(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
28 July 2016 (28.07.2016)

WIPO | PCT

(10) International Publication Number
WO 2016/115709 A1

(51) International Patent Classification:
A01N 43/42 (2006.01) *A01N 43/76* (2006.01) *A01N 47/36* (2006.01) *A01N 43/40* (2006.01) *A01N 43/54* (2006.01) *A01N 43/78* (2006.01) *A01N 39/04* (2006.01) *A01P 13/00* (2006.01)

(21) International Application Number:
PCT/CN2015/071348

(22) International Filing Date:
22 January 2015 (22.01.2015)

(25) Filing Language: English

(26) Publication Language: English

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report (Art. 21(3))*

WO 2016/115709 A1

(54) **Title**: A SAFE, ONE-SHOT RICE HERBICIDE COMPOSITION AND WEED CONTROL METHOD IN RICE FIELDS

(57) **Abstract**: The invention provides a one-shot herbicide composition comprising at least one herbicidal active ingredient selected from Groups 1 and/or Group 2, and at least one polymer where the composition is formulated into a granule or sprayable form, wherein Group 1 consists of bispyribac-sodium, penoxsulam, cyhalofop-butyl, metamifop, pyribenzoxim, azimsulfuron, flucetosulfuron, metazosulfuron, pyrimisulfan, profoxydim, pyriminobac-methyl, mefenacet, benzobicyclon, clomazone, fenoxaprop-P-ethyl, quinclorac, and propyrisulfuron; Group 2 consists of members of the protoporphyrinogen oxidase inhibitor group (PPO inhibitor herbicides) and the branched-chain amino acid inhibitor group (ALS or AHAS inhibitor herbicides) other than those listed in Group 1; and one or more of the at least one herbicidal active ingredient is/are dispersed in the at least one polymer. The invention also provides a weed control method in rice fields by applying the one-shot herbicide composition.

A Safe, One-Shot Rice Herbicide Composition and Weed Control Method in Rice Fields

Technical Field

The invention relates generally to agrochemical products, more specifically to an herbicide composition and preferably a one-shot rice herbicide composition used in both direct-seeded rice and transplanted rice. The invention further relates to a weed control method in rice fields by using the one-shot herbicide composition.

Background Art

The world's rice demand is projected to increase by 25% from 2001 to 2025 to keep pace with population growth. Meeting this ever-increasing rice demand in a sustainable way with shrinking resources therefore presents a great challenge. Part of this resource shortage comes from less available water for irrigation and labor force urbanization in developing countries that also happen to be major rice producers. Consequently direct-seeded rice, which eliminates the laborious process of planting and reduces the crop's water requirement, has taken the market share from transplanted rice in the past 20 years growing at a rate of approximately one percentage-point per year. However, weed control is more difficult in direct-seeded rice than in transplanted rice.

Weeds are a major source of yield reduction in rice production. Global yield losses due to pests have been estimated at ~40%, of which weeds have the highest loss potential (32%). The worldwide estimated loss in rice yield from weeds is around 10% of the total production. Ramzan reported yield reduction is higher in direct-seeded rice because of more weeds than transplanted rice (Ramzan M (2003) Evaluation of various planting methods in rice-wheat cropping systems, Punjab, Pakistan. *Rice Crop Report* 2003-2004, p. 4-5). Manifestly it becomes critical to develop an effective method of weed control in the race to keep up with rice demand.

Soil is essentially a big bank of weed seeds. Under suitable conditions, these seeds will germinate and grow, competing with rice for sunlight, nutrients, space, and water. The hot and wet rice cultivation environment lends itself especially well to weed proliferation. Weed factors that affect rice yield include weed species, weed density, and weed competition timing. Weed competition timing refers to the time window during which a weed species will have its maximum impact on crop yield due to its particular growth characteristics. To minimize this crop yield loss, one needs to know the time of the particular weed's emergence and the optimal time to remove it. Moreover, it is critical in general to control weeds within the first 40 days after direct seeding or transplanting while rice canopies have yet to develop large enough to block sunlight from reaching the soil surface and encouraging weed growth. In the absence of an effective weed control program, weeds will outgrow rice.

The main challenge to controlling all major weeds with a one-shot application is the aforementioned weed competition timing. For example, *Echinochloa crus-galli*, which could cause 70% of rice yield loss, needs to be controlled in the rice early season while *Heteranthera limosa*, 21% of yield loss, needs to be eliminated in the rice middle season. Combined with an herbicide's natural post-application tendency to quickly lose its activity owing to binding to the soil and/or degradation processes, it becomes hard for a single herbicide application to account for all major weed varieties during the entirety of a rice cycle which generally spans 110-160 days.

Chemical treatment remains one of the most effective means of controlling weeds. However, as of today, there is neither a single herbicide product nor a single herbicidal application that can control all major rice weeds. This is due to the following: First, a product containing a single herbicidal active ingredient cannot single-handedly control all major weeds because an effective herbicide is designed specifically to bond to a particular target receptor. Different weeds have different receptors. Therefore a single active ingredient cannot address different receptors equally well. Second, nor has there been reported a product containing 2 or 3 herbicidal active ingredients that can control all major weeds, due to the challenge in achieving a right combination of herbicides. For instance, cyhalofop-butyl, an excellent grass-control herbicide, often causes an antagonistic effect when mixed with other herbicides either in a premade product or in spray-tank mixing. Third, there lacks generally an approved set of guidelines for tank mixing with different herbicide products and it is therefore fully at the grower's own risk in doing so. The results are unpredictable. For example, 2,4-D, an effective broadleaf- and sedge-control herbicide, applied concurrently with the grass herbicide at 15 DAS would kill young rice seedlings that mature by the time it is otherwise traditionally used. Fourth, several major weeds have developed herbicide-resistance, which reduces the effectiveness of current weed control applications. *Echinochloa crus-galli,* for instance, has been reported to have developed resistance to almost all commonly used rice herbicides (azimsulfuron, bensulfuron-methyl, bispyribac-sodium, butachlor, cyhalofop-butyl, fenoxaprop-P-ethyl, flucetosulfuron, halosulfuron-methyl, imazamox, imazapic, imazethapyr, imazosulfuron, metamifop, molinate, penoxsulam, profoxydim, propanil, pyrazosulfuron-ethyl, pyribenzoxim, pyriminobac-methyl, quinclorac, quizalofop-P-tefuryl, and thiobencarb; see www.weedscience.org (International Survey of Herbicide Resistant Weeds)).

Consequently, rice growers today have no alternative to using multiple applications of an assortment of herbicides to minimize rice yield loss to weeds. To appreciate the magnitude of labor and time involved in these multiple applications, note that when applying a pre-emergence or post-emergence foliar application as most of these herbicides are, it is necessary to first drain off the water from the rice field before the herbicide application, wait 1-2 days after the application, and then re-introduce water to the field.

Recent books and papers have published approaches and recommendations on how to control weeds in rice fields, which improve marginally upon those of 30 years ago. The fundamental principles, such as pre-planting, pre-emergence, and post-emergence treatments, remain the same as before. For example, in the book of "Recent Advance in Weed Management", published as recently as 2014, authors Chauhan, Bhagirath S., Mahajan, Gulshan recommend using various pre- and post-emergence herbicides in direct-seeded rice because of a broad weed spectrum and high weed pressure (*Recent Advances in Weed Management*, Chauhan, Bhagirath S., Mahajan, Gulshan (Eds.), 2014, Springer, ISBN 978-1-4939-1019-9). Others also published articles describing pre-emergence and post-emergence herbicides treatments at different times are needed in rice (page 89, Chapter 7: Weed control in rainfed lowland rice, Kwesi Ampong-Nyarko and S.K. De Datta, *A Handbook for Weed Control*, 1991, IRRI (ISBN 971-22-0020-5)).

To the best of the author of the invention's knowledge, no patents, publications, books, and other sources of information in the literature have described a stand-alone rice herbicide product that can control all major weeds in rice fields at >90% efficacy for up to 42 days or longer with little rice phytotoxicity, particularly in direct-seeded rice, with a single application. The literature revolves chiefly around the conventional approach to controlling weeds in rice fields – a pre-emergent application followed by a second, and sometimes third herbicide or herbicides application. There is also no literature depicting any design of such a one-shot rice herbicide product. Part of the reason to this failure is because major weeds in rice – grasses, sedges, and broadleaf weeds – present their maximum competing strength with rice at different timings and the traditional approaches mentioned heretofore address weed management in a discrete manner.

On the other hand, since FMC first introduced sulfentrazone to the market in 1991 (Van Saun W. A. et al. *Proc. Br. Crop Prot. Conf-Weeds*, 1991, **1**, 77), it and other companies including DuPont, Tenkoz, Inc., Wilbur-Ellis, and Bonide Products, Inc., have produced herbicide products containing sulfentrazone. However, no labels on any such products make mention of use in rice. In fact, every such product's label warns that rice cannot be replanted in the same soil for at least 10 months from the product's last application. This is because sulfentrazone products from the prior art, acting as either pre-emergence or early post-emergence applications, cause unacceptable phytotoxicity in rice.

Summary of Invention

Technical Problem

Rice production yield is maximized when all weeds are controlled. Yet there is no rice herbicide product, much less a single rice herbicide application, that controls all major weeds in rice fields, particularly in direct-seeded rice, with satisfactory results.

Although weeds may be controlled through multiple applications at different times, with each application's composition varying from one to the next, this overcomplicated and inefficient process is becoming progressively less feasible with a diminishing labor force and increasing labor costs in most rice farming regions.

Therefore, there is a necessity to develop such a one-shot rice herbicide composition and a weed control method, which controls all major weeds in rice fields at >90% efficacy for at least 40 days after application with virtually no rice phytotoxicity.

Solution to Problem

In view of the foregoing disadvantages inherent in the current known products and applications that make farmers' practices less efficient, the invention provides a one-shot rice herbicide composition and a weed control method that significantly saves time and labor with a performance better than or equal to that required by traditional multiple applications.

In one aspect, the invention provides an herbicide composition comprising at least one herbicidal active ingredient selected from Group 1 and/or Group 2, and at least one polymer where the composition is formulated into a granule or sprayable form, wherein

Group 1 consists of bispyribac-sodium, penoxsulam, cyhalofop-butyl, metamifop, pyribenzoxim, azimsulfuron, flucetosulfuron, metazosulfuron, pyrimisulfan, profoxydim, pyriminobac-methyl, mefenacet, benzobicyclon, clomazone, fenoxaprop-P-ethyl, quinclorac, and propyrisulfuron;

Group 2 consists of members of the protoporphyrinogen oxidase inhibitor group (PPO inhibitor herbicides) and the branched-chain amino acid inhibitor group (ALS or AHAS inhibitor herbicides) other than those listed in Group 1;

the polymer is made of a cellulose type molecule selected from cellulose acetate, cellulose triacetate, cellulose propionate, cellulose acetate propionate, cellulose acetate butyrate, cellulose nitrate, cellulose sulfate, methylcellulose, ethylcellulose, ethyl methyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, hydroxyethyl methyl cellulose, hydroxypropyl methyl cellulose, ethyl hydroxyethyl cellulose, carboxymethyl cellulose, croscarmellose sodium, and hemicellulose, or mixtures thereof; and

one or more of the at least one herbicidal active ingredient is/are dispersed in the at least one polymer.

The invention also relates to a rice herbicide composition comprising at least one active ingredient selected from Group 1 and at least one active ingredient selected from Group 2.

In another aspect, the invention provides a method of weed control in rice fields, comprising applying the herbicide composition of the invention in the rice fields.

Advantageous Effects of the Invention

The design of our one-shot herbicide composition according to the invention completely departs from the concepts and approaches of the prior art, and in doing so provides a product primarily developed for the purpose of "one shot to control all major weeds" which is not anticipated, rendered obvious, suggested, or even implied by any of the prior art rice herbicides and their applications, either alone or in any combination thereof.

To that end, the present invention generally comprises a two-way or three-way herbicide mixture with a different mode of action for each active ingredient. The total concentration of the accumulated amounts of the active ingredients controlled-released over the first 5-7 days is capable of killing all major weeds that have already emerged. The concentration of the herbicides, already released plus continuously being released, forms a weed-control barrier that prevents the emergence of new weeds. This novel design – killing already emerged weeds first and then preventing the emergence of new weeds in a continuous mode – sets this invention apart from any prior art.

Other objects and advantages of the invention will become obvious to one skilled in the art in view of the following descriptions and it is intended that these objects and advantages are within the scope of the invention.

Brief Description of the Drawings

Figure 1 shows a schematic presentation of rice and weed root zones.

Figure 2 shows the rice injury index caused by sulfentrazone with different 24h release rates.

Description of the Embodiments

It has thus been outlined, rather broadly, the more important features of the invention in order that detailed description thereof may be better understood and in order that the present contribution to the art may be better appreciated. There are additional features of the invention that will be described hereinafter and that will form the subject matter of the claims appended hereto.

In this respect, before explaining at least one embodiment of the invention in detail, it is to be understood that the invention is not limited in its composition, ratio, formulation technique, formulation type (e.g., sprayable or granule), application, crop or non-crop to the details of the following description or to the examples, pictures, or drawings. The invention is capable of other embodiments and of being made, practiced, and carried out in various ways. Also, it is to be understood that the phraseology and terminology employed herein are for the purpose of the description and shall not be regarded as limiting.

Embodiments and examples are provided herein for the purpose of completeness and clearness of this disclosure and will convey the scope of the invention to those skilled in the art.

The invention may be embodied in many different forms and shall not be construed as limited to only the embodiments or examples set forth herein; rather, these embodiments or examples are provided so that this disclosure will satisfy applicable legal requirements. As used in the specification and in the appended claims, the singular forms of “a”, “an”, and “the” include plural referents unless the context clearly indicates otherwise.

Rice Herbicide Product

This invention is a novel one-shot herbicide composition designed to provide control of all (or at least most of) major weeds with >90% efficacy for up to 42 days or longer in both transplanted and wet direct-seeded rice fields for both hot-climate and temperate rice-cultivating regions while causing less than 15% phytotoxicity, if any, with the rice recovering within 14 days.

According to the invention, an herbicide composition comprises at least one herbicidal active ingredient selected from Group 1 and/or Group 2, and at least one polymer where the composition is formulated into a granule or sprayable form, wherein

Group 1 consists of bispyribac-sodium, penoxsulam, cyhalofop-butyl, metamifop, pyribenzoxim, azimsulfuron, flucetosulfuron, metazosulfuron, pyrimisulfan, profoxydim, pyriminobac-methyl, mefenacet, benzobicyclon, clomazone, fenoxaprop-P-ethyl, quinclorac, and propyrisulfuron;

Group 2 consists of members of the protoporphyrinogen oxidase inhibitor group (PPO inhibitor herbicides) and the branched-chain amino acid inhibitor group (ALS or AHAS inhibitor herbicides) other than those listed in Group 1;

the polymer is made of a cellulose type molecule selected from cellulose acetate, cellulose triacetate, cellulose propionate, cellulose acetate propionate, cellulose acetate butyrate, cellulose nitrate, cellulose sulfate, methylcellulose, ethylcellulose, ethyl methyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, hydroxyethyl methyl cellulose, hydroxypropyl methyl cellulose, ethyl hydroxyethyl cellulose, carboxymethyl cellulose, croscarmellose sodium, and hemicellulose, or mixtures thereof; and

one or more of the at least one herbicidal active ingredient is/are dispersed in the at least one polymer.

In the herbicide composition of the invention, the at least one herbicidal active ingredient can be selected from either Group 1 or Group 2. In a prefered embodiment, the herbicide composition of the invention comprises at least two herbicidal active ingredients selected from either Group 1 or Group 2. Prefereably, the herbicide composition of the invention comprises at least one herbicidal active ingredient selected from Group 1 and at least one herbicidal active ingredient selected from Group 2.

In another preferred embodiment, the herbicide composition comprises at least three herbicidal active ingredients selected from Group 1 and Group 2.

Examples of the PPO inhibitor herbicides of Group 2 include, but not limited to, acifluorfen-sodium, bifenox, butafenacil, carfentrazone-ethyl, cinidon-ethyl, flufenpyr-ethyl, flumiclorac-pentyl, flumioxazin, fluoroglycofen-ethyl, fluthiacet-methyl, fomesafen, lactofen, oxadiargyl, oxadiazon, oxyfluorfen, pentoxazone, pyraclonil, pyraflufen-ethyl, sulfentrazone, saflufenacil, and the like.

Examples of the ALS or AHAS inhibitor herbicides of Group 2 include, but not limited to, amidosulfuron, bensulfuron-methyl, chlorimuron-ethyl, chlorsulfuron-ethyl, cinosulfuron, cloransulam-methyl, cyclosulfamuron, diclosulam, ethametsulfuron-methyl, ethoxysulfuron, flazasulfuron, florasulam, flucarbazone-sodium, flupyrsulfuron-methyl-sodium, flumetsulam, foramsulfuron, halosulfuron-methyl, imazamethabenz-methyl, imazamox, imazapic, imazapyr, imazaquin, imazethapyr, imazosulfuron, iodosulfuron-methyl-sodium, iofensulfuron, ipfencarbazone, mesosulfuron-methyl, methiopyrisulfuron, metosulam, metsulfuron-methyl, monosulfuron, nicosulfuron, orthosulfamuron, oxasulfuron, primisulfuron-methyl, propoxycarbazone-sodium, prosulfuron, pyrazosulfuron-ethyl, pyriftalid, pyrithiobac-sodium, pyroxsulam, rimsulfuron, sulfometuron-methyl, sulfosulfuron, thiencarbazone-methyl, thifensulfuron-methyl, triasulfuron, tribenuron-methyl, trifloxysulfuron-sodium, triflusulfuron-methyl, tritosulfuron, zuomihuanglong, and the like.

Such an abovementioned composition preferably contains at least one herbicidal active ingredient from Group 1 to control weeds of the grass family, such as *Echinochloa crus-galli*, that will have already emerged by the time of application. Some of the herbicides in Group 1 are also preferred choices for controlling already emerged sedges and/or broadleaf weeds.

In addition, the composition preferably includes at least one herbicidal active ingredient selected from the following, which is chemically different from already included: azimsulfuron (Group 1), benzobicyclon (Group 1), clomazone (Group 1), flucetosulfuron (Group 1), metazosulfuron (Group 1), carfentrazone-ethyl (PPO inhibitor of Group 2), sulfentrazone (PPO inhibitor of Group 2), saflufenacil (PPO inhibitor of Group 2), bensulfuron-methyl (ALS or AHAS inhibitor of Group 2), chlorimuron-methyl (ALS or AHAS inhibitor of Group 2), cinosulfuron (ALS or AHAS inhibitor of Group 2), cyclosulfamuron (ALS or AHAS inhibitor of Group 2), ethoxysulfuron (ALS or AHAS inhibitor of Group 2), halosulfuron-methyl (ALS or AHAS inhibitor of Group 2), imazosulfuron (ALS or AHAS inhibitor of Group 2), orthosulfamuron (ALS or AHAS inhibitor of Group 2), pyrazosulfuron-ethyl (ALS or AHAS inhibitor of Group 2), or imazamox (ALS or AHAS inhibitor of Group 2). This is to control weeds of the sedge and broadleaf families, such as *Fimbristylis miliacea* and *Monochoria vaginalis*, that have either already emerged or are just emerging. Some of these herbicides can also control already emerged or emerging grass weeds.

In another preferred embodiment, the herbicide composition comprises sulfentrazone, penoxsulam, and clomazone as the herbicidal active ingredients.

In another embodiment, the herbicide composition comprises only one herbicidal active ingredient selected from either Group 1 or 2. Such a product is used in combination with other products for greater flexibility.

The herbicide composition according to the invention is preferably formulated as granule.

In the case of one herbicidal active ingredient in the composition according to the invention, the herbicidal active ingredient can come from either Group 1 or 2. The weight percentage (%) of the herbicidal active ingredient in a granule composition ranges from 0.01% – 14%.

In the case of two herbicidal active ingredients in the composition according to the invention, the two herbicidal active ingredients preferably come from each of the Groups. The two herbicidal active ingredients can also be selected from the same Group, either Group 1 or Group 2. As a prefered embodiment, the composition may comprise clomazone, and at least one herbicidal active ingredient selected from Group 1 other than clomazone. The weight percentage (%) of each of the two herbicidal active ingredients in a granule composition ranges from 0.01% – 13.99% and jointly ranges from 0.02% – 14%.

In the case of three herbicidal active ingredients in the composition according to the invention, the three herbicidal active ingredients consist of at least one member from each of the two Groups. It is preferable to select two members from Group 1 and one member from Group 2. As a prefered embodiment, the composition may comprise clomazone, at least one herbicidal active ingredient selected from Group 1 other than clomazone, and at least one herbicidal active ingredient selected from Group 2. The weight percentage (%) of each of the three herbicidal active ingredients in a granule composition ranges from 0.01 – 13.98% and jointly ranges from 0.03 – 14%.

In the preferred composition case according to the invention, the three individual active ingredients are sulfentrazone, penoxsulam, and clomazone, where individual active ingredient ranges from 0.01 – 4%, 0.01 – 8%, and 0.01 – 13.98%, respectively and jointly ranges from 0.03 – 14%.

The herbicide composition according to the invention can also be formulated into a sprayable formulation type, such as but not limited to, wettable powder (WP) or water–dispersible granules (WDG).

In the case of one herbicidal active ingredient in a sprayable formulation type such as WP or WDG, the active ingredient can come from either Group 1 or 2. The weight percentage (%) of the active ingredient in the final formulated composition ranges from 0.01% – 25%.

In the case of two herbicidal active ingredients in a sprayable formulation type such as WP or WDG, the two herbicidal active ingredients preferably come from each of the Groups. The two herbicidal active ingredients can also be selected from the same Group, either Group 1 or Group 2. The weight percentage (%) of each of the two herbicidal active ingredients in the final formulated composition ranges from 0.01% –

24.99% and jointly ranges from 0.02% – 25%.

In the case of three herbicidal active ingredients in a sprayable formulation type such as WP or WDG, the three active ingredients consist of at least one member from each of the two Groups. It is preferable to select two members from Group 1 and one member from Group 2. The weight percentage (%) of each of the three herbicidal active ingredients in the final formulated composition ranges from 0.01 – 24.98% and jointly ranges from 0.03 – 25%.

In a preferred embodiment, the herbicide composition according to the invention is a rice herbicide composition.

In a preferred embodiment, the herbicide composition comprises a polymer that is made of a polysaccharide, more specifically a cellulose type molecule including but not limited to cellulose acetate, cellulose triacetate, cellulose propionate, cellulose acetate propionate, cellulose acetate butyrate, cellulose nitrate, cellulose sulfate, methylcellulose, ethylcellulose, ethyl methyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, hydroxyethyl methyl cellulose, hydroxypropyl methyl cellulose, ethyl hydroxyethyl cellulose, carboxymethyl cellulose, croscarmellose sodium, or hemicellulose, or mixtures thereof. The weight percentage (%) of the polymer, which can be used as, including but is not limited to, an intermediate, Manufacturing Use Product (MUP), or a finished product, ranges from 0.01 – 18%, preferably 0.01 – 12%, when it is made of a single cellulose. When the polymer is made of a mixture of at least two celluloses, its weight percentage (%) is $\sum X_i$, where i = 1, 2, … , n, X_i is the i^{th} cellulose type in the polymer whose weight percentage (%) ranges from 0.01 to [18-(n-1)x0.01]%, and the total is $\sum X_i \leq 18\%$. In the case of granule formulation, the weight percentage (%) of the polymer preferably ranges from 0.01 – 5%, such as 0.02 – 2%; in the case of sprayable formulation, the weight percentage (%) of the polymer preferably ranges from 2 – 18%, such as 8 – 12%.

The polymer according to the invention has a particle size range of 5 – 800 μm. The polymer particle size according to the invention is used to control the rate of release of herbicidal active ingredients when the polymer content's percentage by weight is fixed.

In a preferred embodiment, the polymer with at least one herbicidal active ingredient comprised in sprayable formulations such as WP or WDG according to the invention has particle sizes in the range of 5 – 25 μm.

In a preferred embodiment, the polymer with at least one herbicidal active ingredient comprised in granule formulations according to the invention has particle sizes in the range of 40 – 750 μm. In the granule, the release rate of the herbicidal active ingredients according to polymer particle size is as follows: 40 – 75 μm: fastest; 75 – 150 μm: faster; 150 – 350 μm: fast; 350 – 420 μm: used for rice herbicides with moderate release rates, which is preferable; 420 – 600 μm: slow; 600 – 750 μm: slower; 750 – 1000 μm: slowest.

The different ranges of particle size described above are all technically very achievable. Selection of a particular range depends upon the specific application.

In another preferred embodiment, the controlled release composition comprises such an abovementioned polymer that contains a single herbicidal active ingredient (such as sulfentrazone alone) or multiple herbicidal active ingredients of Groups 1 and/or 2, and/or other active ingredients.

In another preferred embodiment, the controlled release composition comprises such abovementioned polymers in which each polymer contains a different herbicidal active ingredient; or each polymer contains more than one chemically distinct herbicidal active ingredient; or different polymers contain a same herbicidal active ingredient; or mixtures thereof. The herbicidal active ingredients are those listed in Groups 1 and 2 or other active ingredients.

In another preferred embodiment, the controlled release composition is fomulated into a granule or sprayable form by dispersing one or more herbicidal active ingredient selected from below in the polymer: a PPO inhibitor herbicide of Group 2, including but not limited to, acifluorfen-sodium, bifenox, butafenacil, carfentrazone-ethyl, cinidon-ethyl, flufenpyr-ethyl, flumiclorac-pentyl, flumioxazin, fluoroglycofen-ethyl, fluthiacet-methyl, fomesafen, lactofen, oxadiargyl, oxadiazon, oxyfluorfen, pentoxazone, pyraclonil, pyraflufen-ethyl, sulfentrazone, and saflufenacil; or an ALS or AHAS inhibitor herbicide of Group 2, including but not limited to, imazamethabenz-methyl, imazamox, imazapic, imazapyr, imazaquin, and imazethapyr; or an herbicide active ingredient of Group 1, including but not limited to, profoxydim, clomazone, fenoxaprop-P-ethyl, and quinclorac. This is one of the few limited ways, if such other ways exist, to introduce highly efficacious herbicides as a new application that would be otherwise unsafe to crops, such as sulfentrazone to rice in this invention, or an alternative approach to other herbicide-resistant weeds.

In a further preferred embodiment, a final granule composition according to the invention has in general the following compositions and loadings: 0.02–14% two active ingredients jointly selected from Group 1 and/or Group 2 or 0.03–14% three active ingredients jointly selected from Group 1 and/or Group 2, 0.01–18% polymer (e.g., cellulose acetate butyrate), 0.001–1% C_{18-22} amine (e.g., octadecylamine), 0.25–25% dispersant (e.g., sodium lignosulphonate), 0.05–10% wetting agent (e.g., Supragil WP), 0.125–12.5% binder (e.g., Polyvinylpyrrolidone K-30), 0.05–15% carrier (e.g., Sipernat 50S), and 40-95% inert carrier (e.g., kaolin).

According to this invention, an herbicide composition offers multiple modes of action and a possible synergistic effect against *Leptochloa chinensis*.

For achieving control of all major weeds with >90% efficacy for up to 42 days or longer, our controlled release technology releases initially adequate amounts of the herbicidal active ingredients (such as sulfentrazone, penoxsulam, and clomazone) to eradicate already emerged weeds first, and then continues to release over a period of 40 days to provide long-lasting residual activity to continuously prevent the emergence of new weeds.

Our controlled-release mechanism has made it possible for the highest herbicidal concentration to remain in the top 0-1 inch of soil and to remain there for longer. The top 0-1 inch of soil is the main root zone of already emerged weeds, emerging weeds, and weed seeds which are likely to emerge, and does not coincide with rice's major root zone which is deeper (Figure 1). This root-depth difference, which already exists at time of application, becomes progressively bigger over the rice cycle, thus ensuring minimal to no injury to rice from the start. The invention is therefore able to introduce very efficacious active ingredients that were otherwise unsafe to crops.

For the first time, sulfentrazone (PPO inhibitor herbicide), a highly efficacious active ingredient but unacceptably phytotoxic in prior applications, is safely introduced into rice. Unlike the other active ingredients, sulfentrazone is not only effective in killing already emerged sedges and broadleaf weeds, but is also especially effective in preventing the emergence of most species in all three major categories of weeds. Through this invention, another PPO inhibitor herbicide, Carfentrazone-ethyl, can also be safely introduced to rice. This formulation is much safer than Shark® H_2O, a Carfentrazone-ethyl product that is accompanied by several strict restrictions and hardly ever used by farmers. Even when Shark® H_2O's label is exactly followed, severe rice leaf injury such as brownish burning spots quickly result, which reduces the efficiency of rice photosynthesis, causing adverse effects on growth and yield.

The invention chooses to incorporate PPO inhibitor herbicides as part of the rice product because (a) the inventor has developed mature controlled-release formulation technologies that can finally take advantage of sulfentrazone's high efficacy through reduction or elimination of its severe phytotoxicity in rice; and (b) there are some unique benefits of introducing PPO-inhibiting chemistry into rice fields. First, PPO inhibitor herbicides in general have gained popularity in the past few years to help combat herbicide-resistant weeds. Second, PPO-inhibitor-herbicide resistant weeds are not common. While more and more ALS-inhibitor-herbicide resistant weeds have shown up, PPO inhibitor herbicides are becoming one of the better alternatives in dealing with them.

Clomazone is another one of the active ingredients included in the invention. Clomazone has been approved for use in rice since FMC introduced it to the market; however, its worldwide rice application has been very limited due to its phytotoxicity, often causing "rice whitening or bleaching". Worse, clomazone is drifting and results in large areas of "whitening" if sensitive plants are nearby. Consequently, clomazone is rarely used in China, a country of major rice production. Similarly, the clomazone product Command® 3ME was withdrawn from the market in the Philippines and other countries due to its unacceptable phytotoxicity. In contrast, our granule product has eliminated the drifting and reduced the "whitening" to a nonexistent or otherwise acceptable level when applied to a rice field. Through our invention, clomazone, which is intrinsically highly active towards many of the major rice weeds, such as *Echinochloa crus-galli, Echinochloa colonum, Brachiaria platyphylla, Digitaria spp., Panicum spp., Leptochloa spp*. (FMC Command® 3ME Label) prevalent in both hot-climate and temperate rice fields, is able to be reintroduced from a totally different safety perspective. Another benefit of reintroducing clomazone is its ability to partially control *Oryza punctate,* which is the most rice yield-limiting weed and for which we still lack a selective herbicide.

There is no drifting of clomazone nor any direct clomazone contact with rice leaves. Therefore, it causes far less or no injury to rice as compared to other applications containing clomazone, such as Command® 3ME. Gone are concerns about applying clomazone adjacent to areas with sensitive crops.

In another aspect, the invention provides a rice herbicide composition comprising at least one herbicidal active ingredient selected from Group 1 and at least one herbicidal active ingredient selected from Group 2. In a prefered embodiment, the rice herbicide composition comprising at least three herbicidal active ingredients selected from Group 1 and Group 2. For example, a prefered rice herbicide composition may comprise sulfentrazone, penoxsulam, and clomazone.

A possible synergistic effect against *Leptochloa chinensis*, resulting from the combination of sulfentrazone, penoxsulam, and clomazone, has been observed. For example, our field trials in the Philippines treated with three different test samples (SPC 1, SPC 4, and SPC 5) at 320 g ai/ha each, which contained only 183 g ai/ha of clomazone, showed fast *Leptochloa chinensis* control at efficacies of 86%, 88%, and 88% in only 8 days after the application (Table 29), continuous improvement over subsequent days, and 90%, 88%, and 90% control at 43 DAA. *Leptochloa chinensis* at the time of application was at 3 leaf stages (Table 24).

The likely synergism comes from the fact that 183 g ai/ha of clomazone controlled *Leptochloa chinensis*, which is at only 1/5 – 1/3 of the recommended rate (552-893 g ai/ha of clomazone) for *Leptochloa chinensis* according to the FMC product label (page 7, Command® 3ME published on February 14, 2014).

It should also be pointed out that *Leptochloa chinensis* was artificially introduced in our trials, and therefore presented at likely a much higher density than that of *Leptochloa chinensis* under FMC test conditions, where no such artificial weed introduction was reported.

We further confirmed this likely synergism from a specifically designed experiment. First, the experiment had *Leptochloa chinensis* collected from two different locations in the Philippines to eliminate any possibility, though extrememly unlikely, of a 'defect' in the weed from a particular location. Second, only *Leptochloa chinensis* was introduced into the test fields to avoid any competition to *Leptochloa chinensis* from other major weeds. Third, one of the two application timings was delayed to 15 DAS (in the 10-15 DAS time window) to allow *Leptochloa chinensis* to grow bigger. All these adjustments were to make *Leptochloa chinensis* more difficult to kill than that in earlier trials. The test results against *Leptochloa chinensis* (from both locations), at 320 g ai/ha applied at 10 DAS, revealed the same control efficacy of 91% as soon as 7 DAA and 97% at 21 DAA; at 320 g ai/ha applied at 15 DAS, 96% (location 1) and 95% (location 2) at 7 DAA and the same 98% at 21 DAA (Tables 39-40). Such fast killing under relatively severe conditions at only 1/5 – 1/3 of clomazone's recommended use rate, which has been the status quo for the past 30 years, seems best explained by a synergistic effect.

Synergy, by definition, is the creation of a whole that is greater than the simple sum of its parts. In this case, neither sulfentrazone nor penoxsulam individually has much activity at all towards *Leptochloa chinensis* at the 2.5-4.5 leaf-stage. Moreover, the controlled release technology simply controls the amount released at any given time of sulfentrazone, which, as stated, is inactive against the 2.5-4.5 leaf-stage *Leptochloa chinensis*. We believe this significant synergism (with our clomazone use rate at a very minimal one-fifth to one-third of the recommended rate for the 2.5-4.5 leaf-stage *Leptochloa chinensis*) comes from the combination of clomazone, "*Leptochloa chinensis* inactive sulfentrazone" and "*Leptochloa chinensis* inactive penoxsulam."

In a preferred embodiment, the ratio range of sulfentrazone : penoxsulam : clomazone is (1-5) : (1-4) : (2-12) and any permutation thereof. More preferably, the ratio range of sulfentrazone : penoxsulam : clomazone is (1-2) : (1-3) : (2-8).

Our trials in South Korea, a temperate rice region where sedges and broadleaves, especially sulfonylurea-resistant sedges and broadleaves, are the main challenge, showed good weed control efficacy to all weeds (at 30 DAA, 87% and 91% at 120 g ai/ha and 180 g ai/ha of sulfentrazone, respectively) even with the granules containing only sulfentrazone (Table 51). Among all weeds tested in the trials, *Echinochloa oryzicola, Monochoria vaginalis,* and *Scirpus Juncoides* were reported to be sulfonylurea-resistant for which sulfentrazone was still effective against *Monochoria vaginalis* (Table 45) and *Scirpus Juncoides* (Table 46).

Thanks to the carefully timed controlled release, maximized use of the loaded actives, and overall relatively low use rate, after the crop harvest there is not much residual activity in the soil that could affect new crops, including sensitive crops, planted during the next cycle. No waiting period between harvest and replanting is necessary, as evidenced for example by the immediate planting of soybean, mung bean, corn, onion, and tomato, into the same field treated with our products during the preceding rice season. At no point was any phytotoxicity observed.

Additionally, this product delivers consistently across a wide spectrum of applications – from Japonica rice to Indica rice, from transplanted rice (both machine/hand-transplanted) to direct-seeded rice; and from 10-day old rice seedlings to 30-days or older seedlings.

Weed Control Method

The invention further relates to a weed control method in rice fields, comprising applying the above-mentioned one-shot herbicide composition in the rice fields.

When applied, the total weight of the herbicidal active ingredients ranges from 5-1280 g ai/ha. The use rates of the final formulated composition are preferably 1-75 kg/ha, such as 10 kg/ha, or 20 kg/ha.

In a prefered embodiment, the composition is used in the fields for Indica rice or Japonica rice.

The application timings for direct-seeded rice and transplanted rice are 7-15 DAS (days after seeding) and 5-15 DAT (days after transplanting), respectively, overlapping with initial application timing of almost all herbicides.

The application of the product is lighter, safer, and more efficient and convenient as compared to the predominant sprayable solution form. The composition of the invention leaves traceable marks of granules in the rice field for the convenience of the applicant to keep track of application.

Active Ingredients' Release Profiles

We have selected such combinations of active ingredients, as described above and later, to form products that take advantage of a dual-mode of action and demonstrate a synergistic effect (*vide infra*) against *Leptochloa chinensis*. Such products would have a higher probability of killing these weeds. The mechanism that delivers appropriate amounts of these active ingredients at specific times over a period of approximately 40 days is our controlled release technology. This way, the technology ensures a sufficient herbicidal concentration to the targets that is still safe to the crop to achieve what we call "controlling already emerged weeds first and then preventing the emergence of new weeds in a continuous mode," which sets this invention apart from any prior art.

Because of the timely release of the active ingredients, availability of fresh active ingredients is guaranteed, resulting in a more effective and longer residual activity. The technology contributes in part to the overall excellent performance of our products.

There are reports in the literature describing slow release technologies. It shall be reminded that slow-release technologies are different and confined to their relevant contexts to which they can deliver the value. For example, including but not limited to, in a case where the release of active ingredients is slower but still within a few hours or a day. Slow release technology of this kind would not contribute any additional value to weed control in rice than a similar technology without the slow release.

In conclusion, if a controlled-release technology is to add additional value over a formulation with no such technology in controlling a target, the technology must demonstrate the ability to deliver the active ingredient over a period of at least 30 days.

It is worth mentioning that many reported slow release technologies measure their releases under shaking conditions, which is unrealistic to application in an actual rice field where release occurs under undisturbed conditions.

The production of the herbicide composition of the invention can be done, for example, by first evenly dispersing at least one herbicidal active ingredient as mentioned above in the polymer with or without solvent to form the controlled-release component, and then milling, fiber extruding, or spray cooling (or other appropriate means) the component into particles of predetermined particle size, followed by mixing the obtained particles with other herbicidal active ingredient(s), if any, and other neccesary adjuvants such as dispersant, binder, wetting agent, carrier, surfactant, filler, etc., and then pelletizing and drying the mixture to obtain the herbicide composition.

Examples

Below are the definitions of certain abbreviations, technical terms, and terminologies:

Organizations:

SynTech:	SynTech Research Southeast Asia, Inc., RG Farm, Purok 5, Sta. Arcadia, Cabanatuan City, 3100, Philippines;
SAAS:	Shanghai Academy of Agricultural Sciences, No. 1000, Jin Qi Road, Feng Xian District, Shanghai
SSPC:	Shanghai Shengnong Pesticide Co. Ltd., Room 1201-1204, No. 2, Lane 7866, Humin Road Minhang District, Shanghai 201102, China

Formulation:

Hammermill:	a machine whose purpose is to shred or crush aggregate material into smaller pieces
D_{90}:	90% of all particle sizes

Biology and Commercial Herbicide Products:

AI:	Active ingredient
DAS:	Days after seeding
DAT:	Days after transplanting
DAA:	Days after application
Fb:	followed by
MUP:	Manufacturing Use Product
N/A:	Not Applicable
Nominee®:	100 SC bispyribac-sodium
Clincher®:	100 EC cyhalofop-butyl
Pyzero®:	10 EC metamifop

Spartan® 4F: 39.6% sulfentrazone
Command® 3ME: 31.1% clomazone
Shark® H_2O: 40% carfentrazone-ethyl

Major weeds in hot-climate rice fields (e.g., in Thailand and Philippines): *Echinochloa crus-galli* (grass), *Leptochloa chinensis* (grass), *Ischaemum rugosum* (grass), *Monochoria vaginalis* (broadleaf), *Sphenoclea zeylanica* (broadleaf), *Ludwigia octovalvis* (broadleaf), *Cyperus difformis* (sedge), *Fimbristylis miliacea* (sedge), and *Cyperus iria* (sedge).

Major weeds in temperate rice fields (e.g., in Korea and Japan): *Echinochloa crus-galli* (grass), *Echinochloa oryzicola* (grass), *Sagittaria pygmaea* (broadleaf), *Monochoria vaginalis* (broadleaf), *Ludwigia prostrata* (broadleaf), *Lindernia dubia* (broadleaf), *Lindernia procumbens* (broadleaf), *Cyperus difformis* (sedge), *Scirpus juncoides* (sedge), and *Eleocharis kuroguwai* (sedge), and their sulfonylurea-resistant counterparts.

Example 1: 1.4% Herbicide Granule (0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone)

Table 1. 1.4% Granule (Sulfentrazone 0.2% + Penoxsulam 0.4% + Clomazone 0.8%)

	Ingredients	Weight (g)	Function	CAS No.
Make 57.96% S-MUP	Sulfentrazone 92%	63	AI	122836-35-5
	Cellulose acetate butyrate (MW: 12000)	35	Polymer	9004-36-8
	Octadecylamine	2	Chemical	124-30-1
	Dichloromethane	319	Solvent	75-09-2
Make 1.4% Granule	57.96% S-MUP	0.345	S-MUP	
	Penoxsulam 96.7%	0.414	AI	219714-96-2
	Clomazone 91.8%	0.872	AI	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Kaolin	Fill to 100	Filler	1332-58-7

1. Make 57.96% S-MUP (Sulfentrazone)
   a. Add 35.0 g of cellulose acetate butyrate, 63.0 g of Sulfentrazone 92%, and 2.0 g of octadecylamine into 319.0 g of dichloromethane solvent, and then stir the mixture until completely dissolved.
   b. The solution is poured into a flat chassis which is placed in a chemical hood, the solvent is allowed to evaporate in the hood for 10 – 15 hours, and then the chassis is transferred into an oven for about 2 hours at 55 – 60 ^{0}C. Use a spatula to break the film into small pieces (no greater than 2 cm in diameter). Put all small pieces into a blender and turn the blender on for just 1-3 seconds to crush

the pieces. Sieve crushed particles through a set of 40 and 45 mesh sieves to obtain particles with sizes between approximately 350–420 μm (57.96% Sulfentrazone MUP). The bigger particles can go through the re-crush process to obtain the desired particle size and the smaller particles through a recovery process to minimize the loss (Example 3).

2. Make P-MUP (Penoxsulam) and C-MUP (Clomazone)
   a. 8.15% P-MUP: Add 0.414 g of 96.7% of Penoxulam and 4.5 g of kaolin into a hammermill and homogenize the powder mixture for about 2 minutes to have 8.15% of Penoxsulam MUP. (Total: 4.914 g)
   b. 29.61% C-MUP: Add 20 g of kaolin and 22 g of Sipernat 50S in this order into a blender and mix them for about 30 minutes. Add 20 g of Clomazone 91.8% in 6-8 portions dropwise into the powder mixture, with intermediate mixing after adding each portion. Make sure that all clomazone is thoroughly distributed (Total: 62 g).

3. Make 1.4% Granule (0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone)
   a. In a plastic bag, add 0.345 g of the S-MUP, 4.914 g of the P-MUP, 2.702 g of the C-MUP, 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.
   b. After the above 2-hour mixing, transfer the mixture into a wet mixer. Add 20g of water (tap water) into the mixture, with thorough mixing during adding, over a period of approximately 30-40 minutes. Make sure that water is evenly distributed.
   c. Make granules through a granulator, such as ZLB2-80.
   d. The granules are dried in an oven at ~60℃, checking the water content during the drying process. The final water weight % in the granules is >0.8%, but less than 1.2% (H_2O: 0.8% – 1.2%).

This formula is listed in Table 4, Section F1.

Example 2: Making S-MUP without Solvent

1. Make 57.96% S-MUP (Sulfentrazone)
   a. Mix 35.0 g of cellulose acetate butyrate, 63.0 g of Sulfentrazone 92%, and 2.0 g of octadecylamine well and fill into a beaker containing a magnetic stirrer. Heat the mixture on a 170^{0}C hot plate and stir the melt until a homogenous phase is reached. (The melt is viscous and the beaker must be strong. The ideal stirring is to reach as much the edge or all corners as possible.)

b. Let the homogenous phase cool down to room temperature and then place the beaker in a -20^{0}C refrigerator for 10 minutes for easy removal of the melt from the beaker. Use a spatula to break the melt into small pieces. Put all small pieces into a blender and turn the blender on for just 1-3 seconds to crush the pieces. Sieve crushed particles through a set of 40 and 45 mesh sieves to obtain particles with sizes between approximately 350–420 μm (57.96% Sulfentrazone MUP). The bigger particles can go through the re-crush process to obtain the desired particle size and the smaller particles through a recovery process to minimize the loss (Example 3).

Example 3: Recovery of Fine Particles of S-MUP for Reuse

a. A Petri dish (11 cm diameter), sitting on a heating plate, is first covered with a piece of aluminum foil and then a beaker, and the heating plate is adjusted to 170^{0}C. Pre-warm 50 g fine particles of S-MUP in a separate beaker to 54^{0}C, and remove the beaker and aluminum foil covering the Petri dish, pour the fines into the hot Petri dish, and flatten the surface of the fines by careful pressing with a flat surface object, such as a smaller Petri dish. Cover and wait for 12-15 minutes until a homogenious phase of the melt is formed. Remove the Petri dish from the hot plate and let it cool down to room temperature. The melt formed in the Petri dish can be removed from the dish by cooling the dish to -20^{0}C in a refrigerator for 10 minutes and then carefully bumping.
b. This procedure (step a) can be used to recover fine particles made from both the solvent and no-solvent methods.

Example 4: 2.8% Herbicide Granule (0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone)

a. Similar to Example 1 in a plastic bag, add 0.690 g of 57.96% S-MUP, 9.816 g of 8.15% P-MUP, 5.404 g of 29.61% C-MUP, 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.
b. Follow steps b-d of "3. Make 1.4% Granule" in Example 1 to obtain the final granules.

This formula is list in Table 4, Section F3.

Example 5: 1.4% Herbicide Granule (0.2% Sulfentrazone + 0.4% Bispyribac-sodium + 0.8% Clomazone)

a. Make the S-MUP and C-MUP according to Example 1.

b. Make 8.13% B-MUP (Bispyribac-sodium). Add 0.42 g of 95.2% of Bispyribac-sodium and 4.5 g of kaolin into a hammermill grinder and homogenize the powder mixture for about 2 minutes to have 8.13% of B-MUP (Total: 4.92 g).
c. To a plastic bag, add 0.345 g of 57.96% S-MUP, 4.92 g of 8.13% B-MUP, 2.702 g of 29.61% C-MUP, 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.
d. Follow steps b-d of "3. Make 1.4% Granules" in Example 1 to obtain the final granules.

This formula is listed in Table 4, Section F8.

Example 6: 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.2% Cyhalofop-butyl + 0.4% Clomazone); solvent-free preparation

1. Make 53.36% S-MUP (Sulfentrazone)
   a. Mix 40.0 g of cellulose acetate butyrate, 58.0 g of Sulfentrazone 92%, and 2.0 g of octadecylamine well and fill into a beaker containing a meganetic stirrer (The beaker must be strong.). Heat the mixture on a 170^{0}C hot plate, while stirring, until a homogenous phase of the melt mixture is obtained. The melt mixture can be removed from the beaker by cooling it down to room temperature first, then to -20^{0}C for 10 minutes, and careful bumping.
   b. Break the mixture into small pieces. Put all small pieces into a blender and turn the blender on for just 1-3 seconds to crush the pieces. Sieve crushed particles through a set of 40 and 45 mesh sieves to obtain particles with sizes between approximately 350–420 μm (53.36% Sulfentrazone MUP). The bigger particles can go through the re-crush process to obtain the desired particle size and the smaller particles through a recovery process to minimize the loss (Example 3).

2. Make Cyh-MUP (Cyhalofop-butyl) and C-MUP (Clomazone)
   a. 4.24% Cyh-MUP: Add 0.205 g of 97.4% Cyhalofop-butyl and 4.5 g of kaolin into a hammermill grinder and homogenize the powder mixture for about 2 minutes to have 4.24% of Cyhalofop-butyl MUP (Total: 4.705 g).
   b. 29.61% C-MUP: Obtain according to Example 1.

3. Make 0.8% Granule (0.2% Sulfentrazone + 0.2% Cyhalofop-butyl + 0.4% Clomazone)
   a. In a plastic bag, add 0.375 g of the S-MUP, 4.705 g of the Cyh-MUP, 1.351 g of 29.61% C-MUP, 5.0 g of Borresperse NA, 1.0g of Supragil WP, 2.5 g of

Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.

b. After the above 2-hour mixing, transfer the mixture into a wet mixer. Add 20 g of water (tap water) into the mixture, with thorough mixing during adding, over a period of approximately 30-40 minutes. Make sure that water is evenly distributed.

c. Make granules through a granulator, such as ZLB2-80.

d. The granules are dried in an oven at ~60^0C while checking the water content during the drying process. The final water weight % in the granules is >0.8%, but less than 1.2% (H_2O: 0.8% – 1.2%).

This formula is listed in Table 4, Section F9.

Example 7: 1.4% Herbicide Granule (0.2% Saflufenacil + 0.4% Penoxsulam + 0.8% Clomazone)

Table 2. 1.4% Granule (0.2% Saflufenacil + 0.4% Penoxsulam + 0.8% Clomazone)

	Ingredients	Weight (g)	Function	CAS No.
Make 47.5% Saf-MUP	95% Saflufenacil	50	AI	372137-35-4
	Cellulose acetate butyrate (MW: 12000)	48	Polymer	9004-36-8
	Octadecylamine	2	Chemical	124-30-1
	Dichloromethane	360	Solvent	75-09-2
Make 1.4% Granule	47.5% Saf-MUP	0.421	S-MUP	
	96.7% Penoxsulam	0.414	AI	219714-96-2
	91.8% Clomazone	0.872	AI	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Kaolin	Fill to 100	Filler	1332-58-7

1. Make 47.5% Saf-MUP (Saflufenacil)

a. Add 48.0 g of cellulose acetate butyrate, 50.0 g of 95% Saflufenacil, and 2.0 g of octadecylamine into 360.0 g of dichloromethane solvent, and then stir the mixture until completely dissolved.

b. The solution is poured into a flat chassis which is placed in a chemical hood, the solvent is allowed to evaporate in the hood for 10-15 hours, and then the chassis is transferred into an oven for about 2 hours at about 60℃. Use a spatula to break the film into small pieces (no greater than 2 cm in diameter). Put all small pieces into a blender and turn the blender on for just 1-3 seconds to crush the pieces. Sieve crushed particles through a set of 40 and 45 mesh sieves to obtain particles with sizes between approximately 350–420 μm (47.5% Saflufenacil MUP). The bigger particles can go through the re-crush process to obtain the desired particle size and the smaller particles through a recovery process to minimize the loss (Example 3).

2. Make 8.15% P-MUP (Penoxsulam) and 29.61% C-MUP (Clomazone)
   a. Obtain the P-MUP and C-MUP according to Example 1.

3. Make 1.4% Granule (0.2% Saflufenacil + 0.4% Penoxsulam + 0.8% Clomazone)
   a. In a plastic bag, add 0.421 g of the Saf-MUP, 4.914 g of the P-MUP, 2.702 g of the C-MUP, 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.
   b. After the above 2-hour mixing, transfer the mixture into a wet mixer. Add 20 g of water (tap water) into the mixture, with thorough mixing during adding, over a period of approximately 30-40 minutes. Make sure that water is evenly distributed.
   c. Make granules through a granulator, such as ZLB2-80.
   d. The granules are dried in an oven at ~60℃, while checking the water content during the drying process. The final water weight % in the granules is >0.8%, but less than 1.2% (H_2O: 0.8% – 1.2%).

This formula is listed in Table 4, Section F10.

Example 8: 1% Herbicide Granule (0.35% Sulfentrazone + 0.65% Penoxsulam)

a. In a plastic bag, add 0.604 g of 57.96% S-MUP (Example 1), 7.975 g of 8.15% P-MUP (Example 1), 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.
b. Follow steps b-d of “3. Make 1.4% Granule” in Example 1 to obtain the final granules.

This formula is listed in Table 4, Section F15.

Example 9: 1% Herbicide Granule (0.8% Sulfentrazone + 0.2% Azimsulfuron)

a. Make 4.24% A-MUP (Azimsulfuron): Add 0.21 g of 95% Azimsulfuron and 4.5 g of kaolin into a hammermill grinder and homogenize the powder mixture for about 2 minutes to have 4.24% Azimsulfuron MUP (Total: 4.71 g).

b. In a plastic bag, add 1.380 g of 57.96% S-MUP (Example 1), 4.71 g of the A-MUP, 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-powder mixer and mix for 2 hours.
c. Follow steps b-d of "3. Make 1.4% Granule" in Example 1 to obtain the final granules.

This formula is listed in Table 4, Section F16.

Example 10: 0.4% Herbicide Granule (0.4% Sulfentrazone)

a. In a plastic bag, add 0.690 g of 57.96% S-MUP (Example 1), 5.0 g of Borresperse NA, 1.0 g of Supragil WP, 2.5 g of Polyvinylpyrrolidone K-30, and fill kaolin to a net weight of 100 g (excluding the weight of the bag). Mix the mixture thoroughly for 15 minutes in the bag and then transfer into a dry-power mixer and mix for 2 hours.
b. Follow steps b-d of "3. Make 1.4% Granules" in Example 1 to obtain the final granules.

This formula is listed in Table 4, Section F20.

Example 11: 20% Sulfentrazone Wettable Powder (WP)

Table 3. 20% Sulfentrazone WP

	Ingredients	Weight (g)	Function	CAS No.
Make 61.64% S-MUP	92% Sulfentrazone	67	Active ingredient	122836-35-5
	Cellulose acetate butyrate (MW: 12000)	31	Polymer	9004-36-8
	Octadecylamine	2	Chemical	124-30-1
	Dichloromethane	320	Solvent	75-09-2
Make 20% Sulfentrazone WP	61.64% S-MUP	32.45	S-MUP	
	Nekal Bx	4	Surfactant	25638-17-9
	Borresperse NA	4	Dispersant	8061-51-6
	Supragil WP	2	Surfactant	1322-93-6
	Rhodia ANTAROX® FM33	4	Surfactant	68154-97-2
	Geropon T 36	2	Surfactant	37199-81-8
	Sipernat 50S	29	Carrier	112926-00-8
	Kaolin	Fill to 100	Filler	1332-58-7

<u>1. Make 61.64% S-MUP (Sulfentrazone)</u>

a. Add 31.0 g of cellulose acetate butyrate, 67.0 g of Sulfentrazone 92%, and 2.0 g of octadecylamine into 320.0 g of dichloromethane solvent, and then stir the mixture until completely dissolved.

b. The solution is poured into a flat chassis which is placed in a chemical hood, the solvent is allowed to evaporate in the hood for 10-15 hours, and then the chassis is transferred into an oven for about 2 hours at 55-60℃. Use a spatula to break the film into small pieces (no greater than 2 cm in diameter).
c. Use a hammermill to mill the small pieces to about 400 µm and then use an air jet mill to mill to about 25 µm (D_{90}) (61.64% S-MUP).

2. Make WP Formulation

a. To a container, according to the 20% Sulfentrazone WP formulation in Table 3, add 32.45 g of the S-MUP, 4 g of Nekal BX, 4 g of Borresperse NA, 2 g of Supragil WP, 4 g of Rhodia ANTAROX® FM 33, 2 g of Geropon T 36, 29 g of Sipernat 50S, and fill to the weight of 100 g with kaolin. Transfer the mixture to a hammermill to mill for 5 minutes.
b. Transfer the mixture from the hammermill to an air jet mill to mill to the particle size of D_{90} ≤15 µm, preferably ≤10 µm.

This formula is listed in Table 4, Section F19.

From F1 through F35 in Table 4, it lists a variety of granule formulation types and WP or WDS formulation forms.

Table 4. A List of Examples of Formulas

F1: 1.4% Granule (0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone) The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone				
F1:	**Substance**	**Weight (g/100 g)**	**Function**	**CAS No**
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.414	Active ingredient	219714-96-2
	91.8% Clomazone	0.872	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7
F2: 2.1% Granule (0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone) The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone				
F2	**Substance**	**Weight (g/100 g)**	**Function**	**CAS No**
	92% Sulfentrazone	0.326	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.621	Active ingredient	219714-96-2
	91.8% Clomazone	1.307	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	1.438	Carrier	112926-00-8

	Cellulose acetate butyrate	0.181	Polymer	9004-36-8
	Octadecylamine	0.010	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F3: 2.8% Granule (0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F3	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.827	Active ingredient	219714-96-2
	91.8% Clomazone	1.744	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	1.919	Carrier	112926-00-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.014	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F4: 4.2% Granule (0.6% Sulfentrazone + 1.2% Penoxsulam + 2.4% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F4	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.652	Active ingredient	122836-35-5
	96.7% Penoxsulam	1.241	Active ingredient	219714-96-2
	91.8% Clomazone	2.614	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	2.876	Carrier	112926-00-8
	Cellulose acetate butyrate	0.362	Polymer	9004-36-8
	Octadecylamine	0.021	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F5: 5.6% Granule (0.8% Sulfentrazone + 1.6% Penoxsulam + 3.2% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F5	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.870	Active ingredient	122836-35-5
	96.7% Penoxsulam	1.655	Active ingredient	219714-96-2
	91.8% Clomazone	3.486	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	3.835	Carrier	112926-00-8
	Cellulose acetate butyrate	0.483	Polymer	9004-36-8
	Octadecylamine	0.028	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F6: 7.0% Granule (1.0% Sulfentrazone + 2.0% Penoxsulam + 4.0% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F6	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	1.087	Active ingredient	122836-35-5
	96.7% Penoxsulam	2.068	Active ingredient	219714-96-2
	91.8% Clomazone	4.357	Active ingredient	81777-89-1

	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	4.793	Carrier	112926-00-8
	Cellulose acetate butyrate	0.604	Polymer	9004-36-8
	Octadecylamine	0.035	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7
F7: 12.6% Granule (1.8% Sulfentrazone + 3.6% Penoxsulam + 7.2% Clomazone) The range of the polymer particle sizes: 350-420 µm; the polymer containing sulfentrazone				
F7	**Substance**	**Weight (g/100 g)**	**Function**	**CAS No**
	92% Sulfentrazone	1.957	Active ingredient	122836-35-5
	96.7% Penoxsulam	3.723	Active ingredient	219714-96-2
	91.8% Clomazone	7.843	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	8.628	Carrier	112926-00-8
	Cellulose acetate butyrate	1.087	Polymer	9004-36-8
	Octadecylamine	0.062	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7
F8: 1.4% Granule (0.2% Sulfentrazone + 0.4% Bispyribac-sodium + 0.8% Clomazone) The range of the polymer particle sizes: 300-500 µm; the polymer containing sulfentrazone				
F8	**Substance**	**Weight (g/100 g)**	**Function**	**CAS No**
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	95.2% Bispyribac-sodium	0.420	Active ingredient	125401-92-5
	91.8% Clomazone	0.872	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7
F9: 0.8% Granule (0.2% Sulfentrazone + 0.2% Cyhalofop-butyl + 0.4% Clomazone) The range of the polymer particle sizes: 250-500 µm; the polymer containing sulfentrazone				
F9	**Substance**	**Weight (g/100 g)**	**Function**	**CAS No**
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	97.4% Cyhalofop-butyl	0.205	Active ingredient	122008-85-9
	91.8% Clomazone	0.436	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.480	Carrier	112926-00-8
	Cellulose acetate butyrate	0.15	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7
F10: 1.4% Granule (0.2% Saflufenacil + 0.4% Penoxsulam + 0.8% Clomazone) The range of the polymer particle sizes: 400-600 µm; the polymer containing saflufenacil				

F10:	Substance	Weight (g/100 g)	Function	CAS No
	95% Saflufenacil	0.210	Active ingredient	372137-35-4
	96.7% Penoxsulam	0.414	Active ingredient	219714-96-2
	91.8% Clomazone	0.872	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Cellulose acetate butyrate	0.202	Polymer	9004-36-8
	Octadecylamine	0.008	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F11: 1.4% Granule (0.2% Benzobicyclon + 0.4% Penoxsulam + 0.8% Clomazone)
The range of the polymer particle sizes: 125-250 µm; the polymer containing benzobicyclon

F11:	Substance	Weight (g/100 g)	Function	CAS No
	95% Benzobicyclon	0.210	Active ingredient	156963-66-5
	96.7% Penoxsulam	0.414	Active ingredient	219714-96-2
	91.8% Clomazone	0.872	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Cellulose acetate butyrate	0.072	Polymer	9004-36-8
	Octadecylamine	0.006	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F12: 1.1% Granule (0.2% Sulfentrazone + 0.1% Azimsulfuron + 0.8% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F12:	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	96% Azimsulfuron	0.104	Active ingredient	120162-55-2
	91.8% Clomazone	0.872	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.959	Carrier	112926-00-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F13: 1.4% Granule (0.2% Sulfentrazone + 0.4% Ethoxysulfuron + 0.8% Metamifop)
The range of the polymer particle sizes: 200-420 µm; the polymer containing sulfentrazone

F13:	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	96% Ethoxysulfuron	0.416	Active ingredient	126801-58-9
	96% Metamifop	0.832	Active ingredient	256412-89-2
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F14: 1.2% Granule (0.2% Sulfentrazone + 0.2% Flucetosulfuron + 0.8% Cyhalofop-butyl)
The range of the polymer particle sizes: 125-420 µm; the polymer containing sulfentrazone

F14:	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	95% Flucetosulfuron	0.211	Active ingredient	412928-75-7
	97.4% Cyhalofop-butyl	0.821	Active ingredient	122008-85-9
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F15: 1% Granule (0.35% Sulfentrazone + 0.65% Penoxsulam)
The range of the polymer particle sizes: 355-650 µm; the polymer containing sulfentrazone

F15	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.380	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.672	Active ingredient	219714-96-2
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.211	Polymer	9004-36-8
	Octadecylamine	0.012	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F16: 1% Granule (0.8% Sulfentrazone + 0.2% Azimsulfuron)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F16	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.870	Active ingredient	122836-35-5
	96% Azimsulfuron	0.208	Active ingredient	120162-55-2
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.483	Polymer	9004-36-8
	Octadecylamine	0.028	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F17: 1% Granule (0.4% Sulfentrazone + 0.6% Benzobicyclon)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F17	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	95% Benzobicyclon	0.632	Active ingredient	156963-66-5
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.014	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F18: 1% Granule (0.4% Sulfentrazone + 0.6% Metazosulfuron)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F18	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	95% Metazosulfuron	0.632	Active ingredient	868680-84-6
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.014	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F19: 20% WP (20% Sulfentrazone)
D90: ≤ 12 µm; the polymer containing sulfentrazone

F19	Substance	Weight (g/100g)	Function	CAS No
	92% Sulfentrazone	21.739	Active ingredient	122836-35-5
	Cellulose acetate butyrate	10.060	Polymer	9004-36-8
	Nekal BX	4	Surfactant	25638-17-9
	Octadecylamine	0.649	Chemical	124-30-1
	Borresperse NA	4	Dispersant	8061-51-6
	Supragil WP	2	Surfactant	1322-93-6
	Rhodia ANTAROX® FM33	4	Surfactant	68154-97-2
	Geropon T 36	2	Surfactant	37199-81-8
	Sipernat 50S	29	Carrier	112926-00-8
	Kaolin	Fill to 100	Filler	1332-58-7

F20: 0.4% Sulfentrazone Granule (Controlled Release)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F20	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.014	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F21: 0.8% Granule (0.2% Sulfentrazone + 0.6% Clomazone); 28.5% release of sulfentrazone in 24h
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F21	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	91.8% Clomazone	0.654	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.719	Carrier	112926-00-8
	Cellulose acetate butyrate	0.145	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F22: 0.8% Granule (0.2% Sulfentrazone + 0.6% Clomazone); 37.9% release of sulfentrazone in 24h
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F22	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	91.8% Clomazone	0.654	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.719	Carrier	112926-00-8
	Cellulose acetate butyrate	0.132	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F23: 0.8% Granule (0.2% Sulfentrazone + 0.6% Clomazone); 67.4% release of sulfentrazone in 24h
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F23	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	91.8% Clomazone	0.654	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.719	Carrier	112926-00-8
	Cellulose acetate butyrate	0.100	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F24: 0.8% Granule (0.2% Sulfentrazone + 0.6% Clomazone); 87.1% release of sulfentrazone in 24h
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F24	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	91.8% Clomazone	0.654	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.719	Carrier	112926-00-8
	Cellulose acetate butyrate	0.091	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F25: 0.8% Granule (0.2% Sulfentrazone + 0.6% Clomazone); 93.1% release of sulfentrazone in 24h
The range of the polymer particle sizes: 75-125 µm; the polymer containing sulfentrazone

F25	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	91.8% Clomazone	0.654	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.719	Carrier	112926-00-8
	Cellulose acetate butyrate	0.085	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F26: 0.45% Granule (0.4% Sulfentrazone + 0.05% Azimsulfuron)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F26	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	96% Azimsulfuron	0.052	Active ingredient	120162-55-2
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.013	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F27: 0.47% Granule (0.40% Sulfentrazone + 0.07% Flucetosulfuron)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F27	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	96% Flucetosulfuron	0.073	Active ingredient	412928-75-7
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.013	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F28: 0.8% Granule (0.2% Sulfentrazone + 0.2% Penoxsulam + 0.4% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F28	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.207	Active ingredient	219714-96-2
	91.8% Clomazone	0.436	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.480	Carrier	112926-00-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F29: 0.7% Granule (0.2% Sulfentrazone + 0.1% Bispyribac-sodium + 0.4% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F29	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.217	Active ingredient	122836-35-5
	95.2% Bispyribac-sodium	0.105	Active ingredient	125401-92-5
	91.8% Clomazone	0.436	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.480	Carrier	112926-00-8
	Cellulose acetate butyrate	0.121	Polymer	9004-36-8
	Octadecylamine	0.007	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F30: 0.6% Granule (0.45% Sulfentrazone + 0.15% Penoxsulam)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F30	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.489	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.155	Active ingredient	219714-96-2
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.272	Polymer	9004-36-8
	Octadecylamine	0.016	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F31: 0.48% Granule (0.40% Sulfentrazone + 0.08% Penoxsulam)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F31	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.083	Active ingredient	219714-96-2
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.014	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F32: 0.6% Granule (0.4% Sulfentrazone + 0.2% Clomazone)
The range of the polymer particle sizes: 355-420 µm; the polymer containing sulfentrazone

F32	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	91.8% Clomazone	0.218	Active ingredient	81777-89-1
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	0.240	Carrier	112926-00-8
	Cellulose acetate butyrate	0.242	Polymer	9004-36-8
	Octadecylamine	0.014	Chemical	124-30-1
	Kaolin	Fill to 100	Filler	1332-58-7

F33: 1.02% Granule (0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron)
The range of the polymer particle sizes: 125-250 µm; polymer containing benzobicyclon

F33	Substance	Weight (g/100 g)	Function	CAS No
	95% Benzobicyclon	0.737	Active ingredient	156963-66-5
	98% Carfentrazone-ethyl	0.255	Active ingredient	128639-02-1
	95% Flucetosulfuron	0.075	Active ingredient	412928-75-7
	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Kaolin	Fill to 100	Filler	1332-58-7

F34: 2.8% Granule (0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone)

F34	Substance	Weight (g/100 g)	Function	CAS No
	92% Sulfentrazone	0.435	Active ingredient	122836-35-5
	96.7% Penoxsulam	0.827	Active ingredient	219714-96-2
	91.8% Clomazone	1.744	Active ingredient	81777-89-1

	Borresperse NA	5	Dispersant	8061-51-6
	Supragil WP	1	Wetting agent	1322-93-6
	Polyvinylpyrrolidone K-30	2.5	Binder	9003-39-8
	Sipernat 50S	1.919	Carrier	112926-00-8
	Kaolin	Fill to 100	Filler	1332-58-7
F35: 25% WP (25% Saflufenacil) **D_{90}: ≤12 µm; the polymer containing saflufenacil**				
F35	**Substance**	**Weight (g/100 g)**	**Function**	**CAS No**
	95% Saflufenacil	26.32	Active ingredient	372137-35-4
	Cellulose acetate butyrate	11.82	Polymer	9004-36-8
	Nekal BX	4	Surfactant	25638-17-9
	Borresperse NA	4	Dispersant	8061-51-6
	Supragil WP	2	Surfactant	1322-93-6
	Rhodia ANTAROX® FM33	4	Surfactant	68154-97-2
	Geropon T 36	2	Surfactant	37199-81-8
	Sipernat 50S	29	Carrier	112926-00-8
	Kaolin	Fill to 100	Filler	1332-58-7

Below is the assay with which we use to measure the release of active ingredients in our formulations.

Reagents

(1) Acetonitrile with purity≥99.9% (HPLC grade); (2) Water was Milli Q ultrapure water; (3) Solvent A: mixture of acetonitrile and water in weight ratio of 1:1; (4) Trifluoroacetic Acid (TFA) with purity ≥99.5% (HPLC grade); (5) 0.025%TFA: Transfer 1.0 ml of Trifluoroacetic acid into 4000 ml of water and shake well to mix; Standards: Sulfentrazone (98.3%), Penoxsulam (97.1%), and Clomazone (99.7%)

Instrumentation

(1) Agilent 1200 series HPLC system equipped with an Agilent auto sampler G1329A, or equivalent; (2) Column was Zorbax® XDB-Phenyl, 3.5 µm, 4.6 mm i.d. x 150 mm, Agilent Technologies Part Number: 963967-912, or equivalent; (3) Column heater was Agilent Model G1316A Column heater, set at 30^0C, or equivalent; (4) Detector was Agilent Model G1314B VWD, or equivalent; wavelength set at 250 nm; (5) Pump was Agilent G1316A Quat Pump, or equivalent; (6) Analytical Mode was isocratic program; (7) Time table: when time was 0 and 14.0, 0.025%TFA was 57 and 57, and acetonitrile was 43 and 43; (8) Flow rate: 0.8 mL/min; (9) Injection volume: 5µL; (10) Analysis time was 14 minutes; (11) Data reduction: Agilent EZchrom Elite, or equivalent; (12) Semi microanalytical Balance was Mettler Model XP205, or equivalent, capable of ± 0.1 mg readability

Preparation of Analytical Standard Solutions

a. Standard stock solutions: Accurately weigh about 200 mg of standard Sulfentrazone, 150 mg of standard Penoxsulam, and 400 mg of standard

Clomazone to the nearest 0.1 mg into a 40 ml glass vial, add about 20 grams of acetonitrile. Cap the vial and shake thoroughly to obtain a solution with concentration of about 10.0 mg of Sulfentrazone/g, 7.5 mg of Penoxsulam/g, and 20.0 mg of Clomazone/g, respectively.

b. Standard solution for linearity: Transfer a portion of the standard stock solution and add the same weight of water as the weight of the stock solution, then dilute with solvent A to prepare a set of six standard solutions, ranging 0.009 – 0.170 mg of Sulfentrazone/g, 0.0065 – 0.125 mg of Penoxsulam/g, and 0.020 – 0.350 mg of Clomazone/g, respectively.

Preparation of Sample Solutions

a. Granule sample preparation:
Accurately weigh 1.0 g of formulation sample, to the nearest 0.1 mg, into a 60 ml glass vial. Add 50.0 g of solvent A, cap the vial, sonicate and shake in water bath for 30 minutes, then cool to ambient temperature. Filter sample solution through a 0.45 μm membrane filter into auto-sampler vials and load the auto-sampler rack.
Note: Reduce the sample solution or dilute with solvent A as appropriate to obtain a solution having suitable concentration.
The sample solutions should be prepared at least in duplicate.

b. Sample preparation for release rate evaluation:
Weigh 5.0 g of granules and add them into a 250 ml conical flask that contains 100 g of tap water; shake the conical flask very slightly for 1-2 seconds and then let sit on a table. Measure the concentrations of the three active ingredients at the following times: 1, 3, 5, 7, 10, 14, 21, 28, 35, and 42 days. Before taking the solution for measurement, shake the conical flask very gently for 1-2 seconds. The flask should be covered to avoid water evaporation.
Draw 0.5 ml of solution into a syringe and filter it through a 0.22μm membrane filter into auto-sampler vials containing 100 μl of liner and load the auto-sampler rack.
If the concentration of a particular active ingredient does not change (by no greater than 1%) for three consecutive times, its measurement can be stopped.

Calculation

a. Determine the response factor (RF) of the active ingredient analytical standard from the standard injection:

$$RF = A_{STD}/C_{STD}$$

Where

A_{STD} = Area of the active ingredient standard

C_{STD} = Weight of the active ingredient standard in milligrams, adjusted for purity, and divided by the sum of weights of the active ingredient standard in grams plus solvent in grams (mg/g)

The weight percentage of an active ingredient is calculated for each sample solution as follows:

$$Wt\% = [(A_{SPL}/RF)/C_{SPL}] \times 100$$

Where

C_{SPL} = Weight of sample in milligrams divided by the weight of solvent A in grams (mg/g)

A_{SPL} = Area of the measurement peak of the sample solution

The following examples are the release profiles of our products.

Example 12: Release Profiles of Selected Formulas

Table 5. Controlled-Release Profile of 1.4% Herbicide Granule (0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone); F1 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	38.5	52.7	60.2	67.5	72.4	79.4	82.0	83.5	83.9	84.6
Penoxsulam	30.8	53.7	66.0	70.2	80.5	91.0	98.5	98.3	98.4	98.7
Clomazone	87.7	89.9	89.2	89.0	87.8	87.5	87.9	87.1		

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 6. Controlled-Release Profile of 2.1% Herbicide Granule (0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone); F2 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	39.1	50.2	63.5	68.4	71.4	78.5	79.2	81.5	84.7	88.6
Penoxsulam	35.3	57.1	70.5	73.5	84.3	95.5	96.2	96.8	98.5	100.5
Clomazone	88.1	89.5	89.4	90.1	91.3	90.7				

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 7. Controlled-Release Profile of 2.8% Herbicide Granule (0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone); F3 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	42.0	63.5	70.0	75.2	81.3	82.8	85.4	88.2	89.2	88.7
Penoxsulam	37.9	57.7	72.5	74.8	85.7	94.5	97.2	98.3	98.7	98.9
Clomazone	89.2	91.2	91.5	90.6	90.3					

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 8. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.2% Penoxsulam + 0.4% Clomazone); F28 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	28.0	41.5	48.9	61.0	65.0	74.5	76.2	80.2	83.4	83.7
Penoxsulam	37.0	61.6	72.3	84.2	89.1	95.4	97.7	97.5	98.7	99
Clomazone	92.5	89.4	88.5	87.0	87.2					

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 9. Controlled-Release Profile of 1.4% Herbicide Granule (0.2% Sulfentrazone + 0.4% Bispyribac-sodium + 0.8% Clomazone); F8 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	47	72	73	76	77	79	84	85	86	86
Bispyribac-sodium	99	100	100	101						
Clomazone	88	92	89	91	91					

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 10. Controlled-Release Profile of 0.7% Herbicide Granule (0.2% Sulfentrazone + 0.1% Bispyribac-sodium + 0.4% Clomazone); F29 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	32	48	50	61.8	63.8	66.1	68.8	71	72.3	
Bispyribac-sodium	100.7	101	100							
Clomazone	87.9	89.6	89	83	83.1	84.8	81.5			

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 11. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.2% Cyhalofop-butyl + 0.4% Clomazone); F9 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	22.3	30.6	37.9	38.0	41.5	44.9	60.2	63.8	68.3	
Cyhalofop-butyl	0.0	NA								
Clomazone	79.6	80.9	82.6	83	86	84.3	84.6			

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule; Cyhalofop's solubility is lower than instrument detection.

From Tables 12 – 16 below, we demonstrate the ability of using our formulation techniques to control the release of sulfentrazone at different rates recorded over a time span of 11 days. Having such ability yields a wide variety of effective applications.

Table 12. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.6% Clomazone); F21 in Table 4

	D1	D2	D3	D6	D8	D9	D11
Sulfentrazone	28.5	41.7	51.9	68.9	76.3	82.3	82.3
Clomazone	92.4	95.5	91.6	87.8	88.6	88.2	88.2

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 13. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.6% Clomazone); F22 in Table 4

	D1	D2	D3	D6	D8	D9	D11
Sulfentrazone	37.9	52.7	62.7	78.5	82.4	84.6	84.6
Clomazone	91.1	90.9	90.0	89.8	90.1	89.4	89.4

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 14. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.6% Clomazone); F23 in Table 4

	D1	D2	D3	D6	D8	D9	D11
Sulfentrazone	67.4	74.9	81.0	83.9	85.5	87.0	87.0
Clomazone	89.7	89.5	92.2	88.6	86.9	84.5	84.5

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 15. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.6% Clomazone); F24 in Table 4

	D1	D2	D3	D6	D8	D9	D11
Sulfentrazone	87.1	93.0	94.2	96.8	97.8	99.4	99.4
Clomazone	89.2	91.3	90.7	91.0	91.3	91.5	91.5

Note: D = Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 16. Controlled-Release Profile of 0.8% Herbicide Granule (0.2% Sulfentrazone + 0.6% Clomazone); F25 in Table 4

	D1	D2	D5	D8	D11
Sulfentrazone	93.1	98.8	99.9	99.2	99.9
Clomazone	93.1	91.7	92.2	90.7	89.1

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 17. Controlled-Release Profile of 0.4% Herbicide Granule (0.4% Sulfentrazone); F20 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	35.0	44.9	53.6	62.4	70.3	73.5	78.8	80.2	84.6	

Note: D=Day; released is the weight % of the active ingredient with respect to the total in the granule.

Table 18. Controlled-Release Profile of 0.45% Herbicide Granule (0.4% Sulfentrazone + 0.05% Azimsulfuron); F26 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	52.7	68.3	77.1	81.2	83.6	85.8	88.9	89.5	88.6	
Azimsulfuron	103.6	102.3	101.8							

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 19. Controlled-Release Profile of 0.47% Herbicide Granule (0.4% Sulfentrazone + 0.07% Flucetosulfuron); F27 in Table 4

	D1	D3	D5	D7	D10	D14	D21	D28	D35	D42
Sulfentrazone	46.6	71.5	73.4	76.8	79.3	80.1	84.2	85.3	86.4	
Flucetosulfuron	85.3	89.9	95.6	98.9	98.4	98.8				

Note: D=Day; released is the weight % of each active ingredient with respect to its total in the granule.

Table 20. Fast-Release Profile of 0.60% Herbicide Granule (0.45% Sulfentrazone + 0.15% Penoxsulam); F30 in Table 4

	D1	D4	D5	D8	D11	D18	D21	D25	D27	D28	D32	D35
Sulfentrazone	57.6	74.9	81.5	86.7	87.7	90.6	93.7	98.8	99.7	98.2	98.7	98.8
Penoxsulam	33.9	94	105.6									

Note: D=Day; released is the weight % of each active ingredients with respect to its total in the granule.

Table 21. Controlled-Release Profile of 20% Sulfentrazone WP; F19 in Table 4

	2H	D1	D3	D5	D7	D10	D14
Sulfentrazone	62	75	82	86	88	88	87

Note: D=Day; released is the weight % of the active ingredients with respect to the total in the granule; H=hours.

Biological Results

We report here the results of our field trials conducted in the following countries: Philippines, Thailand, China, and South Korea, thusly both hot-climate and temperate rice regions are represented. Some of the trials in the Philippines were conducted during both the rainy season and dry season.

All field trials were carried out by independent contracted research organizations. Prior to each field trial, we discussed with the contracted organization to collectively design a test protocol to evaluate the claims of our products. Because different testing organizations have their own rice-farming practices, both the testing party and we agreed that our samples were to be applied in accordance with the local farmers' best practices, namely the local rice varieties, cultivation methods, commercial standards, number of applications, application timings, etc. No restrictions were set as to how the comparison was to be made.

Example 13: Phytotoxic Effect on Rice of Spartan® and Controlled-Release Sulfentrazone

Spartan® and three granules with different sulfentrazone release profiles were tested in the field in Hainan, China. The purpose was to assess the degree of phytotoxicity in rice caused by different amounts of sulfentrazone at any given time. The release rates of sulfentrazone from Spartan® and the three granules at the end of 24 hours were determined to be 100%, 77%, 57% and 33%, respectively, in the laboratory under undisturbed conditions at ambient temperature (~25°C). In fact, Spartan®'s 100% sulfentrazone release occurred in less than 2 hours. The higher the release rate, the more of sulfentrazone is released at any given time.

The different release rates were obtained through adjustment of the polymer content (cellulose acetate butyrate), from the fastest to the slowest as follows: 0.28%, 0.16%, and 0.18% polymer by weight, while the particle size range for all three samples was held fixed at 355-420 µm. Sulfentrazone content percentage by weight for the three granule samples with release rates of 77%, 57%, and 33% were 0.6%, 0.3%, and 0.3%, respectively.

The amount of granules for each sample, equivalent to 120 g ai/ha of sulfentrazone, was determined based on the active ingredient loading (%) in the sample.

The crop was transplanted Indica rice with 28-day old rice seedlings and the application timing was 10 DAT. Each plot size was 5 M^2. Each treatment had three replications.

This field trial was a phytotoxicity test only and no efficacy of weed control was assessed. The rice injury scale was 0-100%, 0% denoting no injury and 100% denoting all rice dead.

The phytotoxicity survey was stopped at 7 DAA because (1) Spartan® and the 77% release rate caused unacceptable initial injury (Table 22) and (2) 57% and 33% release rates would not further cause injury in rice if no phytotoxic symptoms emerged during the first 7 days after application (Table 22 and Figure 2). It should be noted that the 24-h release rate of sulfentrazone is one but not the only predictive factor of injury to rice.

Table 22. Effect of Controlled-Release Sulfentrazone Granules and Spartan on Rice Seedlings at Different Assessment Times

Treatment			Injury to Rice Seedlings (%)			
No	Total Sulfentrazone (g ai/ha)	24-h Release Rate	1 DAA	2 DAA	3 DAA	7 DAA
1	120	100% (Spartan®)	0	6	12	48
2	120	77.0%	0	7.5	10.5	39
3	120	57.0%	0	0	0	1.5
4	120	33.0%	0	0	0	0

Example 14: Performance Comparison between Three Granule Samples (1.4% AI; 2.1% AI; 2.8% AI) and Two Standards in Direct-Seeded Rice

In this trial, the active ingredients in each of the three granule samples were sulfentrazone, penoxsulam, and clomazone. The total content of the active ingredients in the samples were 1.4%, 2.1% and 2.8%, respectively. Their formulas are listed in Table 4 as F1, F2, and F3.

SynTech conducted this trial at its research station in Cabanatuan City, Philippines. The crop was direct-seeded Indica rice and the application timing was 10 DAS.

To ensure all hot-climate rice major weeds were present at a meaningful infestation density, weed seeds (*Echinochloa crus-galli, Leptochloa chinensis, Ischaemum rugosum, Monochoria vaginalis, Sphenoclea zeylanica, Ludwigia octovalvis, Cyperus difformis, Fimbristylis miliacea*, and *Cyperus iria*) were introduced to the field all at 2 kg/ha, except *Ischaemum rugosum* which was at 6 kg/ha, in addition to their natural occurrence. In priming weed seeds, only soaking of all weed seeds for 24 hours was done. Primed weed seeds were thoroughly mixed with fine and dry soil for ease of broadcasting by hand. Indica rice (NSIC Rc216) was seeded at 40 kg/ha. Pre-germinated rice and the weed seeds were hand-broadcast into trial plots. Soil was wet but with no standing water at seeding.

Rice and weed germination and growth were normal, except for *Monochoria vaginalis* which did not germinate, and by application day (10 DAS) the overall weed pressure was extremely high. Detailed rice and weeds data are listed in Table 24. The leaf stage and height of the rice and weeds were measured at 9 DAS rather than 10 DAS since such measurements concurrent with herbicide treatment application was not feasible.

The trial followed a randomized complete block design (RCBD). Each block consisted of 22 plots wherein treatments were randomly assigned, and was separated by a 1-meter wide canal used for irrigation and drainage. Each plot size was 20 M^2 (4.0 m x 5.0 m) and plots were separated by earthen levees. Each treatment had four replications.

Test granules were hand-broadcast into the plots at 10 DAS. The commercial standards 'Nominee 100SC' (Bispyribac-sodium) and 'Pyzero 10EC' (Metamifop) were applied by foliar spray at 15 DAS according to their labels, followed by 'Clincher 100EC' (Cyhalofop-butyl) at 28 DAS and '2,4-D' at 39 DAS, respectively. A knapsack sprayer fitted with a hollow cone nozzle was used for applying the herbicide standards. Spray volume was 200 L/ha. Spray guards were used during applications to prevent possible spray drifts to adjacent plots.

Water depth was about 0.5 cm at the time of application of the three granule samples and was maintained for three days after the application. It was then raised to 1-3 cm at 4 to 10 DAA and maintained at 3-5 cm thereafter.

In plots sprayed with the standard herbicides, there was no standing water at time of spraying, water depth of 3 cm was introduced to the plots at 3 DAA and maintained at 3-5 cm at 11 DAA and thereafter.

Preventive measures were taken to minimize potential error, including bird scarers. Four spray rounds of Niclosamide 70 WP (molluscicide) at 4, 10, 15, and 17 DAS were undertaken against golden apple snail, *Pomacea canaliculata*. The following insecticides and fungicides were used at their label recommendation at the following times: 'Furadan 3G' (carbofuran) at 10 DAS; 'Actara 25WG' (thiamethoxam) at 14 DAS; 'Bigboss' (lambda-cyhalothrin) at 20 DAS; 'Applaud 10WP + Philtap' 50SP' (buprofezin + Cartap hydrochloride) at 25 & 32 DAS; 'Chix 2.5 EC + Applaud 10WP' (beta-cypermethrin + buprofezin) at 42 DAS; 'Benophyl 50WP' (benomyl) at 43 DAS; 'Score 250EC' (difenoconazole) at 50 & 57 DAS. In addition, the recommended nutrient management was carried out as follows: 120-40-40 kg each of NPK/ha was applied on three split occasions – basal at 40 kg each of NPK/ha at 14 DAS, followed by 40 kg N/ha at 28 and 54 DAS. (The trial was continued in order to measure rice yield as well as residual activity after rice harvest by planting and observing new sensitive crops in the same field.)

The following data were gathered: seedling injury, based on seedling stand and appearance in reference to the untreated, was observed at 3, 8, and 15 DAA (surveyed at 8 and 15 DAA rather than 7 and 14 DAA because the latter fell on weekends); plant height was measured and the number of tillers was counted, both based on 10 sample hills per plot, at 36 DAA (46 DAS) and 43 DAA (53 DAS); weed control by species per plot was assessed at 8, 15, 22, 29, and 43 DAA; weed population by species was counted at 43 DAA sampled from four 0.25 M^2 quadrats each placed in four corners of a plot at a distance of 50 cm away from levees; data were analyzed using ARM; weather data was taken from the Philippine Atmospheric, Geophysical and Astronomical Services Administration (PAGASA) in Cabanatuan City, which is about 3.2 km away from the SynTech field station.

Two sets of commercial rice herbicide products, Nominee 100SC + Clincher 100EC and Pyzero 10EC + 2,4-D IBE, were used to compare with the three granule samples. SynTech recommended using these two sets of rice herbicides because they are the top choices locally. The amount, application order, and timing of the standards were followed strictly according to their labels.

For each test granule sample, there were six use rates ranging from 190 g ai/ha to 760 g ai/ha. There were also two controls – one was un-weeded and the other was hand-weeded. Detailed information about test samples, standards, controls, use rates, plot size, and application timing is summarized in Table 23.

Table 23. Use Rates of Granule Samples and Standards and Application Timings

Treatment				Requirements g/plot (20 M^2)
No	Product	Rate (g ai/ha)	Application Timing	
1	SPC 1	190	10 DAS	28.15
2	SPC 1	260	10 DAS	38.52
3	SPC 1	320	10 DAS	47.41
4	SPC 1	380	10 DAS	56.30
5	SPC 1	640	10 DAS	94.81
6	SPC 1	760	10 DAS	112.59
7	SPC 4	190	10 DAS	18.45
8	SPC 4	260	10 DAS	25.24
9	SPC 4	320	10 DAS	31.07
10	SPC 4	380	10 DAS	36.89
11	SPC 4	640	10 DAS	62.14
12	SPC 4	760	10 DAS	73.79
13	SPC 5	190	10 DAS	12.97
14	SPC 5	260	10 DAS	17.75
15	SPC 5	320	10 DAS	21.84
16	SPC 5	380	10 DAS	25.94
17	SPC 5	640	10 DAS	43.69
18	SPC 5	760	10 DAS	51.88
19	Nominee fb Clincher	25 fb 100	15 DAS fb 28 DAS**	
20	Pyzero fb 2,4-D	100 fb 400*	15 DAS fb 39 DAS***	
21	Hand-weeded	-	14 & 28 DAS	
22	Control/Un-weeded	-	-	

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;
fb: followed by;
*: 400 g acid; **: equivalent to 13 days after application (DAA) of 'Nominee'; ***: equivalent to 24 DAA

of ‘Pyzero’;
‘Nominee 100SC’ (Byspiribac-sodium);
‘Clincher 100EC’ (Cyhalofop-butyl);
‘Pyzero 10EC’ (Metamifop).

Table 24. Average Leaf Stage and Average Height of Rice and Major Weeds

No	Rice/Weed	Seeded Rate* (kg/ha)	Plant Height (cm)	Leaf Stage
1	Rice (NSIC Rc216)	40	14.8	3.0 – 3.5
2	*Echinochloa crus-galli*	2	6.8	3.0 – 3.5
3	*Leptochloa chinensis*	2	1.8	3
4	*Ischaemum rugosum*	6	3.0	2.0 – 3.0
5	*Monochoria vaginalis*	2	-	-
6	*Sphenoclea zeylanica*	2	0.4	4
7	*Ludwigia octovalvis*	2	0.4	4
8	*Cyperus difformis*	2	1.1	3
9	*Fimbristylis miliacea*	2	1.4	4
10	*Cyperus iria*	2	1.1	3

*: In addition to their natural occurrence, the nine weeds were also introduced to ensure their presence and the density;
Monochoria vaginalis did not germinate due to lack of water during the first 10 days; the introduction of water during the first 10 days would have threatened the germination of *Leptochloa chinensis* and *Ischaemum rugosum*; *Cyperus iria* on 9 DAS was difficult to differentiate from *Cyperus difformis* as both looked alike.

Table 25. Effect of Three Granules and Standards on Rice Seedlings at Different Assessment Timings

Treatment			Injury to Rice Seedlings (%)				
No	Product	Rate (g ai/ha)	3 DAA	8 DAA	15 DAA	22 DAA	29 DAA
1	SPC 1	190	0 a	0 a	0 a		
2	SPC 1	260	0 a	0 a	0 a		
3	SPC 1	320	0 a	0 a	0 a		
4	SPC 1	380	0 a	0 a	0 a		
5	SPC 1	640	0 a	0 a	0 a		
6	SPC 1	760	0 a	0 a	0 a		
7	SPC 4	190	0 a	0 a	0 a		
8	SPC 4	260	0 a	0 a	0 a		
9	SPC 4	320	0 a	0 a	0 a		
10	SPC 4	380	0 a	0 a	0 a		
11	SPC 4	640	0 a	0 a	0 a		
12	SPC 4	760	0 a	0 a	0 a		
13	SPC 5	190	0 a	0 a	0 a		
14	SPC 5	260	0 a	0 a	0 a		
15	SPC 5	320	0 a	0 a	0 a		
16	SPC 5	380	0 a	0 a	0 a		
17	SPC 5	640	0 a	0 a	0 a		
18	SPC 5	760	0 a	0 a	0 a		
19	Nominee fb Clincher	25 fb 100	0 a	0 a	0 a		
20	Pyzero fb 2,4-D	100 fb 400	0 a	0 a	0 a		
21	Hand-weeded	-	0 a	0 a	0 a		
22	Control/Unweeded	-	0 a	0 a	0 a		

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;

In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 26. Effect of Three Granules and Standards on Plant Height and Tiller Number Taken at Different Timings

Treatment			Plant Height (cm)		Tiller Count	
No	Product	Rate (g ai/ha)	36 DAA	43 DAA	36 DAA	43 DAA
1	SPC 1	190	72.9 a	75.8 a	13.7 a	13.7 a
2	SPC 1	260	75.2 a	78.2 a	13.7 a	13.6 a
3	SPC 1	320	70.8 a	78.3 a	13.4 a	13.5 a
4	SPC 1	380	72.8 a	76.9 a	13.2 a	13.0 a
5	SPC 1	640	70.4 a	77.5 a	12.6 a	12.6 a
6	SPC 1	760	75.5 a	76.9 a	13.7 a	13.7 a
7	SPC 4	190	74.4 a	77.8 a	13.4 a	13.4 a
8	SPC 4	260	75.1 a	78.0 a	14.5 a	14.2 a
9	SPC 4	320	74.6 a	79.5 a	14.0 a	14.0 a
10	SPC 4	380	75.9 a	79.5 a	14.2 a	14.3 a
11	SPC 4	640	75.5 a	78.9 a	13.5 a	13.5 a
12	SPC 4	760	70.5 a	80.3 a	12.6 a	12.6 a
13	SPC 5	190	71.9 a	79.4 a	13.2 a	13.0 a
14	SPC 5	260	70.6 a	79.4 a	13.0 a	12.6 a
15	SPC 5	320	71.2 a	80.1 a	13.9 a	13.9 a
16	SPC 5	380	72.9 a	80.5 a	12.9 a	12.9 a
17	SPC 5	640	75.2 a	79.2 a	12.8 a	13.0 a
18	SPC 5	760	72.6 a	79.5 a	12.5 a	12.5 a
19	Nominee fb Clincher	25 fb 100	73.6 a	79.3 a	13.5 a	13.5 a
20	Pyzero fb 2,4-D	100 fb 400	72.5 a	78.1 a	12.6 a	12.6 a
21	Hand-weeded	-	73.6 a	78.1 a	13.0 a	13.1 a
22	Control/Unweeded	-	76.5 a	79.1 a	12.8 a	12.8 a

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

SynTech reported no phytotoxic indications on rice seedlings for any of the three granule samples during the scheduled surveys at 3, 8, and 15 DAA, even at the highest rate of 760 g ai/ha. Since there was no phytotoxicity in rice for the first two weeks, the surveys at 22 and 29 DAA have been omitted. Rice plant heights and tillers in plots treated with the granule samples were within normal range of the heights and tillers of plots that were hand-weeded. Nor was any phytotoxicity observed from the two commercial standards.

Tables 27–36 contain the efficacy information to all weed control and to major weed control by species at 8, 15, 22, 29, and 43 DAA, respectively (7, 14, 21, 28, and 42 DAA fell on weekends).

Against all weeds, each of the three test granules (SPC 1, SPC 4 and SPC 5), even at the low rate of 260 g ai/ha, showed the same 95% control efficacy at 15 DAA, compared to only 83% for Nominee and 65% for Pyzero, respectively. Unlike the test granules, each of which controlled grasses, broadleaves, and sedges with a single application, Nominee and Pyzero both required a second application of other herbicides to target some major weeds against which the standards lack sufficient intrinsic activity. That is, Nominee needed a different grass-control herbicide, in this case Clincher, to kill *Leptochloa chinensis*, a major yield-reduction grass weed. Pyzero required an additional broadleaves and sedge-control herbicide, in this case 2,4-D, to control those weeds.

Against the three major target grasses (*Echinochloa crus-galli, Leptochloa chinensis*, and *Ischaemum rugosum*) as a group, the three test granules, each at 260 g ai/ha, provided average control efficacies of 89%, 92%, and 93% at 15 DAA, respectively, as compared to 77% at 15 DAA for Nominee, and 95% at 15 DAA for Pyzero. Pyzero is a grass-control herbicide and is able to manage these three grasses. However, Nominee cannot control *Leptochloa chinensis* as depicted by the low average of 77% (Tables 28-30).

Against the two major target broadleaves (*Sphenoclea zeylanica* and *Ludwigia octovalvi*) as a group, all three granules, each at 260 g ai/ha, provided the same average control efficacy of 98% at 15 DAA, as compared to 97% at 15 DAA for Nominee, and 51% at 15 DAA for Pyzero (Tables 31-32). The grass-control herbicide Pyzero cannot control these two broadleaves.

Against the three major target sedges (*Cyperus difformis, Fimbristylis miliacea,* and *Cyperus iria*) as a group, all three test samples, each at 260 g ai/ha, showed the same average control efficacy of 98% at 15 DAA, as compared to 95% at 15 DAA for Nominee, and 20% at 15 DAA for Pyzero (Tables 33-35). In addition to broadleaves, Pyzero also cannot control these three sedges.

When Nominee was followed by an application of another herbicide Clincher, the overall weed control and the average control of the three categories of weeds was comparable to that of each of the three one-shot test granules (93-100%). Even after Pyzero was followed by an application of 2,4-D, the overall weed control and the control to broadleaves and sedges were still inferior to that of the three test granules and the Nominee+Clincher combination.

Not only was the trial conducted in the rice field and not the lab, but also the dense and uniform distribution of the 8 aforementioned major weeds with their artificial introduction compounds and far exceeds the severity of any possible real world scenario. The test results under such extreme conditions lend confidence to the product's claims of unique safe performance, fast-acting, and sustainably preventive, against the three major weed varieties.

Table 27. Overall Weed Control/Plot at Different Assessment Timings

Treatment	Overall Weed Control (%)

No	Product	Rate (g ai/ha)	8 DAA	15 DAA	22 DAA	29 DAA	43 DAA
1	SPC 1	190	91.0 b	93.8 a	93.8 ab	95.8 ab	93.8 ab
2	SPC 1	260	90.5 b	95.0 a	95.0 ab	95.8 ab	96.5 ab
3	SPC 1	320	92.8 b	95.0 a	95.0 ab	96.5 ab	95.8 ab
4	SPC 1	380	93.5 b	95.8 a	95.0 ab	95.8 ab	96.5 ab
5	SPC 1	640	93.0 b	96.5 a	95.8 ab	96.5 ab	94.0 ab
6	SPC 1	760	95.0 b	96.5 a	96.5 ab	97.3 ab	98.5 ab
7	SPC 4	190	90.5 b	95.0 a	95.0 ab	95.0 ab	98.0 ab
8	SPC 4	260	91.0 b	95.0 a	95.0 ab	95.8 ab	97.3 ab
9	SPC 4	320	92.0 b	94.3 a	93.8 ab	95.0 ab	95.8 ab
10	SPC 4	380	91.8 b	95.0 a	95.0 ab	95.8 ab	95.8 ab
11	SPC 4	640	92.8 b	95.0 a	95.0 ab	95.8 ab	95.8 ab
12	SPC 4	760	91.8 b	95.0 a	95.8 ab	97.3 ab	96.5 ab
13	SPC 5	190	92.0 b	95.0 a	91.3 b	92.8 b	95.0 ab
14	SPC 5	260	91.0 b	95.0 a	95.0 ab	95.0 ab	96.3 ab
15	SPC 5	320	91.0 b	93.8 a	95.0 ab	95.8 ab	95.8 ab
16	SPC 5	380	91.5 b	95.0 a	95.8 ab	96.5 ab	97.8 ab
17	SPC 5	640	93.0 b	95.8 a	95.8 ab	95.8 ab	95.8 ab
18	SPC 5	760	94.3 b	96.5 a	95.8 ab	97.8 ab	93.8 ab
19	Nominee fb Clincher	25 fb 100	85.0 c	82.5 b	87.5[A] c	95.8[B] ab	95.0[C] ab
20	Pyzero fb 2,4-D	100 fb 400	57.5 d	65 c	63.8 d	73.8[D] c	92.5[E] b
21	Hand-weeded	-	100 a	100 a	100 a	100 a	100 a
22	Control/Unweeded	-	0 e	0 d	0 e	0 d	0 c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;
A: 9 days after application of ‘Clincher’; B: 16 days after application of ‘Clincher’; C: 30 days after application of ‘Clincher’;
D: 5 days after application of ‘2,4-D’; E: 19 days after application of ‘2,4-D’;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

<u>Grasses: *Echinochloa crus-galli, Leptochloa chinensis, and Ischaemum rugosum* (Tables 28-30)</u>

Table 28. Control of *Echinochloa crus-galli* at Different Assessment Timings

Treatment			Control of *Echinochloa crus-galli* (%)				
No	Product	Rate (g ai/ha)	8 DAA	15 DAA	22 DAA	29 DAA	43 DAA
1	SPC 1	190	85.0 cd	86.3 c	93.8 b	95.3 a	95.3 a
2	SPC 1	260	83.8 d	88.8 bc	95.8 ab	97.5 a	97.5 a
3	SPC 1	320	91.3 a-d	93.8 abc	96.5 ab	99.5 a	99.5 a
4	SPC 1	380	92.5 a-d	93.8 abc	95.8 ab	98.3 a	98.3 a
5	SPC 1	640	92.5 a-d	95.8 ab	98.0 ab	99.5 a	99.5 a
6	SPC 1	760	92.0 a-d	96.5 ab	97.3 ab	100 a	100 a
7	SPC 4	190	92.5 a-d	92.5 abc	95.8 ab	99.0 a	99.0 a
8	SPC 4	260	88.8 bcd	93.8 abc	96.5 ab	97.5 a	97.5 a
9	SPC 4	320	93.8 a-d	92.5 abc	96.5 ab	98.3 a	98.3 a
10	SPC 4	380	92.5 a-d	95.8 ab	96.5 ab	100 a	100 a
11	SPC 4	640	93.8 a-d	96.5 ab	98.0 ab	100 a	100 a
12	SPC 4	760	94.5 a-d	96.5 ab	98.0 ab	99.5 a	99.5 a
13	SPC 5	190	91.3 a-d	92.5 abc	93.3 b	92.0 a	92.0 a

14	SPC 5	260	90.0	a-d	93.8	abc	96.5	ab	98.3	a	98.3	a
15	SPC 5	320	92.5	a-d	95.8	ab	97.3	ab	98.8	a	98.8	a
16	SPC 5	380	95.0	abc	96.5	ab	97.3	ab	98.8	a	98.8	a
17	SPC 5	640	95.8	abc	97.3	ab	98.0	ab	98.8	a	98.8	a
18	SPC 5	760	96.5	ab	97.3	ab	98.0	ab	99.5	a	99.5	a
19	Nominee fb Clincher	25 fb 100	92.5	a-d	97.3	ab	98.0[A]	ab	99.5[B]	a	100[C]	a
20	Pyzero fb 2,4-D	100 fb 400	93.8	a-d	96.0	ab	96	ab	97.3[D]	a	98.0[E]	a
21	Hand-weeded	-	100	a	100	a	100	a	100	a	100	a
22	Control/Unweeded	-	0	e	0	d	0	c	0	b	0	b

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 29. Control of *Leptochloa chinensis* at Different Assessment Timings

Treatment			Control of *Leptochloa chinensis* (%)									
No	Product	Rate (g ai/ha)	8 DAA		15 DAA		22 DAA		29 DAA		43 DAA	
1	SPC 1	190	76.3	c	77.5	b	77.5	b	83.8	ab	85.8	a
2	SPC 1	260	82.5	bc	83.8	ab	85.0	ab	86.3	ab	86.3	a
3	SPC 1	320	86.3	bc	86.3	ab	87.5	ab	89.5	ab	89.5	a
4	SPC 1	380	86.3	bc	86.3	ab	86.3	ab	86.3	ab	86.3	a
5	SPC 1	640	88.8	abc	90.8	ab	90.0	ab	90.0	ab	90.0	a
6	SPC 1	760	93.3	ab	92.0	ab	94.0	ab	94.0	ab	94.8	a
7	SPC 4	190	86.3	bc	86.3	ab	82.5	ab	86.3	ab	86.3	a
8	SPC 4	260	88.8	abc	87.5	ab	91.3	ab	91.3	ab	91.3	a
9	SPC 4	320	87.5	abc	87.5	ab	86.3	ab	87.5	ab	87.5	a
10	SPC 4	380	87.5	abc	87.5	ab	88.8	ab	88.8	ab	88.8	a
11	SPC 4	640	90.0	ab	91.3	ab	90.0	ab	91.3	ab	91.3	a
12	SPC 4	760	90.0	ab	92.0	ab	93.3	ab	94.8	ab	94.8	a
13	SPC 5	190	87.5	abc	85.0	ab	86.3	ab	87.8	ab	87.8	a
14	SPC 5	260	87.5	abc	87.5	ab	88.8	ab	88.8	ab	88.8	a
15	SPC 5	320	87.5	abc	90.8	ab	90.0	ab	89.5	ab	89.5	a
16	SPC 5	380	88.8	abc	88.8	ab	90.0	ab	92.0	ab	92.0	a
17	SPC 5	640	90.0	ab	93.8	ab	94.5	ab	94.5	ab	94.5	a
18	SPC 5	760	91.3	ab	93.3	ab	94.5	ab	97.0	ab	97.0	a
19	Nominee fb Clincher	25 fb 100	55.0	d	37.5	c	32.5[A]	c	81.3[B]	b	94.5[C]	a
20	Pyzero fb 2,4-D	100 fb 400	93.8	ab	92.8	ab	92.8	ab	92.8[D]	ab	92.8[E]	a
21	Hand-weeded	-	100	a	100	a	100	a	100	a	100	a
22	Control/Unweeded	-	0	e	0	d	0	d	0	c	0	b

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 30. Control of *Ischaemum rugosum* at Different Assessment Timings

Treatment			Control of *Ischaemum rugosum* (%)				
No	Product	Rate (g ai/ha)	8 DAA	15 DAA	22 DAA	29 DAA	43 DAA
1	SPC 1	190	75.0 c	86.3 b	95.3 b	97.3 ab	97.3 ab
2	SPC 1	260	81.3 bc	93.3 ab	96.0 ab	96.0 b	96.0 b
3	SPC 1	320	86.3 abc	95.3 ab	97.3 ab	98.0 ab	98.0 ab
4	SPC 1	380	87.5 abc	95.3 ab	97.3 ab	98.0 ab	98.0 ab
5	SPC 1	640	88.8 abc	95.8 ab	98.0 ab	98.0 ab	98.0 ab
6	SPC 1	760	93.3 ab	98.0 a	98.0 ab	98.0 ab	98.0 ab
7	SPC 4	190	88.8 abc	95.8 ab	98.0 ab	97.3 ab	97.3 ab
8	SPC 4	260	87.5 abc	93.3 ab	96.0 ab	97.3 ab	97.3 ab
9	SPC 4	320	90.0 abc	94.5 ab	98.0 ab	98.0 ab	98.0 ab
10	SPC 4	380	88.8 abc	96.5 a	98.0 ab	98.0 ab	98.0 ab
11	SPC 4	640	91.3 abc	97.3 a	98.0 ab	98.0 ab	98.0 ab
12	SPC 4	760	91.3 abc	97.3 a	98.0 ab	98.0 ab	98.0 ab
13	SPC 5	190	86.3 abc	93.8 ab	96.0 ab	96.0 b	96.0 b
14	SPC 5	260	88.8 abc	96.5 a	98.0 ab	98.0 ab	98.0 ab
15	SPC 5	320	88.8 abc	96.5 a	98.0 ab	98.0 ab	98.0 ab
16	SPC 5	380	88.8 abc	98.0 a	98.0 ab	98.0 ab	98.0 ab
17	SPC 5	640	92.5 ab	98.0 a	98.0 ab	98.0 ab	98.0 ab
18	SPC 5	760	91.3 abc	98.0 a	98.0 ab	98.5 ab	98.5 ab
19	Nominee fb Clincher	25 fb 100	92.5 ab	97.3 a	98.0[A] ab	98.5[B] ab	98.5[C] ab
20	Pyzero fb 2,4-D	100 fb 400	93.8 ab	96.0 ab	96.0 ab	97.3[D] ab	97.3[E] ab
21	Hand-weeded	-	100 a	100 a	100 a	100 a	100 a
22	Control/Unweeded	-	0 d	0 c	0 c	0 c	0 c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application; DAS: Days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Broadleaves: *Sphenoclea zeylanica* and *Ludwigia octovalvi* (Tables 31-32)

Table 31. Control of *Sphenoclea zeylanica* at Different Assessment Timings

Treatment			Control of *Sphenoclea zeylanica* (%)				
No	Product	Rate (g ai/ha)	8 DAA	15 DAA	22 DAA	29 DAA	43 DAA
1	SPC 1	190	97.3 ab	98.0 ab	99.5 a	99.5 a	99.5 a
2	SPC 1	260	98.0 ab	98.0 ab	99.5 a	100 a	100 a
3	SPC 1	320	98.0 ab	98.0 ab	100 a	100 a	100 a
4	SPC 1	380	98.0 ab	98.0 ab	100 a	100 a	100 a
5	SPC 1	640	98.0 ab	98.0 ab	100 a	100 a	100 a
6	SPC 1	760	98.0 ab	98.0 ab	100 a	100 a	100 a
7	SPC 4	190	97.3 ab	98.0 ab	100 a	100 a	100 a
8	SPC 4	260	98.0 ab	98.0 b	100 a	100 a	100 a
9	SPC 4	320	98.0 ab	98.0 ab	100 a	100 a	100 a
10	SPC 4	380	98.0 ab	98.0 ab	100 a	100 a	100 a

11	SPC 4	640	98.0	ab	98.0	ab	100	a	100	a	100	a
12	SPC 4	760	98.0	ab	98.0	ab	100	a	100	a	100	a
13	SPC 5	190	98.0	ab	98.0	ab	100	a	99.0	a	100	a
14	SPC 5	260	98.0	ab	98.0	ab	100	a	100	a	100	a
15	SPC 5	320	98.0	ab	98.0	ab	100	a	100	a	100	a
16	SPC 5	380	98.0	ab	98.0	ab	100	a	100	a	100	a
17	SPC 5	640	98.0	ab	98.0	ab	100	a	100	a	100	a
18	SPC 5	760	98.0	ab	98.0	ab	100	a	100	a	100	a
19	Nominee fb Clincher	25 fb 100	96.5	b	97.3	ab	99.5^{A}	a	99.5^{B}	a	99.5^{C}	a
20	Pyzero fb 2,4-D	100 fb 400	32.5	c	52.5	c	52.5	b	68.8^{D}	b	92.5^{E}	b
21	Hand-weeded	-	100	a	100	a	100	a	100	a	100	a
22	Control/Unweeded	-	0	d	0	d	0	c	0	c	0	c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: days after application; DAS: days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 32. Control of *Ludwigia octovalvis* at Different Assessment Timings

Treatment			Control of *Ludwigia octovalvis* (%)									
No	Product	Rate (g ai/ha)	8 DAA		15 DAA		22 DAA		29 DAA		43 DAA	
1	SPC 1	190	97.3	ab	98.0	a	99.5	a	99.5	a	99.5	a
2	SPC 1	260	98.0	ab	98.0	a	99.5	a	99.5	a	99.5	a
3	SPC 1	320	98.0	ab	98.0	a	100	a	100	a	100	a
4	SPC 1	380	98.0	ab	98.0	a	100	a	100	a	100	a
5	SPC 1	640	98.0	ab	98.0	a	100	a	100	a	100	a
6	SPC 1	760	98.0	ab	98.0	a	100	a	100	a	100	a
7	SPC 4	190	97.3	ab	98.0	a	100	a	100	a	100	a
8	SPC 4	260	98.0	ab	98.0	a	100	a	100	a	100	a
9	SPC 4	320	98.0	ab	98.0	a	100	a	100	a	100	a
10	SPC 4	380	98.0	ab	98.0	a	100	a	100	a	100	a
11	SPC 4	640	98.0	ab	98.0	a	100	a	100	a	100	a
12	SPC 4	760	98.0	ab	98.0	a	100	a	100	a	100	a
13	SPC 5	190	98.0	ab	98.0	a	100	a	100	a	100	a
14	SPC 5	260	98.0	ab	98.0	a	100	a	100	a	100	a
15	SPC 5	320	98.0	ab	98.0	a	100	a	100	a	100	a
16	SPC 5	380	98.0	ab	98.0	a	100	a	100	a	100	a
17	SPC 5	640	98.0	ab	98.0	a	100	a	100	a	100	a
18	SPC 5	760	98.0	ab	98.0	a	100	a	100	a	100	a
19	Nominee fb Clincher	25 fb 100	96.5	b	97.3	a	99.5^{A}	a	98.8^{B}	a	99.5^{C}	a
20	Pyzero fb 2,4-D	100 fb 400	32.5	c	50.0	b	50.0	b	65.0^{D}	b	80.0^{E}	b
21	Hand-weeded	-	100	a	100	a	100	a	100	a	100	a
22	Control/Unweeded	-	0	d	0	c	0	c	0	c	0	c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: days after application; DAS: days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';

In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Sedges: *Cyperus difformis*, *Fimbristylis miliacea*, and *Cyperus iria* (Tables 33-35)

Table 33. Control of *Cyperus difformis* at Different Assessment Timings

Treatment			Control of *Cyperus difformis* (%)									
No	Product	Rate (g ai/ha)	8 DAA		15 DAA		22 DAA		29 DAA		43 DAA	
1	SPC 1	190	97.3	ab	98.0	a	99.5	a	99.5	a	99.5	a
2	SPC 1	260	97.3	ab	98.0	a	99.5	a	100	a	100	a
3	SPC 1	320	97.3	ab	98.0	a	100	a	100	a	100	a
4	SPC 1	380	98.0	ab	98.0	a	100	a	100	a	100	a
5	SPC 1	640	98.0	ab	98.0	a	100	a	100	a	100	a
6	SPC 1	760	98.0	ab	98.0	a	100	a	100	a	100	a
7	SPC 4	190	97.3	ab	98.0	a	100	a	100	a	100	a
8	SPC 4	260	98.0	ab	98.0	a	100	a	100	a	100	a
9	SPC 4	320	98.0	ab	98.0	a	100	a	100	a	100	a
10	SPC 4	380	98.0	ab	98.0	a	100	a	100	a	100	a
11	SPC 4	640	98.0	ab	98.0	a	100	a	100	a	100	a
12	SPC 4	760	98.0	ab	98.0	a	100	a	100	a	100	a
13	SPC 5	190	98.0	ab	98.0	a	100	a	100	a	100	a
14	SPC 5	260	98.0	ab	98.0	a	100	a	100	a	100	a
15	SPC 5	320	98.0	ab	98.0	a	100	a	100	a	100	a
16	SPC 5	380	98.0	ab	98.0	a	100	a	100	a	100	a
17	SPC 5	640	98.0	ab	98.0	a	100	a	100	a	100	a
18	SPC 5	760	98.0	ab	98.0	a	100	a	100	a	100	a
19	Nominee fb Clincher	25 fb 100	96.5	b	95.3	a	97.5^{A}	a	97.5^{B}	a	98.3^{C}	a
20	Pyzero fb 2,4-D	100 fb 400	12.5	c	20.0	b	20.0	b	30.0^{D}	b	50.0^{E}	b
21	Hand-weeded	-	100	a	100	a	100	a	100	a	100	a
22	Control/Unweeded	-	0	d	0	c	0	c	0	c	0	c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: days after application; DAS: days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 34. Control of *Fimbristylis miliacea* at Different Assessment Timings

Treatment			Control of *Fimbristylis miliacea* (%)									
No	Product	Rate (g ai/ha)	8 DAA		15 DAA		22 DAA		29 DAA		43 DAA	
1	SPC 1	190	97.3	ab	98.0	a	99.5	a	99.5	a	99.5	a
2	SPC 1	260	97.3	ab	98.0	a	99.5	a	100	a	100	a
3	SPC 1	320	97.3	ab	98.0	a	100	a	100	a	100	a
4	SPC 1	380	98.0	ab	98.0	a	100	a	100	a	100	a
5	SPC 1	640	98.0	ab	98.0	a	100	a	100	a	100	a
6	SPC 1	760	98.0	ab	98.0	a	100	a	100	a	100	a

7	SPC 4	190	97.3 ab	98.0 a	100 a	100 a	100 a
8	SPC 4	260	98.0 ab	98.0 a	100 a	100 a	100 a
9	SPC 4	320	98.0 ab	98.0 a	100 a	100 a	100 a
10	SPC 4	380	98.0 ab	98.0 a	100 a	100 a	100 a
11	SPC 4	640	98.0 ab	98.0 a	100 a	100 a	100 a
12	SPC 4	760	98.0 ab	98.0 a	100 a	100 a	100 a
13	SPC 5	190	98.0 ab	98.0 a	100 a	100 a	100 a
14	SPC 5	260	98.0 ab	98.0 a	100 a	100 a	100 a
15	SPC 5	320	98.0 ab	98.0 a	100 a	100 a	100 a
16	SPC 5	380	98.0 ab	98.0 a	100 a	100 a	100 a
17	SPC 5	640	98.0 ab	98.0 a	100 a	100 a	100 a
18	SPC 5	760	98.0 ab	98.0 a	100 a	100 a	100 a
19	Nominee fb Clincher	25 fb 100	96.5 b	95.3 a	97.5^{A} a	97.5^{B} a	98.3^{C} a
20	Pyzero fb 2,4-D	100 fb 400	12.5 c	20.0 b	20.0 b	30.0^{D} b	52.5^{E} b
21	Hand-weeded	-	100 a	100 a	100 a	100 a	100 a
22	Control/Unweeded	-	0 d	0 c	0 c	0 c	0 c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: days after application; DAS: days after seeding;
A: 9 days after application of ‘Clincher’; B: 16 days after application of ‘Clincher’; C: 30 days after application of ‘Clincher’;
D: 5 days after application of ‘2,4-D’; E: 19 days after application of ‘2,4-D’;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 35. Control of *Cyperus iria* at Different Assessment Timings

Treatment			Control of *Cyperus iria* (%)				
No	Product	Rate (g ai/ha)	8 DAA	15 DAA	22 DAA	29 DAA	43 DAA
1	SPC 1	190	97.3 ab	98.0 a	99.5 a	99.5 a	99.5 a
2	SPC 1	260	97.3 ab	98.0 a	99.5 a	100 a	100 a
3	SPC 1	320	97.3 ab	98.0 a	100 a	100 a	100 a
4	SPC 1	380	98.0 ab	98.0 a	100 a	100 a	100 a
5	SPC 1	640	98.0 ab	98.0 a	100 a	100 a	100 a
6	SPC 1	760	98.0 ab	98.0 a	100 a	100 a	100 a
7	SPC 4	190	97.3 ab	98.0 a	100 a	100 a	100 a
8	SPC 4	260	98.0 ab	98.0 a	100 a	100 a	100 a
9	SPC 4	320	98.0 ab	98.0 a	100 a	100 a	100 a
10	SPC 4	380	98.0 ab	98.0 a	100 a	100 a	100 a
11	SPC 4	640	98.0 ab	98.0 a	100 a	100 a	100 a
12	SPC 4	760	98.0 ab	98.0 a	100 a	100 a	100 a
13	SPC 5	190	98.0 ab	98.0 a	100 a	100 a	100 a
14	SPC 5	260	98.0 ab	98.0 a	100 a	100 a	100 a
15	SPC 5	320	98.0 ab	98.0 a	100 a	100 a	100 a
16	SPC 5	380	98.0 ab	98.0 a	100 a	100 a	100 a
17	SPC 5	640	98.0 ab	98.0 a	100 a	100 a	100 a
18	SPC 5	760	98.0 ab	98.0 a	100 a	100 a	100 a
19	Nominee fb Clincher	25 fb 100	96.5 b	95.3 a	97.5^{A} a	97.5^{B} a	98.3^{C} a
20	Pyzero fb 2,4-D	100 fb 400	12.5 c	20.0 b	20.0 b	30.0^{D} b	52.5^{E} b
21	Hand-weeded	-	100 a	100 a	100 a	100 a	100 a
22	Control/Unweeded	-	0 d	0 c	0 c	0 c	0 c

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4

DAA: Days after application; DAS: Days after seeding;
A: 9 days after application of 'Clincher'; B: 16 days after application of 'Clincher'; C: 30 days after application of 'Clincher';
D: 5 days after application of '2,4-D'; E: 19 days after application of '2,4-D';
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 36. Population Count of Grasses at 43 DAA

Treatment			Population Count of Grasses					
No	Product	Rate (g ai/ha)	*E. crus-galli*		*L. chinensis*		*I. rugosum*	
1	SPC 1	190	2.3	b	11.8	a	4.5	b
2	SPC 1	260	1.3	b	13.0	a	2.8	b
3	SPC 1	320	1.8	b	8.5	a	1.5	b
4	SPC 1	380	4.5	b	18.3	a	2.3	b
5	SPC 1	640	0.3	b	12.0	a	0.5	b
6	SPC 1	760	1.3	b	6.5	a	1.3	b
7	SPC 4	190	0.5	b	12.0	a	1.8	b
8	SPC 4	260	3.3	b	6.3	a	3.0	b
9	SPC 4	320	0.0	b	9.3	a	1.5	b
10	SPC 4	380	0.8	b	11.5	a	2.3	b
11	SPC 4	640	0.0	b	11.5	a	1.3	b
12	SPC 4	760	0.0	b	5.8	a	1.0	b
13	SPC 5	190	3.8	b	7.0	a	2.5	b
14	SPC 5	260	0.0	b	7.8	a	1.5	b
15	SPC 5	320	2.3	b	10.8	a	2.0	b
16	SPC 5	380	1.0	b	9.5	a	1.0	b
17	SPC 5	640	0.5	b	3.8	a	0.8	b
18	SPC 5	760	3.8	b	2.3	a	0.0	b
19	Nominee fb Clincher	25 fb 100	0.8	b	1.5	a	0.3	b
20	Pyzero fb 2,4-D	100 fb 400	0.5	b	3.3	a	0.0	b
21	Hand-weeded	-	2.5	b	7.75	a	0	b
22	Control/Unweeded	-	28.25	a	28	a	10.25	a

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 36 (cont'd). Population Count of Broadleaves at 43 DAA

Treatment			Population Count of Broadleaves				
No	Product	Rate (g ai/ha)	*M. vaginalis*	*S. zeylanica*		*L. octovalvis*	
1	SPC 1	190	-	0	a	0.8	a
2	SPC 1	260	-	0	a	0.5	a
3	SPC 1	320	-	0	a	0	a
4	SPC 1	380	-	0	a	0	a
5	SPC 1	640	-	0	a	0	a
6	SPC 1	760	-	0	a	0	a
7	SPC 4	190	-	0	a	0	a
8	SPC 4	260	-	0	a	0	a
9	SPC 4	320	-	0	a	0	a

10	SPC 4	380	-	0 a	0 a
11	SPC 4	640	-	0 a	0 a
12	SPC 4	760	-	0 a	0 a
13	SPC 5	190	-	0 a	0 a
14	SPC 5	260	-	0 a	0 a
15	SPC 5	320	-	0 a	0 a
16	SPC 5	380	-	0 a	0 a
17	SPC 5	640	-	0 a	0 a
18	SPC 5	760	-	0 a	0 a
19	Nominee fb Clincher	25 fb 100	-	0 a	0 a
20	Pyzero fb 2,4-D	100 fb 400	-	0 a	0 a
21	Hand-weeded	-	-	0 a	0 a
22	Control/Unweeded	-	-	4.3 a	4.8 a

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 36 (cont'd). Population Count of Sedges at 43 DAA

Treatment			Population Count of Sedges		
No	Product	Rate (g ai/ha)	*C. difformis*	*F. miliacea*	*C. iria*
1	SPC 1	190	0 a	0 a	0 a
2	SPC 1	260	0 a	0 a	0 a
3	SPC 1	320	0 a	0 a	0 a
4	SPC 1	380	0 a	0 a	0 a
5	SPC 1	640	0 a	0 a	0 a
6	SPC 1	760	0 a	0 a	0 a
1	SPC 4	190	0 a	0 a	0 a
2	SPC 4	260	0 a	0 a	0 a
3	SPC 4	320	0 a	0 a	0 a
4	SPC 4	380	0 a	0 a	0 a
5	SPC 4	640	0 a	0 a	0 a
6	SPC 4	760	0 a	0 a	0 a
1	SPC 5	190	0 a	0 a	0 a
2	SPC 5	260	0 a	0 a	0 a
3	SPC 5	320	0 a	0 a	0 a
4	SPC 5	380	0 a	0 a	0 a
5	SPC 5	640	0 a	0 a	0 a
6	SPC 5	760	2.0 a	0 a	0 a
7	Nominee fb Clincher	25 fb 100	7.5 a	0 a	0 a
8	Pyzero fb 2,4-D	100 fb 400	18.0 a	3.0 a	0 a
9	Hand-weeded	-	0 a	0.8 a	0 a
10	Control/Unweeded	-	47.5 a	3.0 a	0.8 a

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone; F1 in Table 4
SPC 4: 0.3% Sulfentrazone + 0.6% Penoxsulam + 1.2% Clomazone; F2 in Table 4
SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone; F3 in Table 4
DAA: Days after application;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Example 15: Against *Leptochloa chinensis* – Confirmation of Synergism Resulting from the Combination of Sulfentrazone, Penoxsulam, and Clomazone

In this trial, the active ingredients in the granule sample (SPC 5) were sulfentrazone, penoxsulam, and clomazone. The total content of the active ingredients in the sample was 2.8%. The formula is listed in Table 4 as F3.

SynTech conducted this trial at its research station in Cabanatuan City, Philippines. The crop was directed-seeded Indica rice and the application timings were 10 and 15 DAS.

To ensure *Leptochloa chinensis* with a meaningful infestation, its seeds were introduced to the field at 2 kg/ha in addition to other natural occurrence of weeds. In priming the weed seeds, only soaking of the seeds for 24 hours was done. Primed weed seeds were thoroughly mixed with fine and dry soil for ease of broadcasting by hand. Indica rice (NSIC Rc216) was seeded at 40 kg/ha. Pre-germinated rice and the weed seeds were hand-broadcast into trial plots. No standing water at seeding and soil condition was at saturation point.

Rice and weed germination and growth were normal. By application day (10 and 15DAS) *Leptochloa chinensis*' pressure was extremely high.

The average leaf stage of *Leptochloa chinensis* was as follows: 2.5 – 3.5 leaf stage at 10 DAS; 3.5 – 4.5 leaf stage at 15 DAS.

The trial followed a randomized complete block design (RCBD). Each block consisted of seven plots wherein the treatments were randomly assigned. Each plot size was 5 M^2 (2.5 m x 2.0 m) and plots were separated by earthen levees. Each treatment had four replications.

Test granules were hand-broadcast into the plots at 10 DAS and 15 DAS. The commercial standard 'Nominee 100SC' (Bispyribac-sodium) was applied by foliar spray at 15 DAS according to its labels. Spray volume was 200 L/ha.

Water depth was about 0.5 cm at the time of application and was maintained for three days after granular application. The water level was increased to 1-3 cm at 4 to 10 DAA, and was maintained at 3-5 cm thereafter. In plots sprayed with standard herbicides, about 3 cm water was introduced to the plots at 3 DAA and maintained at 3-5 cm in accordance with the timing of water introduction to plots treated with test granules.

Preventive measures similar to those in Example 14 were taken during the trial.

Table 37. Use Rates of SPC 5 Granule and Standards and Their Application Timings

Treatment				Requirements g/plot (5 M^2)
No	Product	Rate (g ai/ha)	Application Timings	
1	SPC 5	320	10 DAS	5.46
2	SPC 5	320	15 DAS	5.46
3	SPC 5	380	15 DAS	6.48
5	SPC 5	640	15 DAS	10.92

7	Nominee 100 SC	25	15 DAS	
10	Control/Unweeded	-	-	

SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone (F3 in Table 4);
DAS: Days after seeding;

SynTech reported that no phytotoxicity in rice was observed at 3, 7, and 14 DAA (Table 38). If no phytotoxicity is observed by 7 DAA, we know from past experience that the treatment will not cause rice injury later on. SynTech also reported that plant height and tiller number were not affected by the test granules regardless of their application timings and rates, thereby confirming selectivity to rice seedlings.

For both sources of *Leptochloa chinensis*, our SPC 5 granules demonstrated fast and excellent control efficacy (91-96%) by 7 DAA and 97-98% by 21 DAA (Tables 39-40).

Table 38. Effect of Three Granules and Standards on Rice Seedlings at Different Assessment Timings

Treatment			Injury to Rice Seedlings (%)			
No	Product	Rate (g ai/ha)	3 DAA	7 DAA	14DAA	21 DAA
1	SPC 5	320	0 a	0 a	0 a	
2	SPC 5	320	0 a	0 a	0 a	
3	SPC 5	380	0 a	0 a	0 a	
4	SPC 5	640	0 a	0 a	0 a	
5	Nominee 100SC	25	0 a	0 a	0 a	
6	Control/Unweeded	-	0 a	0 a	0 a	

SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone (F3 in Table 4);
DAA: Days after application;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 39. Control of *Leptochloa chinensis* (collected from the Soledad area)

Treatment			Application Timing (DAS)	Control of *Leptochloa chinensis* (%)		
No	Product	Rate (g ai/ha)		7 DAA	14 DAA	21 DAA
1	SPC 5	320	10	91.3 a	92.8 a	96.5 a
2	SPC 5	320	15	95.8 a	95.3 a	98.0 a
3	SPC 5	380	15	93.3 a	93.5 a	97.3 a
4	SPC 5	640	15	95.0 a	96.5 a	97.3 a
7	Nominee 100 SC	25	15	6.3 b	5.0 b	16.3 b
10	Control/Unweeded	-	-	0 b	0 b	0 c

SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone (F3 in Table 4);
DAA: Days after application; DAS: Days after seeding;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Table 40. Control of *Leptochloa chinensis* (collected from the Bakod Bayan area)

Treatment	Application Timing	Control of *Leptochloa chinensis* (%)

No	Product	Rate (g ai/ha)	(DAS)	7 DAA		14 DAA		21 DAA	
1	SPC 5	320	10	91.3	a	92.8	a	97.3	a
2	SPC 5	320	15	94.5	a	96.5	a	98.0	a
3	SPC 5	380	15	95.8	a	96.5	a	98.0	a
4	SPC 5	640	15	95.0	a	97.3	a	98.0	a
7	Nominee 100 SC	25	15	5.0	b	5.0	b	17.5	b
10	Control/Unweeded	-	-	0	b	0	b	0	c

SPC 5: 0.4% Sulfentrazone + 0.8% Penoxsulam + 1.6% Clomazone (F3 in Table 4);
DAA: Days after application; DAS: Days after seeding;
In a column, means followed by same letter do not significantly differ (P=.05, Student-Newman-Keuls)
Mean comparisons performed only when AOV Treatment P (F) is significant at mean comparison OSL

Example 16: Performance Comparison between Granule Sample (1.4% AI) and Standard in Transplanted Rice

Table 41. Use Rates of Granule Sample and Standard and Their Application Timings

Treatment				Requirements g/plot (54 M^2)
No	Product	Rate (g ai/ha)	Application Timings	
1	SPC 1	315	8 DAT	121.5
2	SPC 1	730	8 DAT	281.6
3	Bensulfuron-methyl 10% WP	45	8 DAT	2.4
4	Control/Unweeded	-	-	

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone (F1 in Table 4)

The purpose of this trial was to compare our test granule with the rice herbicide product most popular locally, bensulfuron-methyl. The active ingredients in the test granule were sulfentrazone, penoxsulam, and clomazone. The total content of the active ingredients in the granule was 1.4%. The formula is listed in Table 4 as F1.

This field trial was carried out in Lu Lai village, Bo Lian Zhen, Lin Gao County, Hainan Province, China, sponsored by SSPC and SAAS. The crop was hand-transplanted Indica rice with 30-day old rice seedlings.

The testing field was prepared according to local farmers' practice. Water was first introduced into the field for three days, and the field was plowed and leveled. After leveling, the water level was raised to and maintained at 3-5 cm.

The trial followed a randomized complete block design (RCBD). Each block, consisting of four plots wherein the treatments were randomly assigned, was separated by a small canal used for irrigation and drainage. Each plot size was 54 M^2 (3 m x 18 m) and plots were in parallel and separated by earthen levees. Each treatment received three replications save for the twice-repeated blank control due to limited space.

Weeds were naturally-occurring and heavy. The major weeds in the field prior to and during the trial were: *Echinochloa crus-galli, Digitaria setigera, Commelina communis, Mint, Bermudagrass, Cyperus difformis, Cyperus iria, Mentha haplocalyx Briq., peppermint,* and *Lindernia procumbens*.

The test granules and the commercial standard (bensulfuron-methyl) were mixed separately with soil according to local practice and then hand-broadcast into separate plots at 8 DAT.

After the applications, the water level was raised to and maintained at 10 cm for about a week. After this week, there were intermittent periods lasting several days during which plots did not have water, for (1) local custom dictates letting rice roots grow deep; (2) the water level in the irrigation system was low. Other than in those periods, water was always maintained.

Fertilizer, insecticides, and fungicides were applied at the local farmers' discretion.

There were only two use rates, 315 g ai/ha and 730 g ai/ha, for the granule sample. The lower rate was used for performance comparison with bensulfuron-methyl and 730 g ai/ha was used to assess the granule safety to rice. Detailed information about the test sample, comparison standard, control, use rates, plot size, and application timing is summarized in Table 41.

In the plots treated with the test granules, weeds started showing symptoms of being poisoned by 3 DAA and very sporadic, slight rice phytotoxicity (<0.1%) was also observed. There were only two rice seedlings in all granule-treated plots that showed "whitening" signs.

By 20 DAA, the contrast in performances was salient. *Echinochloa crus-galli* had grown as tall as the rice in both the control plots and the plots treated with bensulfuron-methyl, while no *Echinochloa crus-galli* was observed at all in plots treated with the test granules.

By 43 DAA, there was a total of 40 *Echinochloa crus-galli* found in two control plots (108 M^2), 39 in all three plots (162 M^2) treated with bensulfuron-methyl, and only 4 in all six plots (324 M^2) treated with the granules, respectively. In addition, there were nearly no other weeds found in the six plots treated with the granules while there were many other types of weeds in the control and bensulfuron-methyl treated plots.

Table 42. Fresh Weight of Weeds

	SPC 1 (281.6 g/54 M^2)	SPC 1 (121.5 g/54 M^2)	Bensulfuron-methyl	Control
Replication 1	40	0	120	200
Replication 2	0	0	160	200
Replication 3	0	0	140	
Average	13.33	0	140	200

SPC 1: 0.2% Sulfentrazone + 0.4% Penoxsulam + 0.8% Clomazone (F1 in Table 4);
Weeds: *Echinochloa crus-galli, Digitaria setigera, Commelina communis, Mint, Bermudagrass, Cyperus difformis, Cyperus iria, Mentha haplocalyx Briq., and Lindernia procumbens.*

Table 43. Rice Grain Yield (kg/M^2)

	SPC 1 (281.6 g/54 M^2)	SPC 1 (121.5 g/54 M^2)	Bensulfuron-methyl	Control
Replication 1	1.54	1.52	1.54	1.40
Replication 2	1.64	1.44	1.40	1.37
Replication 3	1.54	1.62	1.51	

Average	1.57	1.53	1.48	1.39

Rice grains were collected near plot edges for all treatments and control, and left to dry naturally. The grains were not fully dry at weighing.

Table 44. Thousand-Grain Weight (g)

	SPC 1 (281.6 g/54 M^2)	SPC 1 (121.5 g/54 M^2)	Bensulfuron-methyl	Control
Replication 1	31.94	31.51	31.42	29.67
Replication 2	32.68	32.12	30.87	31.47
Replication 3	33.00	32.91	32.67	
Average	32.54	32.18	31.72	30.57

The rice yield collected from plots treated with the granules (730 g ai/ha) was 6.08% and 12.95% higher than that collected from the plots treated with bensulfuron-methyl and the control, respectively; the thousand-grain weight was 2.59% and 6.44% higher than that from the plots treated with bensulfuron-methyl and control, respectively.

The rice yield collected from plots treated with the granules (315 g ai/ha) was 3.38% and 10.07% higher than that collected from the plots treated with bensulfuron-methyl and the control; the thousand-grain weight was 1.45% and 5.27% higher, respectively.

Example 17: Efficacy of Controlled-Release Sulfentrazone and Other Formulations on Major Weeds in Temperate Rice Fields

This trial was conducted in South Korea, a temperate rice region, where some major rice weeds are different from those prevalent in hot-climate rice regions. In temperate rice regions, broadleaves and sedges and their ALS-resistant counterparts prove to be a main challenge. We have introduced sulfentrazone to our rice product for its high efficacy towards broadleaves and sedges and with no resistance to date in order to address these regional discrepancies.

The crop was transplanted Japonica rice with rice seedlings of 15-20 days old. Each plot size was 10 M^2. There were three replications for each treatment. The application timing was 13 DAT.

The following were the major weeds found in the field: *Echinochloa oryzicola* (sulfonylurea-resistant weed or SU-resistant weed), *Scirpus juncoides* (SU-resistant weed), *Monochoria vaginalis* (SU-resistant weed), *Eleocharis kuroguwai*, *Cyperus difformis*, *Lindernia dubia*, and *Ludwigia prostrata*.

From the trial (Tables 45-52), it is clear that sulfentrazone was efficacious against the abovementioned broadleaves and sedges, including even SU-resistant *Scirpus juncoides* and *Monochoria vaginalis*.

Echinochloa oryzicola can be controlled with our proposed product candidates in the invention. Even SU-resistant *Echinochloa oryzicola* can be controlled by one of our proposed candidates (0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron) and applying at 6-8 DAT (Table 52).

Table 45. Control of *Monochoria vaginalis* at Different Assessment Timings

Treatment				Control of *Monochoria vaginalis** (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after transplanting)	10 DAA	20 DAA	30 DAA
1	S	120	13	83	80	87
2	S	180	13	85	85	87
3	S	240	13	93	90	97
4	S	360	13	97	100	97
5	S	480	13	97	98	98
6	SF	120 + 21	13	20	53	87
7	SP	120 + 24	13	40	42	87
8	SC	240 + 120	13	100	100	97
9	BCF	210 + 75 + 21	13	100	100	100
10	Untreated	0		0	0	0

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4);
*: *Monochoria vaginalis* was sulfonylurea-resistant.

Table 46. Control of *Scirpus Juncoides* at Different Assessment Timings

Treatment				Control of *Scirpus Juncoides** (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after transplanting)	10 DAA	20 DAA	30 DAA
1	S	120	13	67	73	73
2	S	180	13	80	80	80
3	S	240	13	90	87	87
4	S	360	13	100	100	100
5	S	480	13	100	100	100
6	SF	120 + 21	13	38	47	83
7	SP	120 + 24	13	45	33	83
8	SC	240 + 120	13	87	87	100
9	BCF	210 + 75 + 21	13	100	100	100
10	Untreated	0		0	0	0

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4);
*: *Scirpus Juncoides* was sulfonylurea-resistant.

Table 47. Control of *Lindermia procumbens* at Different Assessment Timings

Treatment				Control of *Lindermia procumbens* (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after transplanting)	10 DAA	20 DAA	30 DAA
1	S	120	13	-	72	80
2	S	180	13	-	83	83
3	S	240	13	-	97	100
4	S	360	13	-	100	100
5	S	480	13	-	100	100
6	SF	120 + 21	13	-	97	93
7	SP	120 + 24	13	-	92	97
8	SC	240 + 120	13	-	100	97
9	BCF	210 + 75 + 21	13	-	100	100
10	Untreated	0		-	0	0

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4).

Table 48. Control of *Lindermia dubia* at Different Assessment Timings

Treatment				Control of *Lindermia dubia* (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after transplanting)	10 DAA	20 DAA	30 DAA
1	S	120	13	-	80	85
2	S	180	13	-	87	88
3	S	240	13	-	87	93
4	S	360	13	-	97	97
5	S	480	13	-	100	100
6	SF	120 + 21	13	-	85	85
7	SP	120 + 24	13	-	83	80
8	SC	240 + 120	13	-	100	100
9	BCF	210 + 75 + 21	13	-	100	100
10	Untreated	0		-	0	0

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4).

Table 49. Control of *Ludwigia prostrata* at Different Assessment Timings

Treatment				Control of *Ludwigia prostrata* (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after	10 DAA	20 DAA	30 DAA

			transplanting)			
1	S	120	13	-	-	67
2	S	180	13	-	-	87
3	S	240	13	-	-	100
4	S	360	13	-	-	100
5	S	480	13	-	-	100
6	SF	120 + 21	13	-	-	100
7	SP	120 + 24	13	-	-	100
8	SC	240 + 120	13	-	-	100
9	BCF	210 + 75 + 21	13	-	-	100
10	Untreated	0		-	-	0

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4).

Table 50. Control of *Eleocharis kuroguwai* at Different Assessment Timings

Treatment				Control of *Eleocharis kuroguwai* (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after transplanting)	10 DAA	20 DAA	30 DAA
1	S	120	13	85	82	80
2	S	180	13	83	83	82
3	S	240	13	88	93	80
4	S	360	13	88	90	88
5	S	480	13	93	94	94
6	SF	120 + 21	13	53	57	53
7	SP	120 + 24	13	53	50	60
8	SC	240 + 120	13	77	77	73
9	BCF	210 + 75 + 21	13	68	63	70
10	Untreated	0		0	0	0

1-5: S means 0.4% Sulfentrazone; (F20in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4).

Table 51. Control of All Weeds at Different Assessment Timings

Treatment				Control of All Weeds (%)		
No	Product	Rate (g ai/ha)	Application Timing (Days after transplanting)	10 DAA	20 DAA	30 DAA
1	S	120	13			87
2	S	180	13	-	-	91
3	S	240	13	-	-	94
4	S	360	13	-	-	96
5	S	480	13	-	-	97

6	SF	120 + 21	13	-	-	90
7	SP	120 + 24	13	-	-	90
8	SC	240 + 120	13	-	-	93
9	BCF	210 + 75 + 21	13	-	-	93
10	Untreated	0		-	-	0

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4);.

Table 52. Control of *Echinochloa oryzicola* at Different Assessment Timings

Treatment				Control of *Echinochloa oryzicola** (%)		
No	Product	Rate (g ai/ha)	Application Timing (DAT)	10 DAA	20 DAA	30 DAA
1	S	120	6	0	0	-
2	S	180	6	0	0	-
3	S	240	6	0	0	-
4	S	360	6	0	0	-
5	S	480	6	0	0	-
6	SF	120 + 21	6	0	0	-
7	SP	120 + 24	6	0	0	-
8	SC	120 + 60	6	3	0	
9	SC	240 + 120	6	70	0	-
10	SC	480 + 240	6	87	73	
11	BCF	210 + 75 + 21	6	100	100	-
12	Untreated	0		0	0	-

1-5: S means 0.4% Sulfentrazone; (F20 in Table 4);
6: SF: 0.4% Sulfentrazone + 0.07% Flucetosulfuron (F27 in Table 4);
7: SP: 0.4% Sulfentrazone + 0.08% Penoxsulam (F31 in Table 4);
8: SC: 0.4% Sulfentrazone + 0.2% Clomazone (F32 in Table 4);
9: BCF: 0.7% Benzobicyclon + 0.25% Carfentrazone-ethyl + 0.07% Flucetosulfuron (F33 in Table 4);
*: This sulfonylurea-resistant weed could be controlled with the BCF granule at 6 DAT.

What has been described and illustrated herein are preferred embodiments of the invention along with some of its variations. The terms, descriptions, and data used herein are set forth by way of illustration only and are not meant as limitations. Those skilled in the art will recognize that many variations are possible within the spirit and scope of the invention, which is intended to be defined by the following claims (and their equivalents) in which all terms are meant in their broadest reasonable sense unless otherwise indicated. Any headings utilized within the description are for convenience only and have no legal or limiting effect.

Industrial Applicability

This invention can be used in the field of agriculture for weed control with industrial applicability.

What is claimed is

1. An herbicide composition comprising at least one herbicidal active ingredient selected from Group 1 and/or Group 2, and at least one polymer where the composition is formulated into a granule or sprayable form, wherein

Group 1 consists of bispyribac-sodium, penoxsulam, cyhalofop-butyl, metamifop, pyribenzoxim, azimsulfuron, flucetosulfuron, metazosulfuron, pyrimisulfan, profoxydim, pyriminobac-methyl, mefenacet, benzobicyclon, clomazone, fenoxaprop-P-ethyl, quinclorac, and propyrisulfuron;

Group 2 consists of members of the protoporphyrinogen oxidase inhibitor group (PPO inhibitor herbicides) and the branched-chain amino acid inhibitor group (ALS or AHAS inhibitor herbicides) other than those listed in Group 1;

the polymer is made of a cellulose type molecule selected from cellulose acetate, cellulose triacetate, cellulose propionate, cellulose acetate propionate, cellulose acetate butyrate, cellulose nitrate, cellulose sulfate, methylcellulose, ethylcellulose, ethyl methyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, hydroxyethyl methyl cellulose, hydroxypropyl methyl cellulose, ethyl hydroxyethyl cellulose, carboxymethyl cellulose, croscarmellose sodium, and hemicellulose, or mixtures thereof; and

one or more of the at least one herbicidal active ingredient is/are dispersed in the at least one polymer.

2. The herbicide composition according to Claim 1, wherein the composition comprises at least two herbicidal active ingredients selected from Group 1 or Group 2.

3. The herbicide composition according to Claim 1 or Claim 2, wherein the composition comprises at least one herbicidal active ingredient selected from Group 1 and at least one herbicidal active ingredient selected from Group 2.

4. The herbicide composition according to Claim 3, wherein the composition comprises at least three herbicidal active ingredients selected from Group 1 and Group 2.

5. The herbicide composition according to any one of the preceding claims, wherein the herbicidal active ingredient(s) dispersed in the at least one polymer is/are one or more herbicidal active ingredient(s) selected from the group consisting of: acifluorfen-sodium, bifenox, butafenacil, carfentrazone-ethyl, cinidon-ethyl, flufenpyr-ethyl, flumiclorac-pentyl, flumioxazin, fluoroglycofen-ethyl, fluthiacet-methyl, fomesafen, lactofen, oxadiargyl, oxadiazon, oxyfluorfen, pentoxazone, pyraclonil, pyraflufen-ethyl, sulfentrazone, saflufenacil, imazamethabenz-methyl, imazamox, imazapic, imazapyr, imazaquin, imazethapyr, profoxydim, clomazone, fenoxaprop-P-ethyl, and quinclorac.

6. The herbicide composition according to any one of the preceding claims, wherein the total weight of the herbicidal active ingredients based on the total weight of the composition of the granule form is up to 14%, or the total weight of the herbicidal active ingredients based on the total weight of the composition of the sprayable form is up to 25%.

7. The herbicide composition according to any one of the preceding claims, wherein the composition comprises sulfentrazone as the herbicidal active ingredient.

8. The herbicide composition according to any one of the preceding claims, wherein the composition comprises sulfentrazone, clomazone, and penoxsulam as the herbicidal active ingredients.

9. The herbicide composition according to Claims 7 or 8, wherein the composition is formulated into a granule or sprayable form by dispersing sulfentrazone in the at least one polymer.

10. The herbicide composition according to any one of the preceding claims, wherein the composition comprises the polymer in an amount of 0.01 – 18%, by weight, based on the total weight of the composition.

11. The herbicide composition according to any one of the preceding claims, wherein the composition comprises 0.02–14% by weight of two herbicidal active ingredients jointly selected from Groups 1 and/or Group 2 or 0.03–14% of three herbicidal active ingredients jointly selected from Groups 1 and/or Group 2, 0.01–18% by weight of the polymer, 0.001–1% by weight of C_{18-22} amine, 0.25–25% by weight of dispersant, 0.05–10% by weight of wetting agent, 0.125–12.5% by weight of binder, 0.05–15% by weight of carrier, and 40-95% by weight of inert carrier.

12. The herbicide composition according to any one of the preceding claims, wherein the composition comprises 0.01–4% by weight of sulfentrazone, 0.01–8% by weight of penoxsulam, 0.01–13.98% by weight of clomazone, 0.01–18% by weight of cellulose acetate butyrate, 0.001–1% by weight of octadecylamine, 0.25–25% by weight of Borresperse NA, 0.05–10% by weight of Supragil WP, 0.125–12.5% by weight of Polyvinylpyrrolidone K-30, 0.05–15% by weight of Sipernat 50S, and 40-95% by weight of kaolin.

13. The herbicide composition according to any one of the preceding claims, wherein the composition comprises clomazone, at least one herbicidal active ingredient selected from Group 1 other than clomazone, and at least one herbicidal active ingredient selected from Group 2.

14. The herbicide composition according to any one of the preceding claims, wherein the composition comprises clomazone, and at least one herbicidal active ingredient selected from Group 1 other than clomazone.

15. The herbicide composition according to any one of the preceding claims, wherein the polymer in the sprayable form has particle sizes in the range of 5-25 μm, or the polymer in the granule form has particle sizes in the range of 40-750 μm.

16. The herbicide composition according to any one of the preceding claims, wherein the herbicide composition is used for rice.

17. A rice herbicide composition comprising at least one herbicidal active ingredient selected from Group 1 and at least one herbicidal active ingredient selected from Group 2, wherein

Group 1 consists of bispyribac-sodium, penoxsulam, cyhalofop-butyl, metamifop, pyribenzoxim, azimsulfuron, flucetosulfuron, metazosulfuron, pyrimisulfan, profoxydim, pyriminobac-methyl, mefenacet, benzobicyclon, clomazone, fenoxaprop-P-ethyl, quinclorac, and propyrisulfuron; and

Group 2 consists of members of the protoporphyrinogen oxidase inhibitor group (PPO inhibitor herbicides) and the branched-chain amino acid inhibitor group (ALS or AHAS inhibitor herbicides) other than those listed in Group 1.

18. The rice herbicide composition according to Claim 17, wherein the composition comprises at least three herbicidal active ingredients selected from Group 1 and Group 2.

19. The rice herbicide composition according to Claim 17, wherein the composition comprises sulfentrazone, penoxsulam, and clomazone.

20. A weed control method in rice fields, comprising applying the herbicide composition according to any one of Claims 1 through 19 in the rice fields.

21. The weed control method according to Claim 20, wherein the composition is applied in the fields with an use rate of 1-75 kg/ha.

22. The weed control method according to any one of Claims 20 through 21, wherein the composition is applied in the fields to provide a total weight of the herbicidal active ingredients ranging from 5-1280 g ai/ha.

23. The weed control method according to any one of Claims 20 through 22, wherein the composition is applied in the fields for Indica rice or Japonica rice.

24. The weed control method according to any one of Claims 20 through 23, wherein the composition is applied in the fields at application timings for direct-seeded rice and transplanted rice are 7-15 DAS and 5-15 DAT, respectively.

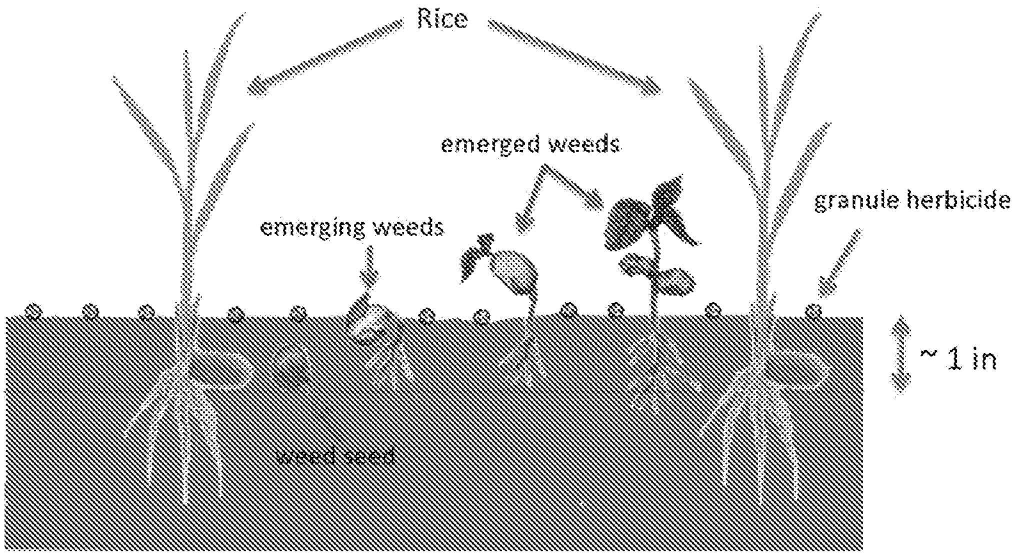

Figure 1

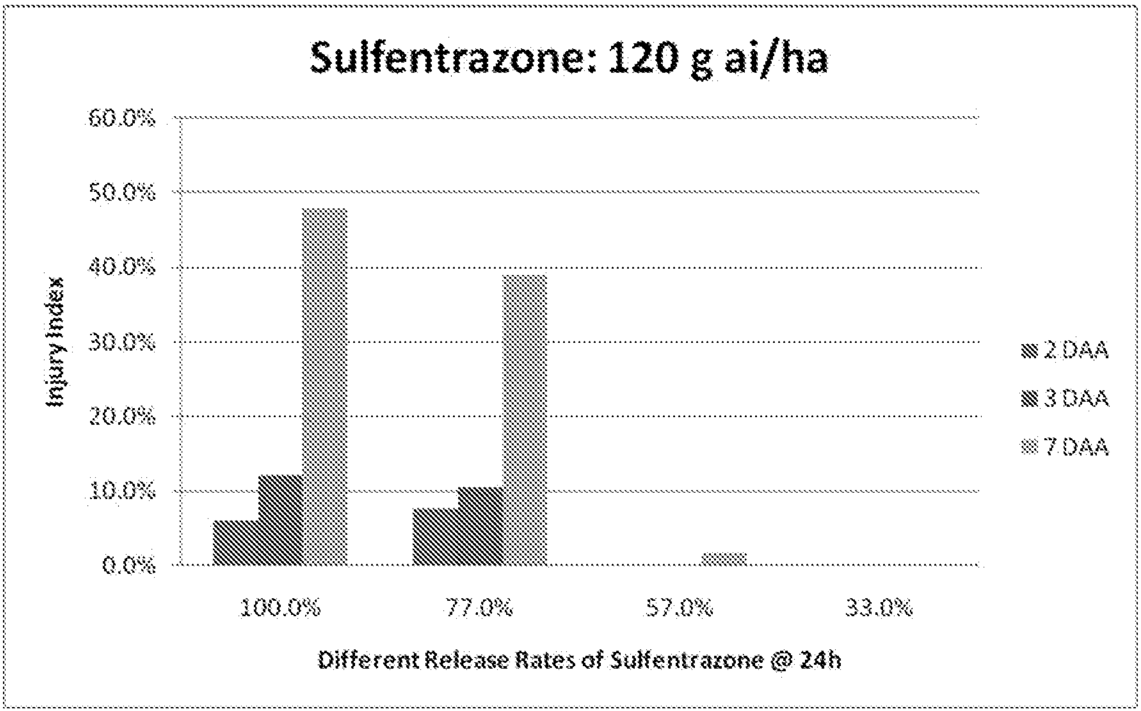

Figure 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2015/071348

A. CLASSIFICATION OF SUBJECT MATTER

A01N 43/42(2006.01)i； A01N 47/36(2006.01)i； A01N 43/54(2006.01)i； A01N 39/04(2006.01)i； A01N 43/76(2006.01)i； A01N 43/40(2006.01)i； A01N 43/78(2006.01)i； A01P 13/00(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A01N 43; A01N 47; A01N 39; A01P 13

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

SIPOABS, DWPI, CNABS, CPRSABS, CNKI, REGISTRY, CAPLUS(STN) herbicide, clomazone, sufentrazone, penoxsulam, bispyribac-sodium, penoxsulam, cyhalofop-butyl, metamifop, pyribenzoxim, azimsulfuron, flucetosulfuron, metazosulfuron, pyrimisufan, profoxydim, pyriminobac-methyl,mefenacet, benzobicyclon, fenoxaprop-P-ethyl, quinclorac, propyrisulfuron, ALS, AHAS, rice

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 101390516 A (REN ZHENGJUN) 25 March 2009 (2009-03-25) see examples 1-8	1-16，20-24
X	CN 102461543 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 9 and 10	17-24
X	CN 103039478 A (NANJING HUAZHOU PHARM CO LTD) 17 April 2013 (2013-04-17) see claims 1， 8 and 9	17-24
X	CN 102461505 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 9 and 10	17-24
X	CN 102461534 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 7 and 8	17-24
X	CN 104285957 A (HENAN ZHONGTIAN HENGXIN BIOCHEMICAL TECH) 21 January 2015 (2015-01-21) see claims 1 and 10	17-24

☑ Further documents are listed in the continuation of Box C. ☑ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
15 October 2015	**03 November 2015**

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 104115858 A (JIANGSU INST ECOMONES CO LTD) 29 October 2014 (2014-10-29) see claims 1 and 9	17-24
X	CN 102461538 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 7 and 8	17-24
X	CN 102461539 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 7 and 8	17-24
X	CN 102461540 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 7 and 8	17-24
X	CN 102461542 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 7 and 8	17-24
X	CN 102461544 A (NANJING HUAZHOU PHARM CO LTD) 23 May 2012 (2012-05-23) see claims 1， 7 and 8	17-24
A	WO 2008152074 A1 (BASF SE ET AL.) 18 December 2008 (2008-12-18) see the whole document	1-24

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2015/071348

Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
CN	101390516	A	25 March 2009	CN	101390516	B	21 September 2011
CN	102461543	A	23 May 2012		None		
CN	103039478	A	17 April 2013		None		
CN	102461505	A	23 May 2012		None		
CN	102461534	A	23 May 2012	CN	102461534	B	25 December 2013
CN	104285957	A	21 January 2015		None		
CN	104115858	A	29 October 2014		None		
CN	102461538	A	23 May 2012	CN	102461538	B	27 November 2013
CN	102461539	A	23 May 2012	CN	102461539	B	27 November 2013
CN	102461540	A	23 May 2012	CN	102461540	B	27 November 2013
CN	102461542	A	23 May 2012	CN	102461542	B	18 September 2013
CN	102461544	A	23 May 2012	CN	102461544	B	07 August 2013
WO	2008152074	A1	18 December 2008	UY	31149	A1	05 January 2009
				AU	2008263903	A2	22 July 2010
				AU	2008263903	A1	18 December 2008
				US	2010190794	A1	29 July 2010
				EA	200901658	A1	30 June 2010
				CA	2690072	A1	18 December 2008
				IL	202050	D0	16 June 2010
				TW	200911120	A	16 March 2009
				KR	20100018066	A	16 February 2010
				AR	066991	A1	23 September 2009
				PE	03662009	A1	26 April 2009
				CN	101677540	A	24 March 2010
				EP	2157856	A1	03 March 2010
				BR	PI0812954	A2	07 October 2014
				JP	2010529170	A	26 August 2010