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**PHF1320 : HERBICIDE ANTI-DICOTYLEDONES LARGE SPECTRE POUR LE DESHERBAGE
DE POST-LEVÉE DES CÉRÉALES A PAILLE**

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RÉSUMÉ

Depuis plus de 4 ans, Philagro travaille au développement d'une nouvelle spécialité herbicide antidicotylédones de post-levée sur céréales à paille. Cette nouvelle préparation, nommée PHF1320, est une formulation de type OD contenant 40 g/l de diflufenicanil et 400 g/l de mécoprop-P. Elle s'utilise du stade 3 feuilles au stade 1 nœud des céréales.

L'association de ces deux substances actives aux modes d'action différents confère à cette spécialité un contrôle efficace d'un large spectre d'adventices. Les propriétés de ces 2 substances s'additionnent pour contrôler la véronique à feuilles de lierre et celle de perse, la pensée des champs et le gaillet et elles se complètent pour contrôler le coquelicot et la renouée liseron.

Cette formulation, de type dispersion dans l'huile, assure la sélectivité de la préparation PHF1320 sur un grand nombre de variétés de céréales et permet également à cette spécialité d'être associée avec de nombreux anti-graminées.

Mots-clés : herbicide, anti-dicotylédone, difufenican, mécoprop-P, céréale.

ABSTRACT

PHF1320: LARGE SPECTRUM HERBICIDE FOR THE CONTROL OF BROADLEEVES WITH A POST-EMERGENCE APPLICATION OF CEREALS

For more than 4 years, Philagro develops a new broadleaf herbicide for post-emergence in cereals. This new herbicide, PHF1320, is an OD-type formulation, containing 40 g/l of diflufenican and 400 g/l of mecoprop-P. It is used from the 3-leaf stage to the 1 node stage of the cereals.

The combination of these two active substances with different modes of action gives to this specialty an effective control of a wide spectrum of weeds. The properties of these two substances combine to control ivyleaf and Persian speedwells, fieldpansy and cleaver and synergize to control poppy and wild buckwheat.

This formulation, dispersion in oil type, provides the selectivity of PHF1320 on many cereal varieties and also allows this specialty to be associated with many anti-grasses.

Keywords: herbicide, broadleaf control, difufenican, mecoprop-P, cereal.

INTRODUCTION

A large part of the herbicides used to control broadleaves in cereals are based on three active ingredients diflufenican, ioxynil and bromoxynil. Lot of products have been withdrawn from the market since the active ingredient ioxynil has not been supported for a re-inclusion under annex II of regulation 1107/2009/CE. Others active substances with a wide range of efficacy and different mode of action have been or will be withdrawn.

As a result, a lot of herbicides products have disappeared and farmers have less solutions to control the weeds in their fields.

Weeds are responsible for important damages on the crops in various areas in the world. In France, these different damages include yield reduction, commercial losses, impact on organoleptic properties, toxicological issues for men and animals in case of toxics seeds, host of pests for the cereals, problems for mechanical harvest, ...

Because of all these undesirable effects, farmers and technicians recommend a strict limitation of weeds populations. This strict limitation of weeds population has also the advantage to be an help in order to prevent the occurrence of resistant broadleaves.

Thereby, four years of experimental trials have been undertaken with the preparation PHF1320, containing 40 g/l of diflufenican and 400 g/l of mecoprop-P to collect a solid knowledge on efficacy and selectivity of this preparation, and allow it registration.

MATERIAL AND METHODS

The preparation PHF1320 is a dispersion in oil (OD) containing 40 g/L of diflufenican and 656.8 g/l of mecoprop-P (ester form). It can be used on durum and winter wheat, spring and winter barley, rye and triticale. The recommended rate is 1.5 l/ha on all these cereals which is equivalent to 60 g/ha of diflufenican and 600 g/ha of mecoprop-P. Only one foliar application is proposed per crop per year. Water volume for the spray application per hectare can vary from 75 l/ha to 300 l/ha.

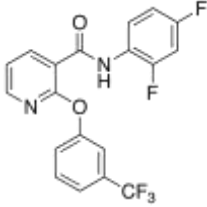
ACTIVE SUBSTANCES

Diflufenican (HRAC (Herbicide Resistance Action Committee) group F1)

Diflufenican belongs to the family of pyridine-carboxamides. It is an inhibitor of the enzyme phytoene desaturase which is involved in the synthesis pathway of carotenoids, chlorophylls protective pigment. Its site of action is the chloroplast: carotenoids being no longer produced, chlorophylls are no longer protected against photo-oxidation reactions and are rapidly destroyed. In the absence of chlorophyll, photosynthesis cannot take place and the plant dies.

Diflufenican is an herbicide that acts in pre and post-emergence of weeds. It acts against annual broadleaved plants. In pre-emergence, it enters in the weed by the stalk. In post-emergence, it penetrates mainly in young tissues. Its contact action is effective until the 4 leaves stage.

Table I : Diflufenican characteristics
(Caractéristiques du diflufenican)

Common name	diflufenican
IUPAC name	2',4'-difluoro-2-(α,α,α -trifluoro-m-tolyloxy)nicotinilide
CAS Registry No.	83164-33-4
Empirical formula	$C_{19}H_{11}F_5N_2O_2$
Molecular mass	394 g/mol
Structural formula	

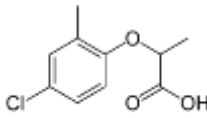
Mecoprop-P (HRAC group O)

Mecoprop-P belongs to the family of Phenoxy-carboxylic-acid. It acts like indole acetic acid (IAA), as synthetic auxins similar to that of endogenous auxin. The primary action of these compounds affects cell wall plasticity and nucleic acid metabolism. These compounds are thought to acidify the cell wall by stimulating the activity of a membrane-bound ATPase proton pump. The reduction in apoplastic pH induces cell elongation by increasing the activity of enzymes responsible for cell wall loosening.

Low concentrations of auxin-mimicking herbicides also stimulate RNA polymerase, resulting in subsequent increases in RNA, DNA, and protein biosynthesis. Abnormal increases in these processes presumably lead to uncontrolled cell division and growth, which results in vascular tissue destruction.

In contrast, high concentrations of these herbicides inhibit cell division and growth, usually in meristematic regions that accumulate photosynthate assimilates and herbicide from the phloem. Eventually this leads to the death of the plant.

Table II : Mecoprop-P characteristics
Caractéristiques du mécoprop-P

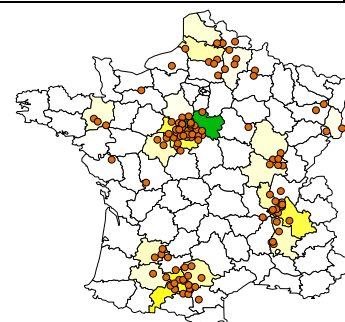
Common name	mecoprop-P
IUPAC name	(R)-2-(4-chloro-o-tolyloxy)-propionic acid
CAS Registry No.	16484-77-8
Empirical formula	$C_{10}H_{11}ClO_3$
Molecular mass	214.65 g/mol
Structural formula	

EXPERIMENTAL DETAILS

Location and period

Figure 1 : Location of trials/Implantation des essais

Data of trials carried out in France is available from the Maritime and the Mediterranean EPPO (European and Mediterranean Plant Protection Organization) climatic zones in a total of 12 trials in 2012-2013, 82 trials in 2013-2014, 79 trials in 2014-2015 and 18 trials in 2015-2016. For practical reasons, we present a selection of the most



representative results obtained during all those years.

All trials were officially recognized in accordance with the Principles of Good Experimental Practice (GEP).

Efficacy and selectivity trials were performed in compliance with the CEB method M013 (méthode d'essai d'efficacité pratique des herbicides destinés au désherbage des cultures de céréales à paille, blé, orge, avoine, seigle, triticale) (Curé *et al.*, 2012). They were implemented either in farmer field condition or in experimental facilities. Infestations of broadleaves and grasses were natural and homogenous.

Efficacy trials were implemented with semi-adjanted control and 3 replica.

Selectivity trials were implemented with an included single control randomized in each of the 4 blocks and harvested.

Application of products

REFERENCES

Table III : list of the products tested in the trials
liste des préparations appliquées dans les essais

	Ref.	Trade name	fomulation	content
Herbicide	REF 1	MEXTRA	EC	mecoprop-p 290 g/l + ioxynil 180 g/l
	REF 2	PICOTOP	SC	dichlorprop-p 600 g/l + picolinafen 20 g/l
	REF 3	FOXPRO D+	SC	bifenox 300 G/l + mecoprop p 260 G/l + ioxynil 92 G/l
	REF 4	ATLANTIS WG	WG	mesosulfuron-methyl 30 g/kg + iodosulfuron méthyl sodium 6 g/kg + méfenpyr-diéthyl 90 g/kg
Adjuvant	REF 5	ACTIROB B	EC	Esterified rape oil 842 g/l
Herbicide	REF 6	DEFI	EC	prosulfocarbe 800 g/l
	REF 7	MATIN EL	SC	isoproturon 500g/l
	REF 8	CHLORTOCID EL	SC	chlortoluron 500g/l

From the year 2014, we replaced REF 1 by REF 2, as REF 1 was foreseen at that time to be withdrawn from the market because it contains ioxynil.

Statistical analysis

Unless specified otherwise, trials use a randomized complete blocs design. As a consequence, individual data for every assessment have been submitted to ANOVA (Analysis of variance) followed by Newman & Keuls post-hoc test with 95% confidence.

In addition, when a group of at least 3 trials shares the same assessments and the same treatments, average is calculated and values are submitted to ANOVA as well, followed by Newman & Keuls post-hoc test with 95% confidence. This operation can be repeated for different sets of modalities and trials.

When considering an assessment, figures in summary tables followed by the same letter do not statistically differ one from another.

When an average value is based on 2 trials only, statistical analysis is irrelevant.

Color scale

Tables included use 2 color scales. One is for efficacy, one is for phytotoxicity. Both efficacy and phytotoxicity figures range from 0% to 100%.

Table IV : Efficacy and phytotoxicity scales / Echelles d'efficacité et de phytotoxicité

Efficacy color scale:														
0	15	40	50	60	70	75	80	85	87,5	90	92,5	95	97,5	100
Phytotoxicity color scale:														
0	2,5	5	7,5	10	15	20	25	30	40	50				

RESULTS

EFFICACY OF PHF1320 COMPARED TO REF 1 AND REF 2 (CF TABLE V)

Application in end of winter

Clearly, PHF1320 at 1.5 l/ha equals (mecoprop-p 290 g/l + ioxynil 180 g/l) at 2 l/ha on *Geranium dissectum* (GERDI), *Juncus bufonius* (IUNBU), *Polygonum persicaria* (POLPE), *Sinapis arvensis* (SINAR) and *Veronica arvensis* (VERAR) PHF1320 at 1.5 l/ha has a better efficacy than (dichlorprop-p 600 g/l + picolinafen 20 g/l) at 1.33 l/ha on *Anagallis arvensis* (ANGAR), *Polygonum convolvulus* (POLCO), *Sherardia arvensis* (SHRAR), *Stellaria media* (STEME) (2 trials), *Veronica hederifolia* (VERHE), *Veronica persicae* (VERPE) and *Viola arvensis* (VIOAR) (4 trials). We can also note that on one trial the assessment of the general efficacy of PHF1320 on all the weeds is far above (mecoprop-p 290 g/l + ioxynil 180 g/l). PHF1320 is effective on a wide range of weeds and could easily be used to control those weeds instead of REF 1, a product previously registered in France.

Considering the reference REF 2, PHF1320 equals the performance on *Anagallis arvensis* (ANGAR), *Aphanes arvensis* (APHAR), *Brassica napus* (BRNN), *Centaurea cyanus* (CENCY), *Cerastium glomeratum* (CERGL), *Galium aparine* (GALAP), *Juncus bufonius* (IUNBU), *Polygonum convolvulus* (POLCO), *Polygonum persicaria* (POLPE), *Rumex acetosella* (RUMAA), *Senecio vulgaris* (SENVU), *Sherardia arvensis* (SHRAR), *Sinapis arvensis* (SINAR), *Stellaria media* (STEME) *Veronica hederifolia* (VERHE), *Veronica persicae* (VERPE) and *Viola arvensis* (VIOAR).

Table V : Efficacy of PHF1320 compared to 2 references in application of end of winter (BBCH 28-30)

Efficacité du PHF1320 – comparée à 2 références, en sortie d'hiver (BBCH 28 à 30)

Pest Bayer code	DA-A	Pest density (plants/m ²)	PHF1320 1,5 l/ha	(mecoprop-p 290 g/l + ioxynil 180 g/l) 2 l/ha	(dichlorprop-p 600 g/l + picolinafen 20 g/l) 1,33 l/ha
ANGAR (2)	46-49	10 - 97	100 -	71 -	99 -
APHAR (2)	53-56	15 - 25	33 -	*	58 -
CENCY (2)	44-119	6 - 27	95 -	95 -	93 -
CERGL (1)	56	33	77 a-d	*	82 a-d
GALAP (3)	44-54	5 - 50	89 ab	92 a	88 ab
GERDI (1)	44	25	60 a	65 a	80 a
IUNBU (1)	46	20	98 a	98 a	100 a
MATCH (1)	44	15	65 a	93 a	90 a
PAPRH (3)	44-54	8 - 100	83 b	90	94 a
POLCO (1)	49	55	94 a	57 b	92 a
POLPE (1)	46	8	98 a	100 a	100 a
RUMAA (1)	49	20	63 a	*	53 a
SENVU (1)	54	20	84 ab	97 a	92 a
SHRAR (1)	49	28	97 ab	52 e	92 ab

Pest Bayer code	DA-A	Pest density (plants/m ²)	PHF1320 1,5 l/ha	(mecoprop-p 290 g/l + ioxynil 180 g/l) 2 l/ha	(dichlorprop-p 600 g/l + picolinafen 20 g/l) 1,33 l/ha
SINAR (2)	44-61	5 - 30	100 -	*	100 -
STEME (1)	44	15	98 a	96 a	95 a
STEME (2)	44-49	7 - 15	99 -	*	96 -
VERAR (1)	44	10	48 a	45 a	92 a
VERHE (3)	44-52	5 - 15	96 ns	*	93 ns
VERHE (7)	28-62	5 - 45	91 ns	85 ns	89 ns
VERPE (2)	48-49	5 - 15	98 -	68 -	91 -
VIOAR (3)	46-49	5 - 20	99 a	59 a	95 ab
VIOAR (8)	46-60	5 - 300	92 ab	*	96 a
TTTTT (1)	49	NA	95 a	50 e	95 a

*: no data available

Application in autumn

Efficacy results of PHF1320 at the dose rate of 1.5 l/ha is comparable to efficacy results of (mecoprop-p 290 g/l + ioxynil 180 g/l) 1 at 2l/ha on *Beta vulgaris* (BEAVA), *Galium aparine* (GALAP), *Geranium dissectum* (GERDI), *Lithospermum arvense* (LITAR), *Papaver rhoeas* (PAPRH), *Sinapis arvensis* (SINAR), *Stellaria media* (STEME), *Veronica persicae* (VERPE) and *Viola arvensis* (VIOAR) (see Table V).

On *Centaurea cyanus* (CENCY – 1 trial), the efficacy results of REF 1 at 2 l/ha are better than results obtained with PHF1320 at 1,5 l/ha. By contrast, on *Aphanes arvensis* (APHAR - 1 trial) the efficacy results of PHF1320 at 1,5 l/ha are better than results obtained with (mecoprop-p 290 g/l + ioxynil 180 g/l) at 2 l/ha.

Efficacy results obtained with PHF1320 at 1.5 l/ha are comparable to the efficacy results of (dichlorprop-p 600 g/l + picolinafen 20 g/l) at 1.33 l/ha on all weeds present in the field trial (see Table VI). On *Veronica hederifolia*, the results of PHF1320 are statistically better than the results of (dichlorprop-p 600 g/l + picolinafen 20 g/l) in 4 trials. From these results we can also notice that PHF1320 could bring a slight efficacy improvement for the control of *Alopecurus myosuroides*, better than the efficacy brought by (dichlorprop-p 600 g/l + picolinafen 20 g/l). In the same way, PHF1320 at 1.5 l/ha brings a numerically better (but not statistically different) control of *Papaver rhoeas* (PAPRH - 5 trials) and *Anthemis arvensis* (ANTAR - 1 trial). PHF1320 can perfectly control the presence in the field in autumn of *Aphanes arvensis* (APHAR), *Centaurea cyanus* (CENCY), *Galium aparine* (GALAP), *Matricaria chamomilla* (MATCH), *Papaver rhoeas* (PAPRH), *Sinapis arvensis* (SINAR), *Stellaria media* (STEME), *Veronica persicae* (VERPE), *Veronica hederifolia* (VERHE), *Anthemis arvensis* (ANTAR) and *Raphanus raphanistrum* (RAPRA).

Table VI : Efficacy of PHF1320 – (mecoprop-p 290 g/l + ioxynil 180 g/l) in autumn Efficacité du PHF1320 – (mecoprop-p 290 g/l + ioxynil 180 g/l) à l’automne

Pest Bayer code	PHF1320 1.5 l/ha	(mecoprop-p 290 g/l + ioxynil 180 g/l) 2.0 L/ha
APHAR (1 trial)	100 a	91,7 b
BEAVA (1 trial)	100 a	100 a
CENCY (2 trials)	40 bc	100 a
GALAP (5 trials)	94,2 a	89,6 a
GERDI (2 trials)	70 ab	45 abc
LITAR (1 trial)	100 a	100 a
MATCH (2 trials)	85	99,2
MATCH (3 trials)	84,4 a	
PAPRH (4 trials)	95,3 a	97,1 a
SINAR (3 trials)	100 NS	96,7 NS
STEME (3 trials)	100 NS	98,7 NS
VERHE (4 trials)	99,4 a	69 ab
VERHE (5 trials)	95,5 NS	
VERPE (1 trial)	100 a	100 a
VIOAR (1 trial)	100 a	100 a

Table VII : Efficacy of PHF1320 – (dichlorprop-p 600 g/l + picolinafen 20 g/l) in autumn Efficacité du PHF1320 – (dichlorprop-p 600 g/l + picolinafen 20 g/l) à l’automne

Pest Bayer code	PHF1320 1.5 l/ha	(dichlorprop -p 600 g/l + picolinafen 20 g/l) 1.33 l/ha
ALOMY (1 trial)	50 NS	33,3 NS
APHAR (2 trials)	97,3	95,7
CENCY (1 trial)	100 NS	100 NS
GALAP (4 trials)	85,3 a	83,2 a
MATCH (2 trials)	99,2	100
PAPRH (5 trials)	94 a	90,3 a
SINAR (1 trial)	100 NS	100 NS
STEME (1 trial)	100 NS	100 NS
VERHE (5 trials)	95,5 a	72,1 a
VERPE (3 trials)	99,9 a	100 a
VIOAR (4 trials)	100 a	100 a
LOLMU (3 trials)	0 b	0 b
AVEFA (1 trial)	0 NS	16,7 NS
ANTAR (1 trial)	97,5 a	88,7 a
RAPRA (1 trial)	100 NS	100 NS

EFFICACY OF ASSOCIATION OF PHF1320 AND GRASSKILLERS (CF TABLE VIII)

During Philagro’s experimental phase, PHF1320 have been included in practical program in order to assess the behavior of PHF1320 mix with the most common grasskillers. To highlight complementarity or antagonism phenomenons, PHF1320 have been tested at 0.75 l/ha and 1 l/ha according to the associated grasskiller (see table table VII).

In association with (mesosulfuron-methyl 30 g/kg + iodosulfuron méthyl sodium 6 g/kg) 0.3 kg/ha + adjuvant 1 l/ha, PHF1320 at 0.75 l/ha shows no antagonism on *Alopecurus myosuroides* and complementarity on *Lolium multiflorum*. This complementarity induces a control of *Lolium multiflorum* with an efficacy over 90% in 5 trials. It’s an increase of efficacy of 27%, a very interesting one for the success of the grasses control in a program.

The association of PHF 1320 1 l/ha with (prosulfocarbe 800 g/l) 3 l/ha leads to a significant improvement of the efficacy of the grasskiller on *Avena fatua* and *Lolium multiflorum* to reach an average efficacy over 80% on both grasses. It’s an increase of efficacy of respectively 40% and 10%. PHF1320 1 l/ha + (isoproturon 500g/l) 2.4l/ha shows, in that case too, a complementarity effect on the efficacy on *Avena Fatua*.

The use of PHF1320 1 l/ha mix with (chlortoluron 500g/l) 3.6 l/ha induces a significant increase in the efficacy level on *Avena fatua* compare with the efficacy of the grassekiller alone. This association doesn't show antagonist effect on the efficacy on *Lolium multiflorum*.

As previously investigated by Philagro, the efficacy of grasskillers used in foliar application in autumn or in end of winter is improved by the use of diflufenican based herbicides. The improvement is more significant when the grasskiller has a poor efficacy (Armengaud, 2013). This is clearly the case for the results presented in the table below.

Table VIII: Efficacy of association of PHF1320 and grasskillers
Efficacité des associations PHF1320 et anti-graminées

Adventices (number of trials)	PHF1320 0.75 l/ha	(mesosulfuron- methyl 30 g/kg + iodosulfuron méthyl sodium 6 g/kg) 0.3 kg/ha adjuvant 1 l/ha	PHF1320 0.75 l/ha (mesosulfuron-méthyl 30 g/kg + iodosulfuron méthyl sodium 6 g/kg) 0.3 kg/ha adjuvant 1 l/ha
<i>Alopecurus myosuroides</i> (2)	28	96	97
<i>Lolium multiflorum</i> (5)	0	65	92
	PHF1320 1 l/ha	(prosulfocarbe 800 g/l) 3 l/ha	PHF1320 1 l/ha (prosulfocarbe 800 g/l) 3 l/ha
<i>Avena fatua</i> (1)	0	40	80
<i>Lolium multiflorum</i> (5)	0	72	82
	PHF1320 1 l/ha	(isoproturon 500g/l) 2.4 l/ha	PHF1320 1 l/ha (isoproturon 500g/l) 2.4 l/ha
<i>Avena fatua</i> (1)	0	72	88
	PHF1320 1 l/ha	(chlortoluron 500g/l) 3.6 l/ha	PHF1320 1 l/ha (chlortoluron 500g/l) 3.6 l/ha
<i>Avena fatua</i> (1)	0	63	90
<i>Lolium multiflorum</i> (7)	0	67	67

SELECTIVITY OF PHF1320 COMPARED TO REFERENCES (CF TABLE IX)

In order to assess the phytotoxicity symptoms caused by PHF1320, 112 trials have been set up during three autumn in 2013, 2014 and 2015 and two ends of winter in 2014 and 2015. During those years, the phytotoxicity of PHF1320 and those of the references have been statistically equivalent and constant.

The results obtained with PHF1320 and the different references (REF = REF 1, REF 2 and REF 3) are therefore presented and summarized in table IX as a global range of phytotoxicity (min – max) per crop (all references included).

Table IX : Selectivity of PHF1320 compare to REF on cereals in autumn and end of winter
 Selectivité du PHF1320 comparée aux REF sur céréales à l'automne et en sortie d'hiver

Crop	PHF1320 1.5 l/ha	REF	PHF1320 1.5 l/ha	REF
	AUTUMN		END OF WINTER	
Durum wheat (24 trials)	2.5 – 8.8	0 - 7	7.5 – 10.5	0 - 7
Winter wheat (27 trials)	3.8 – 7.1	0 – 5	4.6 – 9.4	0.3 – 5.5
Spring wheat (2 trials)			5 – 23.8	1.5 – 21.3
Winter barley (29 trials)	4.1 – 10	0,4 – 3	5.4 – 6.6	1.2 – 4.5
Spring barley (10 trials)			5 – 5.3	5 – 5.7
Rye (9 trials)	0 - 11	0 – 1.5	2.5 – 8.5	0 - 6
Triticale (11 trials)	3 – 8.3	3.9 - 5	12.2 – 12.9	5.3 – 9.3

As often seen, the phytotoxicity of diflufenican based products on spring crops is higher than the symptoms observed on winter crop in the trials. Those symptoms are temporary and don't last after 30 days after application.

DISCUSSION & CONCLUSION

On efficacy

In autumn and in end of winter, PHF1320 is able to compete with the references on a wide range of broadleaves, including the major ones. The stage of developpement of the broadleaves is an important factor in the efficacy of PHF1320. Broadleaves treated at an early stage, will be easily controlled.

On selectivity

The selectivity of PHF1320 on durum wheat, winter wheat, spring wheat, winter barley, spring barley, rye and triticale has been investigated during severals years. PHF1320 is selective of all these crops following an application in autumn or in end of winter. As often seen, the phytotoxicity of diflufenican based products on spring crops is higher than the symptoms observed on winter crop in the trials. Those symptoms are temporary and don't last after 30 days after application.

The impact on yield, on transformation process, on adjacent crop and succeeding crops of PHF1320 and the references have otherwise been assessed. Once again, the assessments or differences between PHF1320 and the references were statistically equivalent.

ACKNOWLEDGEMENTS

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Méthode n° 013 compatible avec le guideline OEPP PP 1/93(2) - Association Française de Protection Des Plantes, Commission des Essais Biologiques – 2012 and following

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