant weight of Dibutyl phthalate (Internal standard). The results expressed % relative standard deviation was found to be **0.77**.

Sample #	Weight of PCMM	Area of PCMM-der.	Area of IS	Weight of IS	Factor
ST 1	61.3	947501	1266396	70.0	0.8544
ST 1	61.3	944897	1262778	70.0	0.8545
ST 2	94.6	1467013	1280326	70.0	0.8479
ST 2	94.6	1461888	1277296	70.0	0.8469
ST 3	116.6	1776580	1266542	70.0	0.8421
ST 3	116.6	1762053	1265649	70.0	0.8358
ST 4	151.8	2451286	1328572	70.0	0.8508
ST 4	151.8	2367384	1283350	70.0	0.8506
ST 5	184.7	2853163	1287215	70.0	0.8401
ST 5	184.7	2935201	1324167	70.0	0.8401

Method Precision:

The Method Precision for the PCMM derivative was calculated from duplicate injections of five (5) weights of the same PCMM technical Sample and constant weight of Dibutyl phthalate (Internal standard). The Relative standard deviation was found to be **0.5%**.

Sample #	Weight of PCMM	Area of PCMM-der.	Area of IS	Weight of IS	Factor
# 1	118.4	1826451	1298361	70.0	0.8317
# 1	118.4	1830638	1300476	70.0	0.8322
# 2	137.3	2170449	1331220	70.0	0.8312
# 2	137.3	2154595	1320980	70.0	0.8316
# 3	139.9	2183138	1302208	70.0	0.8388
# 3	139.9	2223149	1326864	70.0	0.8383
# 4	118.7	1880582	1328632	70.0	0.8347
# 4	118.7	1835808	1307390	70.0	0.8281
# 5	114.5	1747376	1299644	70.0	0.8220
# 5	114.5	1757351	1299787	70.0	0.8266

System Precision:

The System Precision for PCMM Derivative was calculated from seven (7) injection of the same PCMM technical Sample and a constant weight of Dibutyl phthalate (Internal standard). The relative standard deviation was found to be **0.3%**.

Sample #	Weight of PCMM.	Area of PCMM-der.	Area of IS	Weight of IS	Factor
ST 3	116.6	1737680	1246956	70.0	0.8366
ST 3	116.6	1750047	1255523	70.0	0.8368
ST 3	116.6	1730340	1250274	70.0	0.8309
ST 3	116.6	1726984	1247109	70.0	0.8313
ST 3	116.6	1722549	1243171	70.0	0.8318
ST 3	116.6	1711887	1237871	70.0	0.8302
ST 3	116.6	1717697	1237883	70.0	0.8330

Safety Precautions:

Normal Safety Precautions; Safe handling practice and standard analytical techniques should be used. Consult the material safety data sheets for full hazard details.

Chemicals, Equipment, Instrumental conditions:

Reagents and Materials:

Solvents: Acetonitrile HPLC Gradient grade, LAB-SCAN.
Water deionized and distilled.

Reagents: Sodium hydrogen phosphate anhydrous
(Na₂HPO₄) >99%; Merck; cat#106586.
PCMM technical, 98% MCW org. laboratory.
Dibutyl phthalate; >99%; Merck; cat# 800919.
N-Ethyl aniline; >98%; Merck; cat# 821662.

Apparatus and HPLC chromatographic conditions:

Chromatograph: Thermo-Finnigan Surveyor, HPLC
Chromatograph with Auto Injector,
and data acquisition system ChromQuest.

Detector : PDA; 225nm

Column: Merck; LiChrospher 100; RP-18e (5µm);
250 x 4.6mm.
Column temperature: 40°C

Eluent: 80% Acetonitrile
20% Buffer Sodium Hydrogen Phosphate 0.005M

Flow: 1,5 ml/min

Injection volume: 5 µl

Retention Time: Dibutyl phthalate: 3.9min;
PCMM derivate: 6.3min.

Procedure:

Preparation of Derivatization Reagent

Weigh accurately about 0.3-0.5gr of N-ethyl aniline into a 10ml volumetric flask, add 5ml of Acetonitrile and mix.

Preparation of Internal Standard Solution

Weigh accurately about 3.50gr of Dibutyl Phthalate into a 250ml volumetric flask, dilute to mark with Acetonitrile and homogenize.

Preparation Standard Solutions

Weigh in triplicate accurately about 70-110mg of PCMM standard into a 50ml volumetric Flask. Add 5ml of the internal standard solution (70mg of Dibutyl phthalate) and dilute to mark with Acetonitrile.

Attention: Weigh and prepare Standard always freshly before analysis!

Sample Preparation

For the determination of PCMM in Test Sample transfer 5ml of the Internal Standard solution into a 50ml volumetric flask and additionally weigh into the same flask Test Sample according to expected concentration (For example: 400mg for 25% concentration). Continue as described under standard preparation.

Derivatization Procedure

Pipette 200 μ l of Derivatization reagent and 200 μ l of standard (or sample) solution into a 2ml injection vial and mixed. Add 900 μ l of Acetonitrile, homogenize and inject.

Calculation:

The concentration of PCMM was calculated as follows:

$$CONC (\%) = \frac{R_{smp} * R_{is(standard)} * C_{std}}{R_{is(sample)} * R_{std}}$$

Where:

R_{smp} = Ratio of Sample Area / Sample Weigh

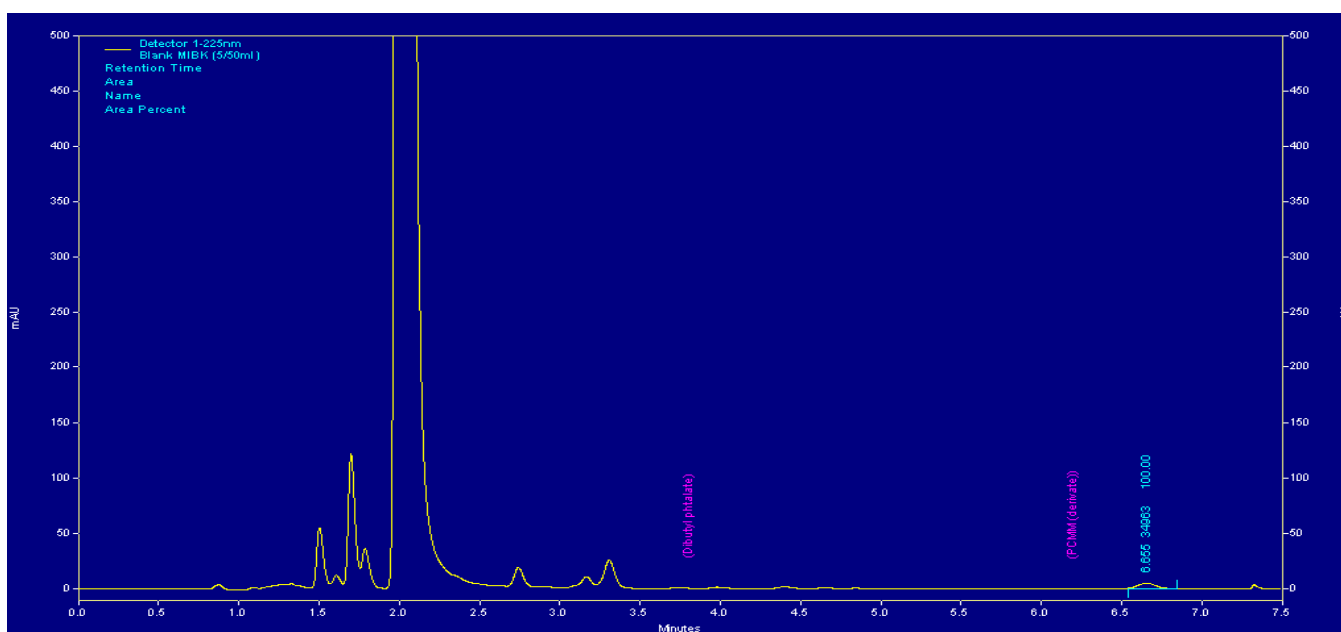
$R_{is(sample)}$ = Ratio of internal standard Area in Sample / Weight of internal standard in Sample.

R_{std} = Ratio of Standard Area / Standard Weight

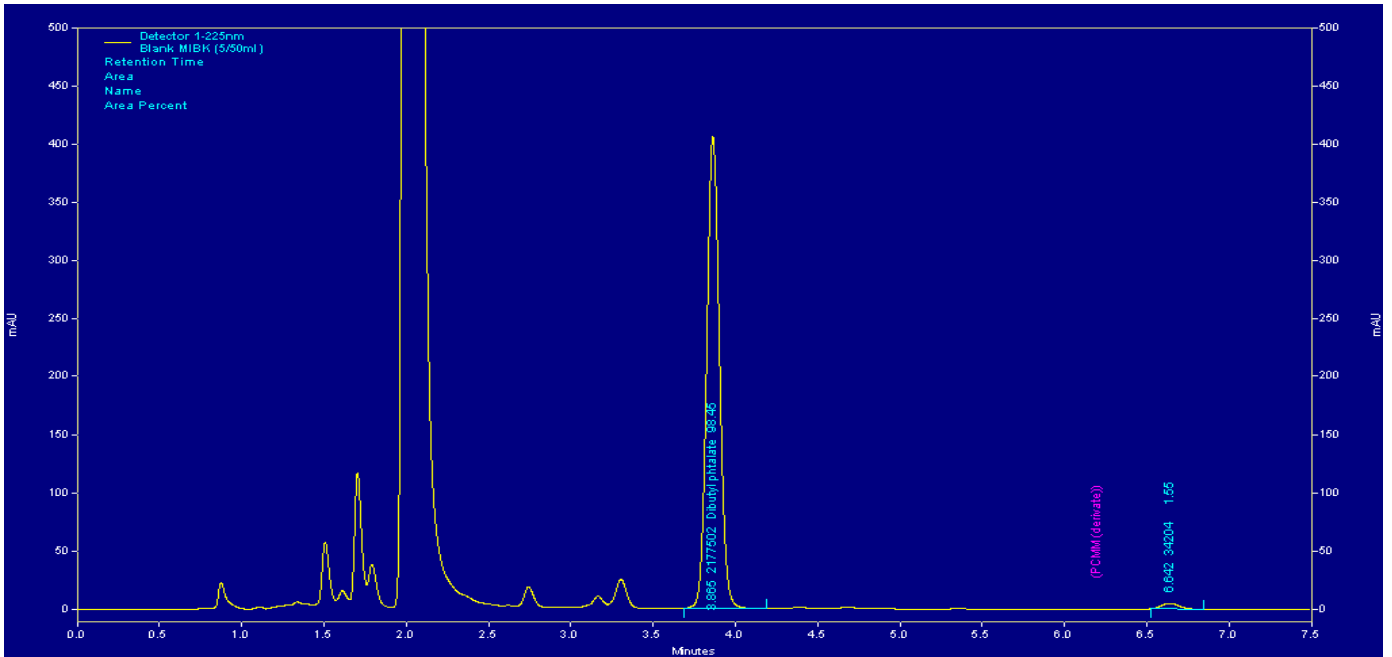
$R_{is(standard)}$ = Ratio of Internal Standard Area / Weight of Internal Standard in PCMM Standard .

C_{std} = Purity of the standard (PCMM).

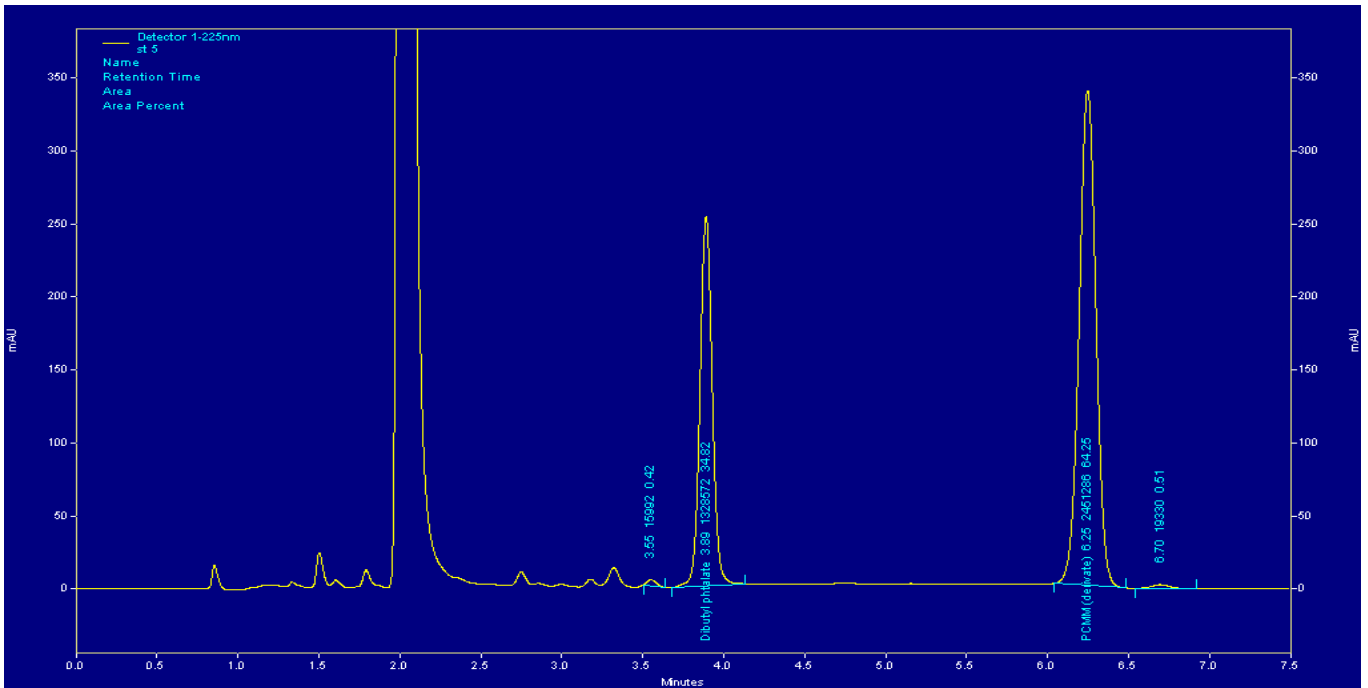
Blank 1 (N-Ethyl aniline)



Blank 2 (N-Ethyl aniline and Dibutyl phthalate)



Chromatogram of PCMM derivate and IS (Dibutyl phthalate)



The specified conditions should be regarded as binding.

If, in exceptional cases, the specific situation in the laboratory (e.g. none-availability of equipment, reagents etc.) necessitates the adaptation of individual parameters of the method, this can be recorded on a supplementary page in conformity with all QA and GLP requirements. This does not change the official analytical method, its code number or its general validity.

The conditions to be entered on the supplementary page of the method documentation are only valid after approval in the form of the signature of the head of the laboratory or test and only for the specified organizational sector. Before approval, it must be ensured that the changes are not relevant for the results.

The supplementary page of the method must not used to correct errors in the method or in cases of fundamental changes. In such case, a new version or analytical method must be drawn up and the author of the method consulted.

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