



## Proposal for field-based definition of soil bound pesticide residues

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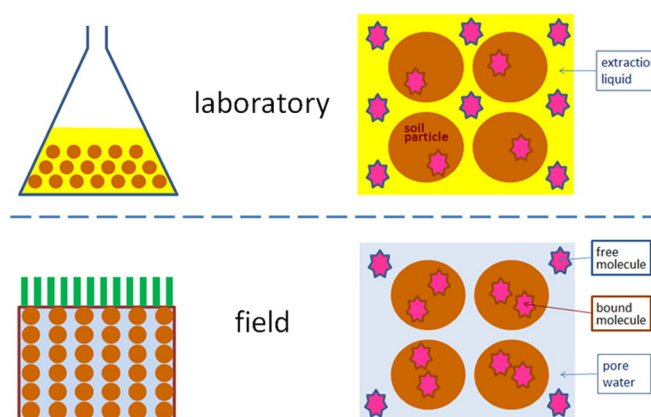
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### HIGHLIGHTS

- A definition of soil bound pesticide residues based on field soil is proposed.
- The definition refers to a specific molecule which may be a soil metabolite.
- Soil bound residue formed from a non-relevant soil metabolite, is also non-relevant.
- Self-exchange extraction procedures are likely to become more important.
- Risk managers need to define which land use changes are relevant for bound residues.

### GRAPHICAL ABSTRACT



### ARTICLE INFO

#### Article history:

Received 14 October 2015

Received in revised form 23 November 2015

Accepted 23 November 2015

Available online xxxx

Editor: D. Barcelo

#### Keywords:

Pesticides

Bound residues

Soil

Conceptual definition

Operational definition

### ABSTRACT

The environmental significance of soil bound pesticide residues (SBPR) is potentially large because approximately one third of the applied mass of the pesticides in agriculture ends up as SBPR. At EU level, there is little regulatory guidance available on the environmental risk assessment of SBPR in spite of some 50 years of SBPR research. This lack of guidance is partially caused by the fact that the current definitions of SBPR are founded on non-extractability in soil in the laboratory whereas for the environmental risk assessment not the soil in the laboratory but the soil in the field is the system of interest. Therefore a definition of SBPR is proposed that is based on the field soil: a molecule (further called 'the mother molecule') is soil bound if a relevant part of this molecule has become part of the solid phase in the soil and if this relevant part will never be released again to the liquid phase in soil under relevant field conditions in the form of this mother molecule or in the form of another molecule that may possibly raise environmental or human toxicological concerns. This mother molecule may be the parent substance that is applied to the soil but it may also be a metabolite of this parent substance. A consequence of the definition is that the SBPR terminology becomes more precise because the mother molecule of the soil bound residue has to be specified. A further consequence is that very strong but reversible sorption of molecules such as paraquat is not considered soil-bound residue anymore (as may be demonstrated by a self-exchange extraction procedure). Furthermore, the definition requires that risk managers have to define what they consider as 'relevant field conditions' (e.g. include also changes of agricultural fields into forests?).

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### 1. Introduction

The environmental significance of soil bound pesticide residues (abbreviated SBPR) is already for decades an issue. Are they a time-bomb

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ticking away which will ‘explode’ once a certain loading is reached or when soil conditions change due to changes in land use (or perhaps even due to climate change)? Or, since 60 years of widespread pesticide use has not apparently caused problems, can we merely assume that incorporation of pesticides into soil organic matter renders them harmless for all time (Bromilow, 1999)?

The environmental significance is potentially large because on average about one third of the total pesticide mass applied to soil ends up as soil bound residue (Barriuso et al., 2008, their Fig. 2), so in the order of 1 kg/ha is yearly added to the SBPR pool of a field under normal agriculture.

In the past 50 years, hundreds of soil bound residue (abbreviated SBR) studies have been conducted with pesticides as follows from a handful of reviews (Khan, 1982; IUPAC, 1984; Calderbank, 1989; Gevao et al., 2000; Barriuso et al., 2008; Kästner et al., 2014). Given these considerable activities over such a long period, amazing little guidance is available for the environmental risk assessment of SBPR in everyday regulatory life as can be illustrated by the pesticide registration at EU level. The European Commission and the European Food Safety Authority produced in the past 20 years guidance opinions and guidance documents on the environmental risk assessment of pesticides that sum up to something like 10 000 pages. However, they produced only *one* page of guidance on SBPR plus a decision-making scheme (European Commission, 2000), essentially saying that if >70% SBR are formed in 100 days at 20 °C in a laboratory study, a risk assessment is needed that shows no unacceptable (i) residues in succeeding crops, (ii) effects on succeeding plants, and (iii) impact on the environment; the scheme refers to a plateau concentration of SBPR, but this is not defined but it should be ‘determined by soil accumulation studies or modelling or another appropriate assessment’. This instruction raises more questions than it answers (e.g. how to determine a plateau SBR concentration from an accumulation study that is commonly carried out with non-radioactive substance?). Nevertheless, this guidance works already more or less satisfactorily over 15 years (see Craven and Hoy, 2005, for background). Thus there is quite a large gap between the knowledge that has been gained in the SBPR research and its use in risk assessment. It seems timely that an attempt is made to bridge this gap. One of the points that may help is a definition of SBPR that is more targeted to the risk assessment.

Since the early days of bound-residue research, the definition of bound residues has been a point of debate (US EPA, 1975). IUPAC (1984) provided the first definition that became widely accepted (Gevao et al., 2000; Kästner et al., 2014): “Non-extractable residues (sometimes referred to as “bound” or “non-extracted” residues) in plants and soils are defined as chemical species originating from pesticides, used according to good agricultural practice, that are unextracted by methods which do not significantly change the chemical nature of these residues. These non-extractable residues are considered to exclude fragments recycled through metabolic pathways leading to natural products.” Nearly 15 years later, Führ et al. (1998) proposed another definition based on ‘intensive expert discussion and several circulars’ which seems to have got wide acceptance meanwhile (Gevao et al., 2000; Barriuso et al., 2008; Kästner et al., 2014): “Bound residues represent compounds in soil, plant or animal which persist in the matrix in

the form of the parent substance or its metabolite(s) after extractions. The extraction method must not substantially change the compounds themselves or the structure of the matrix. The nature of the bond can be clarified in part by matrix-altering extraction methods and sophisticated analytical techniques. To date, for example, covalent, ionic and sorptive bonds, as well as entrapments, have been identified in this way. In general the formation of bound residues reduces the bioaccessibility and the bioavailability significantly.” Führ et al. describe their definition as a modification to the IUPAC definition. This is understandable because both definitions are based on the requirement that SBPR are unextractable with extraction methods that should not substantially or significantly change the SBPR (see Table 1). It is remarkable that the two definitions refer to both soils and plants and the 1998 definition even to animals.

The definition by Führ et al. (1998) contains information on experimental procedures and on properties of SBPR. Information on experimental procedures is not appropriate in a conceptual definition. Furthermore it is strange to include SBPR properties in the definition because these are consequences of the definition, not part of it. In science, it is common to have both a conceptual definition of a concept such as SBPR and one or more operational definitions of such a concept (Fig. 1). The conceptual definition describes a concept in general, abstract or theoretical terms whereas the operational definitions link the concept to the real world by describing the procedures how to observe or measure the concept (e.g. the conceptual definition of adsorption of a substance in a solid–liquid system is commonly ‘accumulation at the solid–liquid interface’ whereas the most common operational definition is to shake a solid–liquid suspension and derive the adsorption from the measured decrease of the concentration in the liquid phase). Fig. 1 shows that the conceptual definition is the basis of the concept and that there may be a number of different parallel operational definitions (e.g. different researchers may use different approaches for measuring SBR of a certain class of pesticides). The figure shows furthermore that properties of SBPR can only be derived after both the conceptual definition and at least one operational definition have been agreed.

The definitions of SBR from IUPAC (1984) and Führ et al. (1998) are founded on non-extractability under laboratory conditions (Table 1). This has historical roots: SBPR research started as the problem of a lacking radioactivity balance in soil metabolism studies, so it was initially the territory of organic chemists which were focussed on laboratory systems rather than on field systems. However, for the environmental risk assessment of SBPR it is more relevant to focus on the behaviour in the field soil. Therefore I propose a conceptual definition of SBPR that is based on their field behaviour. The next sections describe the requirements for such a conceptual definition, the proposed definition and some consequences.

## 2. Requirements for a conceptual definition of soil bound pesticide residues

The proposed conceptual definition of SBPR must fulfil the following requirements: (i) it has to be consistent with current pesticide exposure assessment procedures, in which SBPR are part of the transformation pool of a substance (FOCUS, 2006), (ii) it has to be based on the field

**Table 1**

Schematic representation of the overlaps of and the differences between definitions of soil bound pesticide residues (SBPR); the ‘+’ symbol indicates that the element is part of the definition and ‘–’ that it is not.

Element of definition	Definition of		
	IUPAC (1984)	Führ et al. (1998)	This paper
Non-extractable by extraction methods that do not substantially change the SBPR	+	+	–
Non-extractable by extraction methods that do not substantially change structure of matrix	–	+	–
Recycled fragments leading to natural products are no SBPR	+	–	+
SBPR never desorb under relevant field conditions	–	–	+
SBPR are linked to a specific molecule and SBPR formation is limited to a relevant part of this molecule	–	–	+
Environmental or human toxicological relevance of desorbing molecules included	–	–	+

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