FAO SPECIFICATIONS AND EVALUATIONS

FOR PLANT PROTECTION PRODUCT

DICAMBA

3,6-dichloro-2-methoxy-benzoic acid

2001

FOOD AND AGRICULTURE ORGANIZATION *of* THE UNITED NATIONS

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FAO specifications are developed with the basic objective of ensuring that pesticides complying with them are satisfactory for the purpose for which they are intended so that they may serve as an international point of reference. The specifications do not constitute an endorsement or warranty of the use of a particular pesticide for a particular purpose. Neither do they constitute a warranty that pesticides complying with these specifications are suitable for the control of any given pest, or for use in a particular area. Owing to the complexity of the problems involved, the suitability of pesticides for a particular application must be decided at the national or provincial level.

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INTRODUCTION

FAO establishes and publishes specifications* for technical material and related formulations of plant protection products with the objective that these specifications may be used to provide an international point of reference against which products can be judged either for regulatory purposes or in commercial dealings.

Since 1999 the development of FAO specifications follows the **New Procedure**, described in the 5th edition of the " Manual on the development and use of FAO specifications for plant protection products" (FAO Plant Production and Protection Page No. 149). This **New Procedure** follows a formal and transparent evaluation process. It describes the minimum data package, the procedure and evaluation applied by FAO and the Experts of the "FAO Panel of Experts on Pesticide Specifications, Registration Requirements, Application Standards and Prior Informed Consent."
FAO Specifications now only apply to products for which the technical materials have been evaluated. Consequently from the year 2000 onwards the publication of FAO specifications under the **New Procedure** has changed. Every specification consists now of two parts namely the specifications and the evaluation report(s):

Part One: The Specification of the technical material and the related formulations of the plant protection product in accordance with chapter 4, 5 and 6 of the 5th edition of the "Manual on the development and use of FAO specifications for plant protection products".

Part Two: The Evaluation Report(s) of the plant protection product reflecting the evaluation of the data package carried out by FAO and the Panel of Experts. The data are to be provided by the manufacturer(s) according to the requirements of Appendix A, annex 1 or 2 of the "Manual on the development and use of FAO specifications for plant protection products" and supported by other information sources. The Evaluation Report includes the name(s) of the manufacturer(s) whose technical material has been evaluated. Evaluation reports on specifications developed subsequently to the original set of specifications are added in a chronological order to this report.

FAO Specifications under the **New Procedure** do not necessarily apply to nominally similar products of other manufacturer(s), nor to those where the active ingredient is produced by other methods of synthesis. FAO has the possibility to extend the scope of the specifications to similar products, but only when the Panel of Experts has been satisfied that the additional products are equivalent to those which formed the basis of the reference specification.

* Footnote: The publications are available on Internet under (http://www.fao.org/AG/AGP/AGPP/Pesticid/) or as hardcopy from the Plant Protection Information Officer.

PART ONE

SPECIFICATIONS

DICAMBA

DICAMBA INFORMATION
DICAMBA TECHNICAL MATERIAL
DICAMBA WATER SOLUBLE GRANULES
DICAMBA SOLUBLE CONCENTRATE

DICAMBA

INFORMATION

ISO common name

Dicamba (E-ISO, (m) F-ISO)

Synonyms

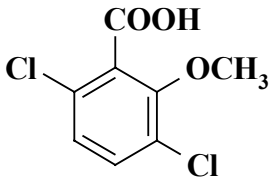
Dicamba (BSI, ANSI, WSSA), MDBA (JMAF)

Chemical names

IUPAC: 3,6-dichloro-*o*-anisic acid

CA: 3,6-dichloro-2-methoxy-benzoic acid

Structural formula



Molecular formula

$C_8H_6Cl_2O_3$ (acid)

Relative molecular mass

221.0 (acid)
266.1 (dimethylammonium salt)
259.1 (potassium salt)
243.0 (sodium salt)

CAS Registry number (acid)

1918-00-9

CIPAC number

85

DICAMBA TECHNICAL MATERIAL

FAO Specification 85/TC (2001)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturer whose name is listed in the evaluation report (85/2001). It should be applicable to relevant products of this manufacturer but it is not an endorsement of those products, nor a guarantee that they comply with the specifications. The specification may not be appropriate for the products of other manufacturers. The evaluation report (85/2001) as PART TWO forms an integral part of this publication.

1 Description

The material shall consist of dicamba, together with related manufacturing impurities, as grey to tan coloured lumps, flakes, granules or powder, free from visible extraneous matter and added modifying agents.

2 Active Ingredient

2.1 **Identity tests** (CIPAC method m/4177 – not yet published) (Note 1)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 **Dicamba content** (CIPAC HPLC method m/4177 – not yet published) (Note 1)

The dicamba content shall be declared (not less than 850 g/kg) and, when determined, the average measured content shall not be lower than the declared minimum content.

Note 1 Method available from the Pesticide Information Officer, FAO Plant Production and Protection Division or from the CIPAC secretariat, http://www.cipac.org

DICAMBA WATER SOLUBLE GRANULES

FAO Specification 85/SG (2001)

*This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturer whose name is listed in the evaluation report (*85/2001*). It should be applicable to relevant products of this manufacturer but it is not an endorsement of those products, nor a guarantee that they comply with the specifications. The specification may not be appropriate for the products of other manufacturers. The evaluation report (*85/2001*) as PART TWO forms an integral part of this publication.*

1 Description

The material shall consist of a homogeneous mixture of technical dicamba, complying with the requirements of FAO specification 85/TC (2001), in the form of dicamba sodium salt, together with carriers and any other necessary formulants. It shall be in the form of granules for application after disintegration and dissolution in water. The product shall be free-flowing, essentially non-dusty, and free from visible extraneous matter and hard lumps. The active ingredient shall be soluble in water. Insoluble carriers and formulants shall not interfere with compliance with clause 3.2.

2 Active Ingredient

2.1 **Identity tests** (CIPAC method m/4177 – not yet published) (Note 1)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 **Dicamba content** (CIPAC HPLC method m/4177–not yet published) (Note 1)

The dicamba content (expressed in terms of dicamba acid) shall be declared (g/kg) and, when determined, the content measured shall not differ from that declared by more than the following amounts:

Declared content, g/kg	Permitted tolerance
Above 25 up to 100	± 10 % of the declared content
Above 100 up to 250	± 6 % of the declared content
Above 250 up to 500	± 5 % of the declared content
Above 500 g/kg	± 25 g/kg
Note: each range includes the upper limit	

3 Physical properties

3.1 **pH range** (MT 75, CIPAC handbook F, page 205)

pH range: 5 to 10.

3.2 **Degree of dissolution and solution stability** (MT 179, CIPAC handbook H, page 307)

Residue of formulation retained on a 75 µm test sieve after dissolution in CIPAC Standard Water D at 30 °C ± 2°C:

Maximum: 2 % after 5 min.
Maximum: 2 % after 18 h.

3.3 **Persistent foam** (MT 47.2, CIPAC handbook F, page 152.) (Note 2)

Maximum: 30 ml after 1 min.

3.4 **Dustiness** (MT 171, gravimetric method, CIPAC handbook F, page 425)

Essentially non-dusty.

3.5 **Flowability** (MT 172, CIPAC handbook F, page 430)

At least 99% of the formulation shall pass through a 5 mm test sieve after 20 drops of the sieve.

4 Storage Stability

4.1 **Stability at elevated temperatures** (MT 46.3, CIPAC handbook J)

After storage at 54 ± 2 °C for 14 days, the determined average active ingredient content must not be lower than 97 %, relative to the determined average content found before storage (Note 3), and the formulation shall continue to comply with the clauses for pH range (3.1), degree of dissolution and solution stability (3.2), and dustiness (3.4).

Note 1 Method available from the Pesticide Information Officer, FAO Plant Production and Protection Division or from the CIPAC secretariat, http://www.cipac.org.

Note 2 The formulation should be tested at 1.4 % w/v.

Note 3 Analysis of the formulation, before and after the storage stability test, should be carried out concurrently to reduce the analytical error.

DICAMBA SOLUBLE CONCENTRATE

FAO Specification 85/SL (2001)

This specification, which is PART ONE of this publication, is based on an evaluation of data submitted by the manufacturer whose name is listed in the evaluation report (85/2001). It should be applicable to relevant products of this manufacturer but it is not an endorsement of those products, nor a guarantee that they comply with the specifications. The specification may not be appropriate for the products of other manufacturers. The evaluation report (85/2001) as PART TWO forms an integral part of this publication.

1 Description

The material shall consist of technical dicamba, complying with the requirements of FAO specification 85/TC (2001), in the form of dicamba dimethylammonium, potassium or sodium salt, dissolved in suitable solvents, together with any other necessary formulants. It shall be in the form of a clear or opalescent liquid, free from visible suspended matter and sediment, to be applied as a true solution of the active ingredient in water.

2 Active Ingredient

2.1 **Identity tests** (CIPAC method m/4177 – not yet published) (Note 1)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 **Dicamba content** (CIPAC method m/4177 – not yet published) (Notes1 and 2)

The dicamba content (expressed in terms of dicamba acid) shall be declared (g/kg or g/l at 20 ± 2°C) and, when determined, the content measured shall not differ from that declared by more than the following amounts:

Declared content, g/kg or g/l	**Permitted tolerance**
Above 25 up to 100	± 10 % of the declared content
Above 100 up to 250	± 6 % of the declared content
Above 250 up to 500	± 5 % of the declared content
Above 500 g/kg	± 25 g/kg
Note: each range includes the upper limit	

3 Physical properties

3.1 **pH range** (MT 75.2, CIPAC handbook F, page 206)

pH range: 5 to 10.

3.2 **Solution stability** (MT 41, CIPAC handbook F, page 131)

The formulation, after the stability test at 54 °C and following dilution (Note 3) with CIPAC standard water D and standing at 30°C ± 2°C for 18 h, shall give a clear or opalescent solution, free from more than a trace of sediment and visible solid particles. Any visible sediment or particles produced shall pass through a 45 µm test sieve.

3.3 **Persistent foam** (MT 47.2, CIPAC handbook F, page 152.) (Note 3)

Maximum: 30 ml after 1 min.

4 Storage stability

4.1 **Stability at 0 °C** (MT 39, CIPAC handbook F, page 128)

After storage at 0 ± 2 °C for 7 days, the volume of solid and/or liquid which separates shall not be more than 0.3 ml.

4.2 **Stability at elevated temperature** (MT 46.3, CIPAC handbook J)

After storage at 54 ± 2 °C for 14 days, the determined average active ingredient content must not be lower than 95 % relative to the determined average content found before storage (Note 4) and the formulation shall continue to comply with the clause for pH range (3.1).

Note 1 Method available from the Pesticide Information Officer, FAO Plant Production and Protection Division or from the CIPAC secretariat, http://www.cipac.org.

Note 2 If the buyer requires both g/kg and g/L, then in case of dispute the analytical results shall be calculated as g/kg.

Note 3 The formulation should be tested at 1.0 % w/v.

Note 4 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

PART TWO

EVALUATION REPORT(S)

DICAMBA

2001 Evaluation report based on joint submission of data from Syngenta, BASF and Gharda Chemicals (TC, SG, SL).

FAO SPECIFICATIONS AND EVALUATIONS FOR
PLANT PROTECTION PRODUCTS

DICAMBA

EVALUATION REPORT 85/2001

Explanation

The data for dicamba were evaluated in support of review of existing FAO specifications AGP:CP/59, 1975.

Dicamba is not under patent.

Dicamba has not been evaluated by the FAO/WHO JMPR and WHO/IPCS. It is to be reviewed by the European Commission as a "list 3" compound (2003 onwards); it is not under review by the US EPA;

The draft specification and the supporting data were provided by Syngenta Crop Protection AG (initially by Novartis Crop Protection), BASF and Gharda Chemicals Ltd. in 1999.

Uses

Dicamba is a selective systemic herbicide, absorbed by the leaves and the roots, with ready translocation throughout the plant via both the symplastic and apoplastic systems. It is used in agriculture (cereals, maize, sorghum, sugar cane, asparagus, perennial seed grasses, turf, pastures, rangeland and non crop land) against annual and perennial broad-leaved weeds and brush species (Pesticide Manual).

Identity of the active ingredient

ISO common name

Dicamba (E-ISO, (m) F-ISO)

Chemical name(s)

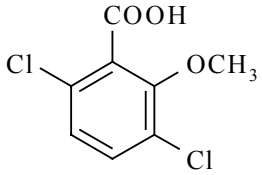
IUPAC: 3,6-dichloro-*o*-anisic acid

CA: 3,6-dichloro-2-methoxy-benzoic acid

Synonyms

Dicamba (BSI, ANSI, WSSA), MDBA (JMAF)

Structural formula



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$C_8H_6Cl_2O_3$ (acid)

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221.0 (acid)
266.1 (dimethylammonium salt)
259.1 (potassium salt)
243.0 (sodium salt)

CAS Registry number (acid)

1918-00-9

CIPAC number

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Identity tests

IR spectrum, HPLC-retention time

Physico-chemical properties of pure dicamba (*Table 1*)

Parameter	Value(s) and conditions	Purity %	Method reference
Vapour pressure	1.67 x 10^{-3} Pa at 25 °C (extrapolated)	99.2	OECD 104 EEC A4
Melting point, boiling point and/or temperature of decomposition	Melting point: 114 - 116 °C Boiling point: 230 °C Decomposition temperature: 230 °C	99.2 99.6	OECD 102 OECD 103
Solubility in water	6.6 g/l at 25 °C at pH 1.8 >250 g/l at pH 4.1 >250 g/l at pH6.8 >250 g/l at pH 8.2	99.6	OECD 105
Octanol/water partition coefficient	log P_{OW} = -0.55 at 25 °C at pH 5.0 log P_{OW} = -1.8 at 25 °C at pH 6.8 log P_{OW} = -1.9 at 25 °C at pH 8.9	99.6	OECD 107
Hydrolysis characteristics	No, or only very slight, degradation was observed at pH 5, 7 and 9 during 30 days at 25°C.		OECD 111 EPA 540/9-82-021
Photolysis characteristics	aqueous photolysis: DT_{50} = 14-50 d (latitude: Cincinnati, Ohio)		EPA N,161-2
Dissociation characteristics	pKa = 1.87	99.6	OECD 112

Chemical composition and properties of dicamba technical material (TC) (*Table 2*)

Manufacturing process, maximum limits for impurities ≥ 1 g/kg, 5 batch analysis data	Confidential information supplied by both manufacturers and held on file by FAO. Mass balances were 96.36 to 100.86% and percentages of unknowns were 0.0 to 3.6%.
Declared minimum dicamba content	850 g/kg
Relevant impurities ≥ 1 g/kg and maximum limits for them	None.
Relevant impurities < 1 g/kg and maximum limits for them:	None.
Stabilisers or other additives and maximum limits for them:	None.
Melting or boiling temperature range of the TC and/or TK	melting point 87– 108 °C

Toxicological summaries

Notes.

(i) The proposers confirmed that the toxicological and ecotoxicological data included in the summary below were derived from dicamba having impurity profiles similar to those referred to in the table above.

(ii) The conclusions expressed in the summary below are those of the proposers, unless otherwise specified.

Table 3. Toxicology profile of dicamba technical material, based on acute toxicity, irritation and sensitization.

Species	Test	Duration and conditions or guideline adopted	Result
Rat male Rat female	oral	acute	LD_{50} = 1879 mg/kg bw LD_{50} = 1581 mg/kg bw
Rabbit	dermal	24 h.	LD_{50} > 2000 mg/kg bw
Rat (dust)	inhalation	4 h.	LC_{50} = 6900 mg/m^3
New Zealand White rabbits	skin irritation	4 h.	Not irritant
New Zealand White rabbits	eye irritation	24 h.	Due to its acidic nature, dicamba is strongly irritant to the eye
Guinea pigs	skin sensitization	acute	Dicamba is not sensitising in the maximisation test (Magnusson and Kligman)

Table 4. Toxicology profile of dicamba technical material based on repeated administration (sub-acute to chronic)

Species	Test	Duration and conditions or guideline adopted	Result
Rats, Beagle dog	oral (sub-acute/sub-chronic toxicity)	rat: 90 d. dog: 1 y.	NOAEL = 239 to 342 mg/kg bw/d (rat), 52 mg/kg bw/d (dog) LOEL = 682 to 1000 mg/kg bw/d (rat)
Rat, Mouse	feeding, carcinogenicity	lifetime	in the combined chronic toxicity/carcinogenicity study, no carcinogenic potential was found up to highest tested dose of 3000 ppm.
Rat	feeding, multi-generation and reproduction	OECD 416	NOAL = 35 to 105 mg/kg bw/d LOEL = 105 to 347 mg/kg bw/d
Rabbit	teratogenicity and developmental toxicity	20 d.	no developmental toxicity or teratogenicity observed
Rat	delayed neurotoxicity		no potential expected from acute and sub-chronic studies

Mutagenicity profile of dicamba technical material based on in vitro and in vivo tests

The mutagenic potential of dicamba has been studied in various *in-vitro* and *in-vivo* test systems (Ames test, CHO cells, micronucleus). Based on these results, dicamba is not considered to be a mutagen.

Table 5. Ecotoxicology profile of dicamba technical material

Species	Test	Duration and conditions	Result
Daphnia magna (water flea)	acute toxicity	24 and 48 h. (static exposure)	EC_{50} = 110.7 mg/l
Rainbow trout Bluegill sunfish Sheepshead minnow	acute toxicity	96 h.(static exposure)	LC_{50} = 135 mg/l LC_{50} = 135 mg/l LC_{50} > 180 mg/l
Scenedesmus subspicatus alga	acute	96 h.	EC_{50} = 269 mg/l NOEC = 250 mg/l
Apis *mellifera* (honey bee)	acute oral, acute contact	72 h.	LD_{50} =>0.1 mg/bee
Bobwhite quail, Mallard duck	acute toxicity	acute	LD_{50} = 216 – 2009 mg/kg bw
Mallard duck	short-term toxicity	5 d.	LC_{50} > 10.000 mg/kg diet

Dicamba has not been evaluated by the WHO/PCS or the FAO/WHO JMPR.

The WHO/PCS hazard classification of dicamba is Class III, slightly hazardous.

Formulations and co-formulated active ingredients

The main formulation types available are SG and SL. In these formulations, dicamba is present in the form of a salt.

Dicamba may be co-formulated with a wide variety of other herbicides, such as 2,4-D or other phenoxy acids, sulfonylureas or triazines.

These formulations are registered and sold in many countries: Argentina, Australia, Belarus, Belgium, Bolivia, Brazil, Bulgaria, Canada, Chile, China, Czech Republic, Denmark, Dominican Republic, Estonia, France, Germany, Greece, Hungary, India, Indonesia, Italy, Iraq, Kazakhstan, Kenya, Korea, Latvia, Lithuania, Malaysia, Mexico, Moldavia, Netherlands, Norway, Panama, Saudi Arabia, Slovak Republic, South Africa, Spain, Serbia, Switzerland, Tanzania, Thailand, Trinidad, Tobago, Turkey, Ukraine, United Kingdom, USA, Uruguay, Uzbekistan, Zimbabwe.

Methods of analysis and testing

The analytical methods for the active ingredient (including identity tests) are CIPAC method 85.102/SL/M/2 or 3 (CIPAC Handbook H, p.128) or the more recent full CIPAC method m/4177 (not yet published but available from FAO Plant Production and Protection Division or the CIPAC secretariat). Dicamba is determined by IR spectroscopy in the earlier method or by reversed-phase HPLC, using UV detection at 280 nm and external standardisation, in the later method. An alternative HPLC method has been validated by Gharda Chemicals according to OECD and US EPA Guidelines for Good Laboratory Practice (GLP).

The analytical methods for determination of (non-relevant) impurities were based on reversed phase HPLC using UV detection at 280 nm and external standardisation.

Test methods for determination of physico-chemical properties of the technical active ingredient were OECD, EEC and EPA, while those for the formulations were CIPAC, as indicated in the specifications.

Physical properties

The physical properties, the methods for testing them and the limits proposed for the SL and SG formulations, comply with the requirements of the FAO Manual (5th edition).

Containers and packaging

No special requirements for containers and packaging have been defined but containers should comply with pertinent national and international transport and safety regulations.

Expression of the active ingredient

The active ingredient is expressed as dicamba (free acid).

Appraisal

The data for dicamba were evaluated in support of review of an existing FAO specification (AGP:CP/59 – 1975). Dicamba is not under patent.

Dicamba is a selective systemic herbicide, used in agriculture, mainly for the control of broadleaved weeds in various monocotyledonous crops. It is not patented and is registered in many countries throughout the world.

Dicamba, in the form of its salts, is highly water soluble and it is formulated (and co-formulated) as soluble concentrates (SL) or soluble granules (SG). In these formulations, dicamba is present as the dimethylamine, sodium or potassium salt but it is determined analytically as dicamba free acid and the content of active ingredient is expressed as the free acid. The proposers initially considered the SG to be a water dispersible granule (WG), and the CIPAC HPLC method was recorded as having been validated for the WG. However, the salt of the active ingredient is fully soluble in water and the "WG" is clearly an SG.

Dicamba is classified as slightly hazardous on the basis of its acute toxicity but, because it is a rather strong acid (pKa 1.9), the free acid is irritating to the eye.

Each of the proposers provided the meeting with confidential information on their manufacturing process and 5 batch analysis data on the technical materials, including all impurities present at > 1 g/kg and some at lower levels. The data submitted were in accordance with the requirements of the FAO Manual (5th edition) and supported the draft specifications.

The IR analytical method and the HPLC analytical method for the determination of the active ingredient in technical and formulated products have been collaboratively tested by CIPAC. The IR method has been published, the HPLC method is not yet published but has been accepted as full CIPAC method. In addition, there is a validated HPLC method from Gharda (method available from the Pesticide Information Officer, FAO Plant Production and Protection Division).

The meeting agreed that the reference profile of purity and impurities should be that of BASF and Syngenta, with a minimum purity of 850 g/kg, because it was supported by the most complete data on toxicology and ecotoxicology. No impurities were considered to be relevant. The meeting agreed that the technical material manufactured by Gharda was equivalent to that of BASF and Syngenta.

Recommendations

The meeting recommended that the specifications for TC, SL and SG should be adopted by FAO and that the reference profile (for the determination of equivalence) should be that submitted by BASF and Syngenta.

References

FAO Manual	Manual on Development and Use of FAO specifications for Plant Protection Products, 5th edition, January 1999, Rome.
CIPAC Handbook H.	Dobrat W and Martijn A (ed), Black Bear Press, Cambridge. 359 p.,1998.
WHO/PCS	The WHO recommended classification of pesticides by hazard and guidelines to classification 1998-1999, WHO/PCS/98.21/rev1, 1998.
Pesticide Manual	Tomlin C.D.S. (ed), The Pesticide Manual, 11th edition. British Crop Protection Council, 1997.
CIPAC Handbook F	Dobrat W and Martijn A (ed), Black Bear Press, Cambridge. 472p., 1995.