EUROPEAN COMMISSION
DIRECTORATE-GENERAL HEALTH & CONSUMER PROTECTION

Directorate E - Public, animal and plant health
Unit E1 Legislation relating to crop products and animal nutrition

Diquat

1688/VI/97-final

22.03.2001

Review report for the active substance **diquat**

Finalised in the Standing Committee on Plant Health at its meeting on 12 December 2000 in view of the inclusion of diquat in Annex I of Directive 91/414/EEC

1. Procedure followed for the re-evaluation process

This review report has been established as a result of the re-evaluation of diquat, made in the context of the work programme for review of existing active substances provided for in Article 8(2) of Directive 91/414/EEC concerning the placing of plant protection products on the market, with a view to the possible inclusion of this substance in Annex I to the Directive.

Commission Regulation (EEC) No 3600/92([1]) laying down the detailed rules for the implementation of the first stage of the programme of work referred to in Article 8(2) of Council Directive 91/414/EEC, as last amended by Regulation (EC) No 1972/99([2]), has laid down the detailed rules on the procedure according to which the re-evaluation has to be carried out. Diquat is one of the 90 existing active substances covered by this Regulation.

In accordance with the provisions of Article 4 of Regulation (EEC) No 3600/92, Zeneca Agrochemicals on 27 July 1993 and Barclay Chemicals on 27 June 1993 notified to the Commission of their wish to secure the inclusion of the active substance diquat in Annex I to the Directive.

In accordance with the provisions of Article 5 of Regulation (EEC) No 3600/92, the Commission, by its Regulation (EEC) No 933/94([3]), as last amended by Regulation (EC) No 2230/95([4]), designated the United Kingdom as rapporteur Member State to carry out the assessment of diquat on the basis of the dossiers submitted by the notifiers. In the same Regulation, the Commission specified furthermore the deadline for the notifiers with regard to the submission to the rapporteur Member States of the dossiers required under Article 6(2) of Regulation (EEC) No 3600/92, as well as for other parties with regard to further technical and scientific information; for diquat this deadline was 30 April 1995.

[1] OJ No L 366, 15.12.1992, p.10.

[2] OJ No L 244, 16.09.1999, p.41.

[3] OJ No L 107, 28.04.1994, p.8.

[4] OJ No L 225, 22.09.1995, p.1.

Both Zeneca Agrochemicals and Barclay Chemicals submitted each a dossier to the rapporteur Member State. Zeneca Agrochemicals was the main data submitter, with a dossier which did not contain substantial data gaps, taking into account the supported uses. The dossier from Barclay Chemicals was limited to data identifying the substance and to published general information on the substance. Information has furthermore been submitted by third parties, in particular a general analysis document prepared by the European Environmental Bureau.

In accordance with the provisions of Article 7(1) of Regulation (EEC) No 3600/92, the United Kingdom submitted on 2 April 1996 to the Commission the report of its examination, hereafter referred to as the draft assessment report, including, as required, a recommendation concerning the possible inclusion of diquat in Annex I to the Directive. Moreover, in accordance with the same provisions, the Commission and the Member States received also the summary dossier on diquat from Zeneca Agrochemicals, on 29 July 1996.

In accordance with the provisions of Article 7(3) of Regulation (EEC) No 3600/92, the Commission forwarded for consultation the draft assessment report to all the Member States as well as to Zeneca Agrochemicals being the main data submitter, on 24 June 1996.

The Commission organised an intensive consultation of technical experts from a certain number of Member States, to review the draft assessment report and the comments received thereon (peer review), in particular on each of the following disciplines:

- identity and physical /chemical properties ;
- fate and behaviour in the environment ;
- ecotoxicology ;
- mammalian toxicology ;
- residues and analytical methods ;
- regulatory questions.

The meetings for this consultation were organised on behalf of the Commission by the Biologische Bundesanstalt für Land und Forstwirtschaft (BBA) in Braunschweig, Germany, from September to December 1996.

The report of the peer review (i.e. full report) was circulated, for further consultation, to Member States and the main data submitter on 16 January 1997 for comments and further clarification.

In accordance with the provisions of Article 7(3) of Regulation (EEC) No 3600/92, the dossier, the draft assessment report, the peer review report (i.e. full report) and the comments and clarifications on the remaining issues, received after the peer review were referred to the Standing Committee on Plant Health, and specialised working groups of this Committee, for final examination, with participation of experts from the 15 Member States. This final examination took place from April 1997 to October 2000, and was finalised in the meeting of the Standing Committee on 12 December 2000.

The present review report contains the conclusions of this final examination; given the importance of the draft assessment report, the peer review report (i.e. full report) and the comments and clarifications submitted after the peer review as basic information for the final examination process, these documents are considered respectively as background documents A, B and C to this review report and are part of it.

These documents were also submitted to the Scientific Committee for Plants for separate consultation. The report of this Committee was formally adopted on 17 March 2000 (SCP/DIQUAT/Final-002 dated 5 April 2000[5]).

2. Purposes of this review report

This review report, including the background documents and appendices thereto, have been developed and finalised in support of the Directive 2001/21/EC concerning the inclusion of diquat in Annex I to Directive 91/414/EEC, and to assist the Member States in decisions on individual plant protection products containing diquat they have to take in accordance with the provisions of that Directive, and in particular the provisions of article 4(1) and the uniform principles laid down in Annex VI.

This review report provides also for the evaluation required under Section A.2.(b) of the above mentioned uniform principles, as well as under several specific sections of part B of these principles. In these sections it is provided that Member States, in evaluating applications and granting authorisations, shall take into account the information concerning the active substance in Annex II of the directive, submitted for the purpose of inclusion of the active substance in Annex I, as well as the result of the evaluation of those data.

In accordance with the provisions of Article 7(6) of Regulation (EEC) No 3600/92, Member States will keep available or make available this review report for consultation by any interested parties or will make it available to them on their specific request. Moreover the Commission will send a copy of this review report (not including the background documents) to all operators having notified for this active substance under Article 4(1) of this Regulation.

The information in this review report is, at least partly, based on information which is confidential and/or protected under the provisions of Directive 91/414/EEC. It is therefore recommended that this review report would not be accepted to support any registration outside the context of Directive 91/414/EEC, e.g. in third countries, for which the applicant has not demonstrated to have regulatory access to the information on which this review report is based.

3. Overall conclusion in the context of Directive 91/414/EEC

The overall conclusion from the evaluation is that it may be expected that plant protection products containing diquat will fulfil the safety requirements laid down in Article 5(1)(a) and (b) of Directive 91/414/EEC. This conclusion is however subject to compliance with the particular requirements in sections 4, 5, 6 and 7 of this report, as well as to the implementation of the provisions of Article 4(1) and the uniform principles laid down in Annex VI of Directive 91/414/EEC, for each diquat containing plant protection product for which Member States will grant or review the authorisation.

Furthermore, these conclusions were reached within the framework of the following uses, which were proposed and supported by the main data submitter:

- crop desiccation in potatoes;

[5] Opinion of the scientific Committee on Plants regarding the inclusion of diquat in Annex I to Council Directive 91/414/EEC concerning the placing of plant protection products on the market

- terrestrial weed control.

Extension of the use pattern beyond those described above will require an evaluation at Member State level in order to establish whether the proposed extensions of use can satisfy the requirements of Article 4(1) and of the uniform principles laid down in Annex VI of Directive 91/414/EEC.

With particular regard to residues, the review has established that the residues arising from the proposed uses, consequent on application consistent with good plant protection practice, have no harmful effects on human or animal health. Based on the FAO/WHO Standard European Diet (August 1994), excluding water and products of animal origin, total dietary intake does not exceed 35 % of the Acceptable Daily Intake (ADI). Additional intake from water and products of animal origin are not expected to give rise to intake problems.

The review has identified several acceptable exposure scenarios for operators, workers and bystanders, which require however to be confirmed for each plant protection product in accordance with the relevant sections of the above mentioned uniform principles.

The review has also concluded that under the proposed and supported conditions of use there are no unacceptable effects on the environment, as provided for in Article 4 (1) (b) (iv) and (v) of Directive 91/414/EEC, provided that certain conditions are taken into account as detailed in section 6 of this report.

4. Identity and Physical/chemical properties

The main identity and the physical/chemical properties of diquat are given in Appendix I.

The active substance shall comply with the FAO specification and there seem not to be reasons for deviating from that specification; the FAO specification is given in Appendix I of this report.

The review has established that for the active substance notified by the main data submitter Zeneca Agrochemicals, none of the manufacturing impurities considered are, on the basis of information currently available, of toxicological or environmental concern.

5. Endpoints and related information

In order to facilitate Member States, in granting or reviewing authorisations, to apply adequately the provisions of Article 4(1) of Directive 91/414/EEC and the uniform principles laid down in Annex VI of that Directive, the most important endpoints as identified during the re-evaluation process are set out under point 1 above. These endpoints are listed in Appendix II.

6. Particular conditions to be taken into account on short term basis by Member States in relation to the granting of authorisations of plant protection products containing diquat

(1) On the basis of the proposed and supported uses, the following particular issues have been identified as requiring particular and short term attention from all Member States, in the framework of any authorisations to be granted, varied or withdrawn, as appropriate:

– For the protection of aquatic organisms, risk mitigation measures must be applied where appropriate.

- For the protection of operators risk mitigation measures must be applied where appropriate.

(2) In addition, for the following uses, which have been evaluated, insufficient information has been submitted at this stage to demonstrate that the requirements of Article 4 (1) of the Directive are fulfilled. Therefore, the following issues have been identified as requiring particular and short-term attention from all Member States in the framework of any authorisations to be granted, varied or withdrawn, for the following uses, as appropriate:

For uses of pre-harvest desiccation of small grain crops Member States must ensure that dietary exposure arising from this use is acceptable.

(3) Finally, on the basis of the information available, it was concluded that aquatic weed control uses do not fulfil the requirements of Article 4 (1) of the Directive. Authorisations for these uses must therefore be withdrawn.

7. List of studies to be generated

No further studies were identified which were at this stage considered necessary in relation to the inclusion of diquat in Annex I under the current inclusion conditions.

Some endpoints however may require the generation or submission of additional studies to be submitted to the Member States in order to ensure authorisations for use under certain conditions. This may particularly be the case for

- For non-professional uses Member States shall ensure that sufficient information is submitted (such as specific field studies on amateur use for diquat containing products) to demonstrate that operator exposure is acceptable

- For use of pre-harvest desiccation of small grain crops Member States shall ensure that sufficient information is submitted to demonstrate that dietary exposure arising from this use is acceptable.

8. Information on studies with claimed data protection

For information of any interested parties, Appendix III gives information about the studies for which the main data submitter has claimed data protection and which during the re-evaluation process were considered as essential with a view to annex I inclusion. This information is only given to facilitate the operation of the provisions of Article 13 of Directive 91/414/EEC in the Member States. It is based on the best information available to the Commission services at the time this review report was prepared; but it does not prejudice any rights or obligations of Member States or operators with regard to its uses in the implementation of the provisions of Article 13 of the Directive 91/414/EEC neither does it commit the Commission.

9. Updating of this review report

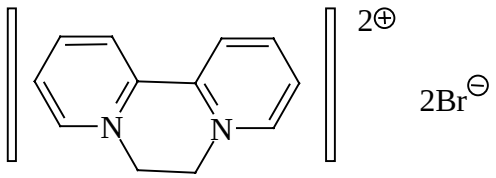
The technical information in this report may require to be updated from time to time in order to take account of technical and scientific developments as well as of the results of the examination of any information referred to the Commission in the framework of Articles 7, 10 or 11 of Directive 91/414/EEC. Such adaptations will be examined and finalised in the Standing Committee on Plant Health, in connection with any amendment of the inclusion conditions for diquat in Annex I of the Directive.

APPENDIX I

Identity, physical and chemical properties

DIQUAT

Common name (ISO)	Diquat (dibromide)
Chemical name (IUPAC)	9,10-dihydro-8a,10a-diazoniaphenanthrene ion (dibromide)
Chemical name (CA)	6,7-dihydrodipyrido[1,2-a:2'1'-c]pyrazinediium(8 & 9 Cl) (dibromide)
CIPAC No	55
CAS No	2764-72-9 (ion), 85-00-7 (dibromide)
EEC No	220-433-0 (ion), 201-579-4 (dibromide)
FAO SPECIFICATION	The material shall consist essentially of an aqueous solution of technical diquat dibromide together with related manufacturing impurities, and may contain small amounts of suspended matter, immiscible solvents and sediment, as specified. The diquat dibromide content shall be declared (not less than 467 g/l at 20 °C, and, when determined, the content obtained shall not differ from that declared by more than ± 25 g/kg. Impurities: For Ethylene Dibromide maximum of 10 mg/kg).
Minimum purity	95 % w/w
Molecular formula	$C_{12}H_{12}N_2$, $C_{12}H_{12}Br_2N_2$ (dibromide)
Molecular mass	184.2, 344 (dibromide)
Structural formula	



$C_{12}H_{12}Br_2N_2$ (diquat dibromide)

Melting point	Decomposes at approximately 325 °C
Boiling point	Decomposes
Appearance	Yellow crystalline solid
Relative density	1.61 $g \cdot cm^{-3}$
Vapour pressure	< 10^{-8} kPa at 25 °C
Henry's law constant	< $5 \cdot 10^{-12}$ $Pa \cdot m^3 \cdot mol^{-1}$
Solubility in water	pH 5.2: 712 g/l at 20 °C pH 7.2: 718 g/l at 20 °C pH 9.2: 713 g/l at 20 °C
Solubility in organic solvents	All at approximately 20 °C: - methanol : 25 g/l - acetone, dichlormethane, toluene, ethyl acetate, hexane : < 0.1 g/l,
Partition co-efficient (log P_{OW})	-4.6 at 20 °C
Hydrolytic stability (DT_{50})	pH 5: stable pH 7: stable pH 9: 8.3 % degradation after 30 d at 25 °C
Dissociation constant	--
Quantum yield of direct photo-transformation in water at ε >290 nm	3.84 x 10^{-4} at 302-303 nm
Flammability	Diquat dibromide technical is an aqueous solution containing approximately 20% w/w (minimum) diquat, it does not evolve flammable gases and the determination of the flammability of diquat dibromide as manufactured is therefore inappropriate
Explosive properties	The chemical structure of diquat does not include bond groupings which confer explosive properties.
UV/VIS absorption (max.)	204 nm, 272 nm, 310 nm
Photostability (DT_{50})	74 d at pH 7 (sunlight Florida) in water

APPENDIX II

END POINTS AND RELATED INFORMATION

DIQUAT

1 Toxicology and metabolism

Absorption, distribution, excretion and metabolism in mammals

Rate and extent of absorption:	Oral, < 10% in 96 h, rat
Distribution:	Highest levels in kidneys, gastro-intestinal tract, lung and liver, certain affinities to eye lens
Potential for accumulation:	Half live in blood 4 h, much longer in eye lens
Rate and extent of excretion:	Absorbed dose extensively excreted in urine and bile within 96 h.
Toxicologically significant compounds:	Diquat ion
Metabolism in animals:	Metabolism was limited, with <20% of the urinary residues (<1% of the administered dose) consisting of metabolites.

Acute toxicity

Rat LD_{50} oral:	LD_{50} (ion) = 214 - 222 mg/kg bw
Rat LD_{50} dermal:	LD_{50} (ion) > 424 mg/kg bw
Rat LC_{50} inhalation:	LC_{50} (ion) = 0.121 - 0.132 mg/l
Skin irritation:	Irritant (dibromide)
Eye irritation:	Irritant (dibromide) - classified on human data
Sensitisation (test method used and result):	Magnusson and Kligman - Positive (dibromide)

Short term toxicity

Target / critical effect:	Cataract (rat), kidney lesions (mouse) - (diquat ion)
Lowest relevant oral NOAEL / NOEL:	0.5 mg/kg bw/d, cataract 1 y dog - (diquat ion)
Lowest relevant dermal NOAEL / NOEL:	No repeat dose study by this route.
Lowest relevant inhalation NOAEL / NOEL:	No repeat dose study by this route.

Genotoxicity

No genotoxicity *in vivo*

Long term toxicity and carcinogenicity

Target / critical effect:	Cataract (rat), kidney lesions (mouse)
Lowest relevant NOAEL:	0.2 mg/kg bw/d, 2 y rat - (diquat ion)
Carcinogenicity:	Negative

Reproductive toxicity

Target / critical effect - Reproduction:	Negative
Lowest relevant reproductive NOAEL / NOEL:	1.4 mg/kg bw/d - general toxicity - rat (NOAEL)
Target / critical effect - Developmental toxicity:	Negative
Lowest relevant developmental NOAEL / NOEL:	1 mg/kg bw/d - NOAEL for maternal toxicity - rabbit

Delayed neurotoxicity

Not relevant

Other toxicological studies

None

Medical data

Published literature and company records report fatalities in cases of oral ingestion of concentrate i.e. not as a consequence of occupational exposure. A few cases of skin irritation and nosebleeds in manufacturing workers.

Summary

	Value	Study	Safety factor
ADI:	0.002 mg/kg bw (diquat ion),	2 y rat	100
AOEL systemic:	0.001 mg/kg bw/d (diquat ion), Corrected for oral absorption.	2 y rat (90 day end point)	100
AOEL inhalation:	not relevant		
AOEL dermal:	not relevant		
ARfD (acute reference dose):	not relevant		

Dermal absorption

About 1 %, human *in vivo*

2 Fate and behaviour in the environment

2.1 Fate and behaviour in soil

Route of degradation

Aerobic:

Mineralization after 100 days:	Microbial degradation has been demonstrated only in isolation due to strong adsorption to soil.
Non-extractable residues after 100 days:	Not relevant. See comment above.
Relevant metabolites above 10 % of applied active substance: name and/or code % of applied rate (range and maximum)	Not relevant. See comment above.

Supplemental studies

Anaerobic:	Relatively stable, withstands degradation
Soil photolysis:	No significant degradation in 32 d
Remarks:	Standard requirements are not applicable due to strong adsorption to soil.

Rate of degradation

Laboratory studies

DT_{50}lab (20 °C, aerobic):	No measurable degradation in soil under laboratory conditions after one year.
DT_{90}lab (20 °C, aerobic):	Not relevant. See comment above.
DT_{50}lab (10 °C, aerobic):	Not relevant. See comment above.
DT_{50}lab (20 °C, anaerobic):	Not relevant. See comment above.

Field studies (country or region)

DT_{50f} from soil dissipation studies:	DT_{50} = 10 - 20 y (UK), 1.2 - 3.6 y (US)
DT_{90f} from soil dissipation studies:	DT_{90} values were never reached
Soil accumulation studies:	Performed as part of US soil dissipation study - refer to detailed results. (16% of diquat applied remained in the soil after 11 years of annual application to the soil at 1 kg diquat/ha/yr)
Soil residue studies:	< 0.05 - 2.3 mg/kg (Denmark 32 sites) 0.11 mg/kg (maximum), 0.03 mg/kg (average) for various Western European Countries

Remarks	
e.g. effect of soil pH on degradation rate	The strong adsorption of diquat to soil precludes diquat degradation in soil being studied effectively by standard guideline methods. The strong adsorption also greatly reduces the rate of formation of degradation products to amounts that would not be detectable using standard methods. Soil microbial studies fulfil the scientific intent of demonstrating the intrinsic degradability of diquat.

Adsorption/desorption

	Following end points based on the results obtained from a soil residue study performed at 32 sites in Denmark. (Bewick *et al*, 1984)
K_f / K_{OC}	Koc values (32 soils in study) ranged from 32,000 to 7,900,000 (very strong adsorption in all the soils tested - with 31 of the soils having Koc values at least one order of magnitude greater than 5,000). Mean Koc value = 2,184,750 Median Koc value = 1,600,000
K_d	Kd values (32 soils in study) ranged from 1,200 to 92,000 (very strong adsorption in all the soils tested) Mean Kd value = 27,100 Median Kd value = 23,500
ph dependence	Not relevant

Mobility

Laboratory studies:

Column leaching:	Not relevant as all studies indicate that diquat is immobile.
Aged residue leaching:	Not relevant as all studies indicate that diquat is immobile.

Field studies:

Lysimeter/Field leaching studies:	Not relevant as all studies indicate that diquat is immobile.

Remarks:	Adsorption is correlated to clay content. Adsorption capacity is quantified by wheat

	bioasssay (SAC-WB). Most soils have a large excess in adsorption capacity. For very sandy soil exceedance may be a possibility following repeated high application rates.

2.2 Fate and behaviour in water

Abiotic degradation

Hydrolytic degradation:	No sterile hydrolysis at environmental pHs.
Relevant metabolites:	None
Photolytic degradation:	DT_{50} < 7 d (UK summer conditions)
Relevant metabolites:	None

Biological degradation

Ready biological degradability:	No, due to rapid adsorption by sediment or suspended solids.
Water/sediment study: DT_{50} water: DT_{90} water: DT_{50} whole system: DT_{90} whole system: Distribution in water / sediment systems (active substance) Distribution in water / sediment systems (metabolites)	DT_{50} = 12 - 24 hours. Aquatic biodegradation studies, (two water/sediment studies performed in the laboratory under aerobic or anaerobic conditions, and a field study performed in natural ponds in the US) show similar results. The primary route of dissipation of diquat from natural water is through very rapid adsorption onto sediment, or by adsorption onto plant material and/or suspended particulate matter which ultimately settle to the bottom of the pond or water course. The field study in natural ponds shows that diquat dispersion within and dissipation from water are both extremely rapid with difficulties in measuring these accurately. Substantial dissipation occurs after a few hours, with estimates of the DT50 for the partition to sediment ranging from <8 to 34 hours, with a mean of 12 to 24 hours. Diquat was stable withstanding degradation under the conditions of the aerobic and anerobic studies conducted in pond water and sand sediment.
Accumulation in water and/or sediment:	Not relevant as diquat dissipates very rapidly by adsorption onto sediment; plant material and/or suspended particulate matter which settle to the bottom of the pond or water course. There is no evidence of desorption of diquat back into the water in the relevant studies.

Degradation in the saturated zone

See above remarks.

Remarks:	None

2.3 Fate and behaviour in air

Volatility

Vapour pressure:	< 10^{-8} kPa at 25 °C
Henry's law constant:	5 · 10^{-12} Pa·m^3·mol^{-1}

Photolytic degradation

Direct photolysis in air:	Not relevant, due to low vapour pressure.
Photochemical oxidative degradation in air DT_{50}:	Not relevant, due to low vapour pressure.
Volatilisation:	Not relevant, due to low vapour pressure.

Remarks:	None

3 Ecotoxicology

Terrestrial Vertebrates

Acute toxicity to mammals:	LD_{50} (diquat ion - rat)= 214 - 222 mg/kg bw
Short term oral toxicity to mammals:	NOAEL 8.9mg /kg bw/d, 90 day rat (diquat ion) NOAEL 0.5 mg/kg bw/d, 1 y dog - (diquat ion)
Acute toxicity to birds:	LD_{50} = 83 mg /kg bw (diquat ion) *Anas platyrhynchos*
Dietary toxicity to birds:	LC_{50} = 721 ppm, 5 d study (diquat ion) *Coturnix japonica*
Reproductive toxicity to birds:	NOEC = 5 mg/kg (diquat ion)

Aquatic Organisms

Acute toxicity fish:	LC_{50} = 21 mg /l, 96 h static study (diquat ion) *Oncorhynchus mykiss* LC_{50} = 6.1 mg /l, 96 h flow through study (diquat ion) *Oncorhynchus mykiss*
Long term toxicity fish:	*Pimephales promelas* 34 day study on embryos/larvae - NOEC (larval weight) considered to be 0.12 mg diquat/litre based on mean measured concentration
Bioaccumulation fish:	Low risk of bioaccumulation
Acute toxicity invertebrate:	EC_{50} = 1.2 mg/l, 48 h study *Daphnia magna* (diquat ion)
Chronic toxicity invertebrate:	21-day LC50 was 0.16 mg/l based on nominal concentration *Daphnia magna* (diquat ion). 21-day NOEC = 0.125 mg/l based on nominal concentration.
Acute toxicity algae:	EC_{50} = 0.011 - 1.0 mg/l, 96 h study (diquat ion) *Psuedokirchneriella subcapitata (syn. Rhapidocellis subcapitata and Selenastrum capricornutum)*
Acute toxicity algae - study in presence of sediment	NOEC biomass = 320 μg/l with EbC50 of >320 μg/l. 72 hours (diquat ion).. NOEC growth rate = 320 μg/l with ErC50 of >320 μg/l. 72 hours (diquat ion). *Psuedokirchneriella subcapitata (syn. Rhapidocellis subcapitata and Selenastrum capricornutum)*

Chronic toxicity sediment dwelling organism:	NOEC > 100 mg diquat ion/kg^{-1} sediment (diquat ion). *Chironomus riparius*
Acute toxicity aquatic plants:	No data requirement set at time of review.

Honeybees

Acute oral toxicity:	LD_{50} = 13 µg /bee (diquat ion)
Acute contact toxicity:	LD_{50} = 60 µg /bee (diquat ion)

Other arthropod species

Test species	% Effect
Aphidius rhopalosiphi	An extended laboratory study. At full field rate (i.e. 5 l/ha) there was significant mortality of wasps in the treatment compared to the control. No adverse effects were noted on either fecundity or behaviour.
Coccinella septumpunctata.	Extended laboratory study. Bean plants treated with 'Reglone' at 5 l/ha (1000 g /ha) - equivalent to the maximum field rate. Larvae of *Coccinella septumpunctata* exposed to residues of the test substance. Corrected pre-imaginal mortality of *Coccinella septumpunctata* was 58%, mortality for the positive control was 78.9%. The reproduction rate was: • 640.9 eggs/female in the treatment • 255.3 eggs/female in the control. R value 151.0%. Results within the range of historical control variability. IOBC classification : slightly harmful
Trichogramma cacocecie	Laboratory study: exposed to 'Reglone' at 1000 g diquat ion/ha - equivalent to maximum field rate. Parisitisation capacity reduced by 58 %. Exposed adults reduced by 98%.
Chrysoperla carnea	Laboratory study: exposed to 'Reglone' at 1600 g diquat ion/ha. 96% mortality recorded in exposed larvae.

Pterosticus melanarius	Exposed to 'Reglone' at 1600g diquat ion/ha on loamy sand. No lethal or sublethal effects.
Pardosa spp.	Exposed to 'Reglone' at 1600g diquat ion/ha on loamy sand. No lethal or sublethal effects.

Earthworms

Acute toxicity:	LC_{50} = 130 mg as/kg soil 14 day (diquat ion) NOEC > 18 mg as/kg soil 14 day (diquat ion)

Soil micro-organisms

Nitrogen mineralization:	No significant effects up to 50.0 kg diquat/ha
Carbon mineralization:	No significant effects up to 720 kg as/ha

APPENDIX III

DIQUAT

List of studies for which the main submitter has claimed data protection and which during the re-evaluation process were considered as essential for the evaluation with a view to Annex I inclusion[6].

B.1 Identity, B.2 Physical and chemical properties, B.3 Data on application and further information, B.4 Proposals for classification and labelling, B.5 Methods of analysis

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 2, 7.2.1.1	Hendley P Upton BP Skidmore MW	1985	Diquat : Hydrolytic stability in water at pH 5, 7 and 9. RJ0452B 5C.1/15 GLP or GEP: yes Published: no	Germany 1994
IIA 2, 7.2.1.2	Moffatt F	1993	Diquat : Environmental half-life and quantum yield for direct photo-transformation in aqueous solution. RJ1545B 5C.1/17 GLP or GEP: yes Published: no	Germany 1994
IIA 2, 7.2.1.2	Tegala B Skidmore MW	1987	Diquat: an aqueous photolysis study. ICI Jealott's Hill Research Station Report No. RJ0613B 5C 1/16. GLP or GEP: yes Published: no	Germany 1994

[6] List based on a detailed analysis from the United Kingdom.

[2] Entries are based on information received from the Notifier. Neither the Commission nor the Member States are responsible for the completeness or validity of this information provided.

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 2	Wollerton C	1987 a	Pure diquat dibromide: Physico-chemical data file. RJ0582B 2B.2/7 GLP or GEP: yes Published: no	Finland 1994
IIA 2	Wollerton C	1987 b	Diquat manufacturing use product : Physico-chemical data file. RJ0534B 2B 2/6 GLP or GEP: yes Published: no	Finland 1994
IIA 4.2.3	Anderson L Earl M	1990	Diquat : Determination of residues in water at 0.1 ug/litre. ME/JAP/DLTL GLP or GEP: no Published: no	
IIA 4.2.4	Anderson L	1994	Paraquat and diquat. Validation of model to determine residues in air. RJ1659B GLP or GEP: yes Published: no	
IIA 4.2.1, IIA 4.2.2	Anderson L	1994	The determination of residues of paraquat and diquat in crops : A second derivative spectrophotometric method. RAM 252/01 GLP or GEP: yes Published: no	
IIA 4.2.2	Anderson L	1994	The determination of residues of paraquat and diquat in soil: a second derivative spectrophotometric method RAM 253/01 GLP : yesPublished: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 4.2.3	Anderson L	1994	The determination of residues of paraquat and diquat in water, milk, oils and other liquids : A second derivative spectrophotometric method with confirmatory method for water residues by HPLC. RAM 254/01 GLP: yes Published: no	
4.2.1, 4.2.2	Anderson L and Boseley A D:	1997	The determination of paraquat and diquat in crops and soil - a high performance liquid chromatography method (RAM 272/02) GLP or GEP: yes Published: no	
IIA 4.2.1	Coombe NB	1994	Validation of Zeneca Agrochemicals standard operating procedures for the analysis of diquat and paraquat residues in crops, soil and water containing both compounds. CEMR - 322 GLP or GEP: yes Published: no	
IIA 4.2.5	Earl M	1992 a	Diquat : Method validation data - determination of residues in milk. ME/JAP/DLTL1 GLP or GEP: yes Published: no	
IIA 4.2.5	Earl M	1992 b	Diquat : Method validation data. Determination of residues in animal tissues. ME/JAP/DLTL2 GLP : yes Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 4.2.5	Earl M	1993	Diquat : Determination of residues of diquat in animal tissues - a spectrophotometric method. RAM 008/01 GLP or GEP: no Published: no	
4.2.1, 4.2.2	James W	1996	Paraquat and diquat: validation of a method for the determination of residues in crops and soil: ZEN 0396 GLP or GEP: yes Published: no	
IIA 4.2.5	Thomas D Woollen BH	1994	Rapid methods for the semi-quantitative determination of paraquat and diquat in urine. CTL/R/1191 (tox series) GLP or GEP: yes Published: no	
IIA 4.2.5	Thomas D	1995	The determination of paraquat and diquat in biological fluids by reversed phase HPLC with UV detection. CT05-287 GLP or GEP: yes Published: no	
IIA 4.1	Thorndycraft MD	1992	The determination of diquat in aqueous concentrates and formulated materials by spectrophotometry. PAM 146 GLP : yes Published: no	

B.6 Toxicology and metabolism

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 5.3.2	Hodge MCE	1989 a	Diquat : 90 day feeding study in rats. CTL/P/1832 (revised) 4B.1/1 GLP or GEP: yes Published: no	Italy 1992 Germany 1994
IIA 5.5	Harling RJ Buist DP Gopinath C	1997	Diquat dibromide: Evaluation of potential carcinogenicity and chronic toxicity by prolonged dietary administration to rats. ICI 406/83763 (CTL/c/1327), Addendum report 2 (2 year data) GLP or GEP: no Published: no	
IIA 5.6.2	Hodge MCE	1989 b	Diquat : Teratogenicity study in the rabbit. CTL/P/2379 4B.4/10 GLP or GEP: yes Published: no	Finland 1994
IIA 5.6.2	Hodge MCE	1989 c	First amendment to Diquat : Teratogenicity study in the rabbit. CTL/P/2379 4B.4/10 GLP or GEP: yes Published: no	
IIA 5.6.2	Hodge MCE	1989 d	Second amendment to Diquat : Teratogenicity study in the rabbit. CTL/P/2379 4B.4/10 GLP or GEP: yes Published: no	Italy 1992
IIA 5.6.1	Hodge MCE	1990	Diquat : Multigeneration study in the rat. CTL/P/2462 4B.4/13 GLP or GEP: yes Published: no	Finland 1994
IIA 5.5	Hodge MCE	1992 a	Diquat : Two year feeding study in mice. CTL/P/3409 4B.2/7 GLP or GEP: yes Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 5.5	Hodge MCE	1992 b	First supplement to Diquat : Two year feeding study in mice. CTL/P/3409 4B.2/7 GLP or GEP: yes Published: no	
IIA 5.6.2	Hodge MCE	1994	Third amendment to Diquat : Teratogenicity study in the rabbit. CTL/P/2379 4B.4/10 GLP or GEP: yes Published: no	
IIA 5.3.2	Hopkins MN	1990	Diquat : 1 year feeding study in dogs. CTL/P/2596 4B.2/6 GLP or GEP: yes Published: no	Italy 1992 Finland 1994 Germany 1994
IIA 5.1.1	Johnston AM Jones C McCallum J Scott G	1991 a	The elimination of [^{14}C]-diquat in the rat following single oral administration (high dose level). IRI 7504 (CTL/C/2555) 4B.6/21 GLP or GEP: yes Published: no	
IIA 5.1.1	Johnston AM Mutch PJ Scott G	1991 b	The elimination of [^{14}C]-diquat in the rat following single oral administration (low dose level). IRI 7417 (CTL/C/2554) 4B.6/22 GLP or GEP: yes Published: no	
IIA 5.1.1	Johnston AM Jones C McCallum J Scott G	1991 c	The disposition of [^{14}C]-diquat in the rat. IRI 7480 (CTL/C/2553) 4B.6/23 GLP or GEP: yes Published: no	
IIA 5.2.1	McCall JC Robinson P	1990 a	Diquat dibromide : Acute oral toxicity to the rat. CTL/P/2999 3B.1/25 GLP or GEP: yes Published: no	Finland 1994 Germany 1994

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 5.2.2	McCall JC Robinson P	1990 b	Diquat dibromide : Acute dermal toxicity to the rat. CTL/P/2982 3B.1/26 GLP or GEP: yes Published: no	Finland 1994 Germany 1994
IIA 5.2.6	Rattray N Robinson P	1990	Diquat : Skin sensitisation to the guinea pig. CTL/P/2773 3B.1/24 GLP or GEP: yes Published: no	Finland 1994
IIA 5.2.2	Scott RC Walker M Mawdsley SJ	1991	First revision to diquat : *In vitro* absorption from technical concentrate ('Reglone 40') and spray strength solution through human skin. CTL/P/970 3E.2/9 GLP or GEP: yes Published: no	Italy 1992 Germany 1994
IIA 5.6.2	Wickramaratne GA	1989	Diquat : Teratogenicity study in the rat. CTL/P/2331 4B.4/9 GLP or GEP: yes Published: no	
IIA 5.6.2	Wickramaratne GA	1989	First supplement to diquat : Teratogenicity study in the rat. CTL/P/ GLP or GEP: yes Published: no	
IIA 5.6.2	Wickramaratne GA	1989	First amendment to diquat : Teratogenicity study in the rat. CTL/P/2331 4B.4/9 GLP or GEP: yes Published: no	
IIA 5.1.2	Williams SGP Cameron BD McGuire GM	1991	Identification of the major radioactive components in urine and faeces from rats following single oral administration of [^{14}C]-diquat. 7563 (CTL/C/2523) 4B.6/24 GLP or GEP: yes Published: no	

B.7 Residue data

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
6	Anderson L	1996	Diquat: storage stability of the residues in frozen carrot, cabbage, wheat grain and soil (final report) TMJ3575B GLP or GEP: yes Published: no	
6.5	Anderson L	1999	Diquat: residue levels in wheat and processed fractions generated during bread production from trials carried out in the United Kingdom during 1998. 98JH073 GLP or GEP: yes Published: no	
IIA 6.3.2i, IIA 6.3.2ii, IIA 6.3.2v	Anderson L Lant MS	1994 a	Diquat: Residue levels in carrots, lettuces and onions from trials carried out in Italy in 1993. RJ1730B GLP or GEP: yes Published: no	
IIA 6.3.1	Anderson L Lant MS Compagnon JM	1994 b	Diquat: Residue levels in grapes from trials carried out in France during 1993. RJ1681B GLP or GEP: yes Published: no	
IIA 6.3.4	Anderson L Elsworth S	1994 e	Diquat: Residue levels in linseed from trials carried out in the United Kingdom during 1993. RJ1727B GLP or GEP: yes Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 6.3.4	Anderson L Lant MS Renard C	1995 a	Diquat: Residue levels in sunflowers from trials carried out in France during 1993. RJ1734B GLP or GEP: yes Published: no	
IIA 6.3.1	Dick JP Taylor PS Bonfanti F	1995 a	Diquat: Residue levels in grapes from trials carried out in Italy during 1993. RJ 1800B GLP or GEP: yes Published: no	
IIA 6.3.1	Dick JP Taylor PS Bonfanti F	1995 b	Paraquat and diquat: Residue levels in olive fruit and oil from trials carried out in Italy during 1993. RJ1810B GLP or GEP: yes Published: no	
IIA 6.3.4	Earl M Anderson L	1989 b	Diquat: Residues in flax from trials carried out in Denmark in 1988. M4911B GLP or GEP: yes Published: no	
IIA 6.3.3	Earl M	1991 b	Diquat: Residues in peas from trials carried out in the United Kingdom during 1990. M5373B GLP or GEP: yes Published: no	
IIA 6.3.1	Earl M	1993 b	Diquat: Residue levels in bananas from trials carried out in Ecuador during 1992/1993. RJ1487B GLP or GEP: yes Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 6.3.1	Earl M	1994 a	Diquat: Residue levels in bananas from trials carried out in Costa Rica and Guatemala during 1992. RJ1534B GLP or GEP: yes Published: no	
IIA 6.3.3	Earl M Hall G	1994 b	Diquat: Residues levels in peas from trials carried out in the United Kingdom during 1992. RJ1502B GLP or GEP: yes Published: no	
IIA 6.2	French DA Leahey JP	1988	Diquat : Quantification and characterisation of radioactive residues in hen tissues and eggs. RJ0622B GLP or GEP: yes Published: no	
IIA 6.1.1	Heath J Leahey JP	1989	Diquat : Degradation on wheat. RJ0731B 4D.1/15 GLP or GEP: yes Published: no	
IIA 6.1.1	Heath J	1992	Diquat : Irradiation in aqueous solutions of glucose. RJ1199B GLP or GEP: yes Published: no	
IIA 6.3.1	Kennedy SH	1987 a	Paraquat/diquat: Residues in olives from trials carried out during 1987 in Spain. M4580B GLP or GEP: yes Published: no	
IIA 6.2	Lappin GJ Platt JA Davies DJ	1993	Diquat wheat chaff residues : Bioavailability study in the rat. CTL/P/4141 4B.6/25 GLP or GEP: yes Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 6.1.2	Lee SGK	1989	Diquat confined accumulation study in rotational crops. MEF-0026 GLP or GEP: yes Published: no	
IIA 6.3.2ii	Massey J	1987 a	Paraquat/diquat : Residues in onions from trials carried out in West Germany during 1984. M4415B GLP or GEP: yes Published: no	Germany 1994
IIA 6.3.2v	Massey J	1987 b	Paraquat/diquat: Residues in lettuce from trials carried out in West Germany during 1984. M4416B GLP or GEP: yes Published: no	Germany 1994
IIA 6.3.2i	Massey J	1987 c	Paraquat/diquat: Residues in carrots from trials carried out in West Germany during 1984. M4417B GLP or GEP: yes Published: no	Germany 1994
IIA 6.3.3	Massey JA	1987 d	Diquat: Residues in peas from trials carried out in Denmark during 1986. M4459B GLP or GEP: yes Published: no	Germany 1994

B.8 Environmental fate and behaviour

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 7.2.1.3.2	Cranor W Daly D	1988	Aerobic aquatic metabolism of ^{14}C-diquat. 36556 GLP or GEP: yes Published: no	
IIA 7.1.1.2.2	Anderson L Earl M	1990	Diquat: Residues in soil following desiccation of crops with 'Reglone' (interim). RJ0862B 5B.2/13 GLP : yes Published: no	
IIA 7.1.1.2.2	Dyson JS Kirsch O Stevens JEB	1995	Diquat: Long-term, soil trial at Goldsboro USA 1979-1991. 1. Trial description and crop measurements. TMJ3330B 5B.2/14 GLP or GEP: no Published: no	
IIA 7.1.1.2.2	Dyson JS Chapman P Farmer K	1995	Diquat: Long-term soil trial at Goldsboro USA 1979-1991 2. Fate of soil residues. TMJ3331B 5B.2/15 GLP or GEP: no Published: no	
IIA 7.1.1.2.2	Dyson JS Chapman P	1995	Diquat: Long-term high-rate trial, Frensham UK. TMJ3431B 5B.1/16 GLP or GEP: no Published: no	
IIA 7.1.2	Fergsuon RE Dyson JS Lane MCG	1994	Diquat: Adsorption and desorption properties in temperate soils. TMJ3310B 5B.1/36 GLP or GEP: no Published: no	
IIA 7.2.1.3.2	Johnston JJ	1988	Anaerobic aquatic metabolism of diquat. MEF-0072 GLP or GEP: yes Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 7.1.1.1.2	Joseph RSI Sidmore MW	1989	Diquat : Photolytic stability on soil surfaces. RJ0573B 5B.1/29 GLP or GEP: yes Published: no	
IIA 7.1.1.1	Rickets D	1997	Diquat - Microbial degradation Technical letter GLP : yes Published: no	

B.9 Ecotoxicology

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
8.2.7	Ashwell J A	1999	Diquat: Sediment toxicity test with *Chironomus riparius*. 98JH195 GLP or GEP: yes Published: no	
IIA 8.3.5	Edwards PJ Earl M Anderson L McIndoe E	1991	Diquat : Effect on plant cover and estimation of dietary exposure of birds following aerial desiccation of lentils. RJ1011B 5E.1(b)/1 GLP or GEP: yes Published: no	
IIA 8.3.3.1	Edwards PJ Coulson JM	1993	Diquat: Toxicity to the earthworm *Eisenia foetida* of a 200 g litre soluble concentrate. TMJ3048B GLP or GEP: no Published: no	

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 8.3.2, IIA 8.3.5	Gough HJ McMullin LC Jackson D White JS	1991	Diquat : Laboratory toxicity to the Carabid beetle *Pterostichus melanarius*, a Lycosid spider *Pardosa spp*. and larvae of the Green Lacewing *Chrysopa carnea* of residues of a 200 g/l aqueous formulation. RJ0922B 5E3(a)/1 GLP or GEP: yes Published: no	
8.3.2	Kuhner C	1997	Reglone - acute toxicity to the ladybird *Coccinella septempunctata* (Coleoptera, Coccinellidae) - extended laboratory test - 97030/01-NECS GLP or GEP: yes Published: no	
8.3.2	Longley M	1996	An extended laboratory test to determine the side-effects of the herbicide Reglone (YF7017A) a soluble concentrate formulation of diquat (220g/l) on adults of the parasitiod *Aphidius rhopalosiphi*. ZEN-96-7 GLP or GEP: yes Published: no	
IIA 8.3.4	Maas C	1990	Anleitung fur die Berichterstattung von versuchen uber Auswirkungen von Pflanzenschutzmitteln auf die Aktivitat der Bodenmikroflora nach Richtlinie Tiel Vi 1-1. BBA No. 30287 5B.3/18 GLP or GEP: no Published: no	
IIA 8.2.5	Rapley JH Hamer MJ	1991	Diquat: chronic toxicity to *Daphnia magna*. RJ0949B 5C.6/7 GLP or GEP: yes Published: no	Italy 1992 Finland 1994 Germany 1994

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 8.2.6	Smyth DV Tapp JF	1988	Diquat : Determination of toxicity to the green alga (*Selenastrum capticornutum*). BL/B/3271 5C.6/4 GLP or GEP: yes Published: no	Italy 1992 Finland 1994 Germany 1994
8.2.6	Smyth D Shillabeer N Magor SEl	1998	Diquat - toxicity to the green alga *Selenastrum capricornutum* in the presence of sediment. BL6471/B GLP or GEP: yes Published: no	
IIA 8.2.2.1	Surprenant DC	1987	The toxicity of diquat concentrate to Fathead minnow (*Pimephales promelas*) embryos and larvae. 981-0287-6113-120 (S-2912) 5C.4/20 GLP or GEP: yes Published: no	
IIA 8.2.1	Tapp JF Caunter JE	1988 a	Diquat: Determination of acute toxicity to Rainbow trout (*Salmo gairdneri*). BL/B/3336 5C.4/15 GLP or GEP: yes Published: no	Italy 1992 Finland 1994 Germany 1994
IIA 8.2.1	Tapp JF Caunter JE	1988 b	Diquat: Determination of acute toxicity to Mirror carp (*Cyprinus carpio*). BL/B/3337 5C.4/16 GLP or GEP: yes Published: no	Italy 1992 Finland 1994 Germany 1994
IIA 8.2.2.2	Tapp JF Sankey SA Caunter JE	1989	Diquat: Determination of the 21 day LC_{50} to Rainbow trout (*Salmo gairdneri*). BL/B/3488 5C.4/17 GLP or GEP: yes Published: no	Italy 1992 Germany 1994

Annex point/ reference number	Author(s)	Year	Title Source (where different from company) Company, Report No. GLP or GEP status (where relevant) Published or not	Reports[2] on previous use in granting national authorizations
IIA 8.3.5	Wilkinson W Cole JFH Everett CJ Riley D	1993 a	Diquat: Long-term ecological trial at Jealotts Hill UK 1964-90. 1. Description of the trial and observations on vegetation. TMJ3057B 5B.3/13 GLP or GEP: no Published: no	
IIA 8.3.5	Wilkinson W Cole JFH Gough HJ	1993 b	Diquat: Long-term ecological trial at Jealotts Hill UK 1964-90. 4. Effect on soil macroarthropods. TMJ3060B 5B.3/16 GLP or GEP: no Published: no	
IIA 8.3.3.2	WilkinsonW Edwards PJ	1993 c	Diquat: Long term ecological trial at Jealotts Hill UK 1964-90. 5. Effect on and residues in earthworms (*Lumbricidae*). TMJ3061B 5B.3/17 GLP or GEP: no Published: no	
8.3.2, 8.3.5	Wrzeciono SM	1991	A study of the effects of Reglone on *Trichogramma cacoceciae*. ICI-63B67A 5E.3(a)/1	Germany 1994