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Distribution of phytoecdysteroids in the Caryophyllaceae

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Dedicated to the memory of Professor Jeffrey B. Harborne

Abstract

Certain genera within the Caryophyllaceae (especially *Silene* and *Lychnis*) have received a significant amount of attention with regard to the isolation and identification of ecdysteroids. However, the taxonomy of this family is difficult. Hence, the occurrence of phytoecdysteroids in members of the Caryophyllaceae is presented, and combined with new data on ecdysteroid agonist (phytoecdysteroid) and antagonist activities, in order to survey the distribution of phytoecdysteroid-containing species within this large family, and to assess the utility of phytoecdysteroids as chemotaxonomic markers. The new data presented (representing ca. 110 species) have been obtained by the application of sensitive biological/biochemical methods for the detection of ecdysteroid agonists and antagonists, using *Drosophila melanogaster* B_{II} bioassay and ecdysteroid-specific immunoassays. In the antagonist version of the B_{II} bioassay, only weak ecdysteroid antagonist activities were detected in a few of the extracts. From both new and previously available data, it was found that phytoecdysteroids were present predominantly in the Genera *Lychnis*, *Petrocoptis*, *Sagina* and *Silene*. Comparison of ecdysteroid occurrence with a molecular phylogeny for the tribe *Sileneae* [Taxon 44 (1995) 525] revealed close association of ecdysteroid occurrence with certain groups of this tribe. In 14 species of *Silene* examined, there is a reasonable, but not absolute, relationship between the presence of ecdysteroids in the seeds and in other plant parts. Where ecdysteroids are present in the plant, highest concentrations are generally present in the roots.

Keywords: Silene; Caryophyllaceae; Phytoecydysteroids; Ecdysteroid; Steroid hormone; Agonist; Antagonist; Bioassay; Chemotaxonomy

1. Introduction

Phytoecdysteroids are insect steroid hormone analogues present in a wide variety of ferns, gymnosperms and angiosperms (Lafont et al., 1991; Dinan, 2001). A survey of 1086 Japanese plants using the *Chilo* dipping test for moulting hormone activity indicated that ca. 6% of these species contained phytoecdysteroids (Imai et al., 1969b). A more recent survey of seeds of 1750 randomly selected species using the B_{II} cell bioassay and ecdysteroid-specific radio-immuno assays (RIAs) also found that 5–6% were positive for phytoecdysteroids and half of the positive species contained moderate to high levels (Dinan, 1995). In some plant species, the concentrations found are very high, e.g. reaching 3.2% of the dry weight in bark of *Diploclisia glaucescens* (Bandara et al., 1989). Phytoecdysteroids are believed to deter invertebrate predators, either by acting as antifeedants or by being toxic (through hormonal disruption) on ingestion (Dinan, 1998).

Over 200 ecdysteroid analogues have been isolated from plant sources (Lafont et al., 2002). The rationale behind the distribution of phytoecdysteroids in the plant world is enigmatic. Since the distribution is very

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uneven, clarification of the rationale could only be expected once a large number of plant species had been assessed within particular families. Recent data on the levels of phytoecdysteroids in seed samples of 200 species within the Chenopodiaceae (Dinan et al., 1998) clearly demonstrated that the presence of ecdysteroids was related to taxonomy within the genus *Chenopodium*, but was less clear-cut for the other large genus in the family *Atriplex*. To extend these studies, we have examined members of the Caryophyllaceae, particularly the *Silene–Melandrium–Lychnis* complex of genera. The borders between these three traditional genera are difficult to delineate and it has been suggested that they be amalgamated into one genus, *Silene*, containing several hundred species allocated to 44 sections (Chowduri, 1957). The distinct advantage of the *Silene* complex for these studies is the extensive previous research on a significant number of species demonstrating that many contain phytoecdysteroids. Also, the taxonomic relationships between species in the complex have been extensively studied both by traditional means and by modern molecular methods. In this report, we extend the available data for the presence or absence of ecdysteroids in members of the Caryophyllaceae, and present a synthesis of our and previous data on phytoecdyteroids in *Silene* spp. in relation to taxonomic position. We also consider the distribution of phytoecdysteroids within growing plants. In future, we shall consider the profiles of phytoecdysteroids present in extracts of seeds and plant parts. Through these studies we wish to shed further light on the chemotaxonomic significance, functions, structural diversity and biosynthesis of phytoecdysteroids, all of which are currently unclear.

2. Results and discussion

2.1. Ecdysteroid occurrence across the Caryophyllaceae

The Caryophyllaceae is a large, widespread family (ca. 2000 species) of mainly northern-hemisphere temperate herbaceous plants, which are allocated to 66 genera (Brummitt, 1992). The centre of distribution is the Mediterranean region and adjoining parts of Europe and Asia. All the larger genera (*Silene*, *Dianthus*, *Arenaria* etc.) are concentrated in this region (Heywood, 1993). The family is generally divided into 3 sub-families: Alsinoideae (*Arenaria*, *Minuartia*, *Honkenya*, *Stellaria*, *Cerastium*, *Sagina*, *Colobanthus*, *Lyallia* etc.), Silenoideae (*Silene*, *Melandrium*, *Dianthus*, *Gypsophila*, *Agrostemma*, *Lychnis* etc.) and Paronychioideae (*Spergula*, *Spergularia*, *Polycarpon* etc.).

The presence or absence of phytoecdysteroids in previously studied species is summarised in Table 1. Phytoecdysteroids have been detected by a variety of means, some more specific and sensitive than others. These

Table 1
Previous literature reports on the presence or absence of phytoecdysteroids (PEs) in species of the Caryophyllaceae

Species	PEs	References
Agrostemma githago	–	Volodin et al. (2002)
Cerastium arvense	–	Revina et al. (1988)
C. davuricum	–	Revina et al. (1988)
C. holosteoides	–	Revina et al. (1988)
	(+)	Volodin et al. (2002)
C. pauciflorum	–	Revina et al. (1988)
Cucubalus baccifer	+	Cheng et al. (2001)
Dianthus hoeltzeri	+	Saatov et al. (1990b)
	+	Saatov et al. (1999)
D. deltoides	(+)	Volodin et al. (2002)
D. superbus	–	Revina et al. (1988)
	(+)	Volodin et al. (2002)
D. versicolor	–	Revina et al. (1988)
Dichodon cerastoides	–	Revina et al. (1988)
	(+)	Volodin et al. (2002)
Elisanthe viscosa	–	Revina et al. (1988)
Eremogone (*Arenaria*) *formosa*	–	Revina et al. (1988)
Eremogone saxatilis	–	Volodin et al. (2002)
Gastrolychnis angustiflora	+	Volodin et al. (2002)
G. (*Melandrium*) *apetala*	+	Zibareva and Revina (1986)
		Revina et al. (1988)
G. (*Melandrium*) *brachypetala*	+	Zibareva and Revina (1986)
		Revina et al. (1988)
G. (*Melandrium*) *tristis*	+	Zibareva and Revina (1986)
		Revina et al. (1988)
Gypsophila altissima	–	Revina et al. (1988)
G. cephalotes	–	Revina et al. (1988)
G. paniculata	–	Revina et al. (1988)
G. patrinii	–	Revina et al. (1988)
G. perfoliata	+	Imai et al. (1969b)
	+	Matsuoka et al. (1969)
G. sericea	–	Revina et al. (1988)
Lychnis alpina	–	Zibareva et al. (1995)
L. arkwrightii	+	Zibareva et al. (1995)
L. chalcedonica	+	Imai et al. (1969a)
	+	Imai et al. (1969b)
	+	Matsuoka et al. (1969)
	+	Revina et al. (1988)
	+	Zibareva and Sviridova (1989)
	+	Zibareva et al. (1991a)
	+	Zibareva et al. (1991b)
	+	Zibareva et al. (1995)
	+	Zibareva et al. (1995)
L. cognata	+	Zibareva et al. (1995)
L. compacta	–	Zibareva et al. (1995)
L. coronaria	+	Abubakirov (1984)
L. flos-cuculi	+	Girault et al. (1990)
	+	Revina et al. (1988)
	+	Louden et al. (2001)
	+	Volodin et al. (2002)
L. fulgens	+	Baltaev et al. (1986)

(*continued on next page*)

Table 1 (*continued*)

Species	PEs	References
	+	Abubakirov (1984)
	+	Zibareva et al. (1995)
L. gracilis	–	Zibareva et al. (1995)
L. haageana	+	Zibareva et al. (1995)
L. miqueliana	+	Imai et al. (1969a)
	+	Imai et al. (1969b)
	+	Matsuoka et al. (1969)
L. samojedorum	+	Volodin et al. (2002)
L. sibirica	+	Zibareva et al. (1995)
L. villosula	+	Zibareva et al. (1995)
L. wilfordii	+	Zibareva et al. (1991a)
	+	Zibareva et al. (1995)
Melandrium (*Vaccaria*) *album*	–	Revina et al. (1988)
	(+)	Volodin et al. (2002)
M. erubescens	+	Abubakirov (1984)
M. (= *Silene*) *nutans*	+	Abubakirov (1984)
	+	Baltaev et al. (1984)
M. ruinarum	+	Abubakirov (1984)
M. turkestanicum	+	Abubakirov (1984)
	+	Saatov et al. (1990c)
	+	Saatov et al. (1991)
	+	Saatov et al (1999)
Minuartia biflora	–	Revina et al. (1988)
M. macrocarpa	(+)	Volodin et al. (2002)
M. verna	–	Revina et al. (1988)
Moehringia lateriflora	–	Revina et al. (1988)
	(+)	Volodin et al. (2002)
M. umbrosa	–	Revina et al. (1988)
Oberna behen	–	Revina et al. (1988)
	–	Volodin et al. (2002)
Sagina procumbens	+	Volodin et al. (2002)
Saponaria officinalis	–	Revina et al. (1988)
Scleranthus annuus	(+)	Volodin et al. (2002)
Silene acaulis	+	Zibareva (1995)
	+	Zibareva (2000)
	+	Volodin et al. (2002)
S. alba	–	Zibareva (2000)
S. alpestris	–	Zibareva (2000)
S. altaica	+	Zibareva (2000)
	+	Zibareva (1999)
	+	Bathori et al. (1995)
S. armeria	–	Zibareva (1997)
	+	Bathori et al. (1995)
	+	Zibareva (2000)
S. antirrhina	+	Meng et al. (2001)
S. asterias	–	Zibareva (2000)
S. bashkirorum	+	Zibareva (2000)
S. bellidifolia	+	Zibareva (2000)
S. boryi	–	Zibareva (2000)
S. brachypoda	+	Zibareva (2000)
S. brahuica	+	Abubakirov (1982)
	+	Saatov et al. (1981)
	+	Saatov et al. (1982a)
	+	Saatov et al. (1982b)
	+	Abubakirov (1984)

Table 1 (*continued*)

Species	PEs	References
	+	Saatov et al. (1984a)
	+	Saatov et al (1984b)
	+	Saatov et al. (1986b)
	+	Dzhukharova et al. (1991)
	+	Saatov et al. (1993)
	+	Dzhukharova et al. (1993)
	+	Dzhukharova et al. (1994)
	+	Dzhukharova et al. (1995)
	+	Sadikov and Saatov (1998)
	+	Saatov et al. (1999)
S. bupleroides	–	Zibareva (2000)
S. campanula	–	Zibareva (2000)
S. campanulata	+	Zibareva (1999)
S. caramanica	+	Zibareva (1999)
S. catholica	+	Zibareva (2000)
	+	Zibareva (1999)
	+	Bathori et al. (1995)
S. chamarensis	+	Saatov et al. (1993)
	+	Revina et al. (1988)
S. chlorantha	+	Sviridova et al. (1995)
	+	Zibareva (1995)
	+	Zibareva (2000)
S. chlorifolia	+	Zibareva (2000)
	+	Zibareva (1999)
	+	Meng et al. (2001)
S. ciliata	+	Zibareva (2000)
	+	Bathori et al. (1995)
S. ciliata var. *graefferi*	+	Zibareva (2000)
S. claviformis	+	Sadikov et al. (2001)
S. coeli-rosa	–	Zibareva (2000)
	+	Bathori et al. (1995)
S. colorata	+	Zibareva and Yeryomina (1996)
	+	Zibareva (1995)
	+	Zibareva (2000)
S. colorata ssp. *trichocalycina*	+	Zibareva (2000)
S. compacta	–	Zibareva (2000)
S. conica	–	Zibareva (1997)
	–	Zibareva (2000)
S. conoidea	–	Zibareva (2000)
S. coronaria	+	Zibareva (1999)
S. cretica	+	Zibareva and Yeryomina (1996)
	+	Zibareva (1997)
	+	Zibareva et al. (1997a)
	+	Meng et al. (2001)
S. densiflora	+	Sviridova et al. (1995)
	+	Zibareva (2000)
S. dichotoma	–	Zibareva (2000)
S. dinarica	–	Zibareva (2000)
S. dioica	+	Girault et al. (1996)
	+	Saatov et al. (1993)
	–	Zibareva (1997)
	–	Zibareva (2000)
S. disticha	+	Zibareva and Yeryomina (1996)
	+	Zibareva (1995)
	+	Zibareva (1997)
	+	Meng et al. (2001)
S. divaricata	–	Zibareva (2000)
S. echinata	+	Meng et al. (2001)
S. fabaria	–	Zibareva (2000)
S. fabariodes	–	Zibareva (2000)
S. firma	+	Zibareva (1999)
S. flavescens	+	Zibareva (1999)

(*continued on next page*)

Table 1 (*continued*)

Species	PEs	References
	+	Bathori et al. (1995)
S. fortunei	+	Gaidi et al. (2002)
S. frivaldszkyana	+	Zibareva et al. (1997b)
	+	Sviridova et al. (1995)
	+	Zibareva (1997)
	+	Louden et al. (2002)
S. fruticosa	+	Zibareva et al. (1997a)
S. fuscata	–	Zibareva (2000)
S. gallica	+	Bergamasco and Horn (1983)
	+	Bathori et al. (1995)
S. gallica var. *quinquevulnera*	+	Zibareva (2000)
	+	Zibareva (1999)
S. gigantea	+	Zibareva (2000)
	+	Zibareva (1999)
S. goulimyi	+	Zibareva (2000)
S. graminifolia	+	Saatov et al. (1993)
	+	Revina et al. (1988)
	+	Zibareva (1997)
S. hifacensis	+	Zibareva (2000)
S. inaperta	–	Zibareva (2000)
S. italica	+	Sviridova et al. (1995)
	+	Zibareva et al. (1997a)
	+	Zibareva (1997)
	+	Zibareva et al. (1997b)
	+	Meng et al. (2001)
S. italica ssp. *nemoralis*	+	Bathori et al. (2002a,b)
	+	Bathori et al. (2000b)
S. jenissensis	+	Saatov et al. (1993)
	+	Revina et al. (1988)
	+	Zibareva (1997)
	+	Zibareva et al. (1997a)
S. laeta	–	Zibareva (2000)
S. latifolia	+	Abubakirov (1982)
	+	Abubakirov (1984)
	+	Saatov et al. (1993)
	+	Bathori et al. (1995)
S. linicola	+	Zibareva and Yeryomina (1996)
	+	Zibareva (1997)
	+	Zibareva et al. (1997a)
	+	Mamadalieva et al. (2002b)
S. longicalycina	+	Abubakirov (1982)
	+	Abubakirov (1984)
	+	Saatov et al. (1993)
	+	Bathori et al. (1995)
S. mellifera	+	Zibareva et al. (1997b)
S. micropetala	+	Zibareva and Yeryomina (1996)
	+	Saatov et al. (1993)
	+	Zibareva (1997)
	+	Bathori et al. (1995)
S. multicaulis	–	Zibareva (2000)
	+	Bathori et al. (1995)
S. multiflora	+	Revina et al. (1988)
	+	Bathori (1998)
S. nemoralis	+	Sviridova et al. (1995)
S. noctiflora	–	Zibareva (2000)
S. nutans	+	Girault et al. (1990)
	+	Bathori et al. (1986a)
	+	Bathori et al. (1987)
	+	Revina et al. (1988)
	+	Baltaev et al. (1985a)
	+	Baltaev et al. (1985b)
	+	Bathori et al. (1986b)
	+	Wilson et al. (1988)

Table 1 (*continued*)

Species	PEs	References
	+	Raynor et al. (1989)
	+	Read et al. (1990)
	+	Lafont et al. (1993)
	+	Davis et al. (1993)
	+	Saatov et al. (1993)
	+	Sviridova et al. (1995)
	+	Zibareva (1997)
	+	Zibareva et al. (1997a)
	+	Ramazanov et al. (1997)
	+	Bathori (1998)
	+	Wilson and Morden (1999)
	+	Zibareva (2000)
	+	Louden et al. (2002)
S. orphanidis	–	Zibareva (2000)
S. otites	+	Girault et al. (1990)
	+	Bathori et al. (1986a)
	+	Bathori et al. (1986b)
	+	Bathori (1986)
	+	Bathori et al. (1988)
	+	Wilson et al. (1988)
	+	Raynor et al. (1989)
	+	Wilson et al. (1990)
	+	Large et al. (1992)
	+	Davis et al. (1993)
	+	Saatov et al. (1993)
	+	Girault et al. (1996)
	+	Bathori et al. (1997)
	+	Zibareva et al. (1997a)
	+	Wilson et al. (1998)
	+	Wilson and Morden (1999)
	+	Wilson et al. (1999)
	+	Bathori et al. (1999)
	+	Zibareva (2000)
	+	Bathori et al. (2000a)
	+	Wilson (2000)
	+	Bathori and Kalasz (2001)
	+	Louden et al. (2002)
S. otites ssp. *hungarica*	+	Bathori et al. (1987)
S. otites parviflorus	+	Zibareva (2000)
S. paradoxa	+	Zibareva (2000)
S. parnassica	+	Zibareva et al. (1997a)
S. patula	+	Zibareva (2000)
S. paucifolia	+	Volodin et al. (2002)
S. polaris	–	Zibareva (1997)
S. portensis	+	Zibareva (1995)
	+	Zibareva (2000)
	+	Meng et al. (2001)
S. praemixta	+	Abubakirov (1982)
	+	Saatov et al. (1979a)
	+	Saatov et al. (1979b)
	+	Abubakirov (1980)
	+	Abubakirov (1984)
	+	Saatov et al. (1985)
	+	Saatov et al. (1993)
S. procumbens	–	Zibareva (2000)
S. pseudotites	+	Zibareva (2000)
	+	Zibareva (1999)
	+	Meng et al. (2001)
S. pseudovelutina	+	Zibareva (2000)
	+	Zibareva (1999)
S. pussila	–	Zibareva (2000)
	+	Dinan et al. (2001)

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Table 1 (*continued*)

Species	PEs	References
S. pygmaea	+	Zibareva (1999)
	+	Bathori et al. (1995)
S. radicosa	+	Zibareva (2000)
	+	Meng et al. (2001)
S. reichenbachii	+	Zibareva (2000)
S. regia	+	Meng et al. (2001)
S. repens	+	Saatov et al. (1993)
	+	Revina et al. (1988)
	+	Zibareva (2000)
S. requienii	–	Zibareva (2000)
S. roemeri	+	Zibareva (2000)
S. rubella	+	Zibareva (2000)
S. scabrifolia	+	Saatov et al. (1986a)
	+	Saatov et al. (1986c)
	+	Saatov et al. (1987a)
	+	Saatov et al. (1987b)
	+	Saatov et al (1990a)
	+	Saatov et al. (1993)
	+	Saatov et al. (1999)
S. schafta	+	Wilson et al. (1988)
	+	Saatov et al. (1993)
S. schumacheri	+	Bathori et al. (1995)
S. schmuckeri	+	Zibareva (2000)
S. schwarzenbergeri	–	Zibareva (2000)
S. secundiflora	+	Zibareva and Yeryomina (1996)
	+	Zibareva (1997)
S. sendtneri	+	Zibareva (1995)
	+	Zibareva (2000)
	+	Bathori et al. (1995)
S. sieberi	+	Zibareva (2000)
S. sobolevskajae	+	Saatov et al. (1993)
	+	Revina et al. (1988)
	+	Ramazanov et al. (1997)
S. succulenta	–	Zibareva (2000)
S. supina	+	Ramazanov et al. (1997)
S. tatarica	+	Girault et al. (1990)
	+	Baltaev et al. (1987)
	+	Saatov et al. (1993)
	+	Bathori and Mathe (1996)
	+	Zibareva and Yeryomina (1996)
	+	Zibareva (1997)
	+	Baltaev (1998)
S. thessalonica	+	Zibareva et al. (1997b)
S. tomentella	+	Ramazanov et al. (1995)
	+	Ramazanov et al. (1996)
S. turgida	–	Revina et al. (1988)
S. vallesia	–	Zibareva (2000)
S. viridiflora	+	Zibareva et al. (1997b)
	+	Zibareva (1995)
	+	Zibareva (1997)
	+	Ramazanov et al. (1997)
S. viscosa	(+)	Volodin et al. (2002)
S. vulgaris	–	Zibareva (1997)
	–	Zibareva (2000)
S. waldsteinii	–	Zibareva (2000)
S. wallichiana	+	Abubakirov (1982)
	+	Abubakirov (1984)
	+	Saatov et al. (1987c)
	+	Saatov et al. (1988)
	+	Saatov et al. (1993)
	+	Saatov et al. (1999)
	+	Mamadalieva et al. (2001)
	+	Mamadalieva et al. (2002a)
S. zawadzkii	–	Zibareva (2000)
	+	Bathori et al. (1995)
Stellaria bungeana	–	Revina et al. (1988)
S. crassifolia	–	Volodin et al. (2002)
S. hebecalyx	–	Volodin et al. (2002)
S. holostea	–	Revina et al. (1988)
	+	Volodin et al. (2002)
S. nemorum	(+)	Volodin et al. (2002)
S. palustris	(+)	Volodin et al. (2002)
S. umbellata	–	Revina et al. (1988)
Steris viscaria	(+)	Volodin et al. (2002)
Spergula arvensis	+	Volodin et al. (2002)
Spergularia rubra	(+)	Volodin et al. (2002)

– ecdysteroid-negative; + ecdysteroid-positive; (+) weakly ecdysteroid-positive.

methods range from activity in bioassays (e.g. the *Chilo* dipping test; Sato et al., 1968) to isolation and full spectroscopic identification. The vast majority of these studies have considered only one or a few species in the Caryophyllaceae and report findings for ecdysteroid-positive species. For more extensive screening, it was necessary to apply a simple, uniform, sensitive and specific strategy. The strategy employed, using a rapid and robust bioassay and ecdysteroid-specific RIAs, has been described previously (Dinan, 1995). In view of the methodological diversity of previous studies, it was worthwhile examining some species which had been assessed previously, in order to compare and confirm data, as well as extending the studies to a selection of new species. The data from this study are summarised in Table 2.

Table 3 summarises the available data for the presence of ecdysteroids in species within the Caryophyllaceae. There are 66 recognised genera, of which 21 have been investigated to some extent for ecdysteroids. 323 species have been tested for the presence of ecdysteroids, of which 125 are positive and 23 are uncertain. However, it must be stressed here that previously published studies have focussed on positive species. No positive species have been detected in some genera: *Arenaria*, *Cerastium*, *Gypsophila*, *Minuartia*, *Spergula*, *Spergularia* and *Stellaria*. Many positive species occur in other genera: *Lychnis*, *Petrocoptis* and *Silene*. Most data regarding ecdysteroid distribution are available for the *Silene*/*Lychnis* complex, into which *Melandrium* has been subsumed.

The Flora Europeae (Tutin et al., 1964) recognises 37 genera in the Caryophyllaceae, divided into three subfamilies: Alsinoideae, Paronychioideae and Silenoideae. All the representatives of the Alsinoideae tested for

Table 2
Species of the Caryophyllaceae for which the seeds were tested for the presence of ecdysteroid agonist and antagonist activities

Species[a]	RIA[b]		Bioassay[c]	
	Black	DBL-1	Agonist	Antagonist
Agrostemma githago (f)	–	–	–	–
Arenaria balearica (c)	–	–	–	–
Arenaria erinacea (b)	–	–	–	–
Arenaria montana (b)	–	–	–	–
Arenaria obtusiloba (e)	–	–	–	–
Cerastium alpinum (b)	–	–	–	–
Cerastium biebersteinii (b)	–	–	–	–
Cerastrium boissieri (c)	–	–	–	–
Cerastium tomentosum (h)	–	–	–	–
Dianthus amurensis (e)	–	–	–	–
Dianthus arenarius (b)	–	–	–	–
Dianthus armeria (b)	–	–	–	–
Dianthus × *arvenensis* (b)	–	–	–	–
Dianthus barbatus (b)	–	–	–	–
Dianthus carthusianorum (b)	–	–	–	–
Dianthus caryophyllus (b)	–	–	–	–
Dianthus chinensis (b)	–	–	–	–
Dianthus deltoides (b)	–	–	–	–
Dianthus gratianopolitanus (b)	–	–	–	–
Dianthus hungaricus (e)	–	–	–	+
Dianthus kitaibelii (b)	–	–	–	–
Dianthus knappii (b)	–	–	–	–
Dianthus plumarius (b)	–	–	–	–
Dianthus seguieri (b)	–	–	–	–
Dianthus shinanensis (e)	–	–	–	–
Dianthus subacaulis (b)	–	–	–	–
Drypis spinosa (b)	–	–	–	–
Gypsophila elegans (b)	–	–	–	–
Gypsophila pacifica (b)	–	1.24	–	–
Gypsophila paniculata (b)	–	–	–	–
Gypsophila repens (b)	–	–	–	–
Lychnis apetala (a)	–	–	–	–
Lychnis × *arkwrightii* (b)	184.5	1170	+ + +	–
Lychnis chalcedonica (b)	85.0	1054	+ + +	–
Lychnis coronaria (b)	27.0	521.8	+ + +	–
Lychnis coronaria occulata (g)	20	3300	+ + +	–
Lychnis flos-cuculi (f)	11.5	1375	+ + +	–
Lychnis flos-cuculi nana (d)	5.2	287.0	+ + +	–
Lychnis flos-jovis (b)	11.0	646.7	+ + +	–
Lychnis flos-jovis (b)	19.0	712.0	+ + +	–
Lychnis × *haageana* (b)	46.0	659.0	+ + +	–
Lychnis miqueliana (b)	72.0	1323	+ + +	–
Lychnis plena (a)	20.5	529.1	+ + (+)	–
Lychnis viscaria (b)	0.65	13.84	+ +	–
Lychnis viscaria (b)	–	–	–	–
Lychnis wilfordii (a)	82.0	656.0	+ +	–
Lychnis yunnanensis (b)	–	–	–	–
Minuartia laricifolia (b)	–	–	–	–
Minuartia verna (b)	–	–	–	–
Myosoton aquaticum (e)	–	–	–	–
Petrocoptis glaucifolia (a)	68.4	413.8	+ + +	–
Petrocoptis hispanica (c)	61.0	381.0	+ + (+)	C–
Petrocoptis hispanica (c)	68.0	377.0	+ + +	–
Petrocoptis pyrenaica (a)	11.5	837.7	+ +	–
Petrorhagia prolifera (c)	–	–	C–	C–
Petrorhagia saxifraga (b)	–	–	–	–
Sagina subulata (b)	–	NL	–	–
Saponaria calabrica (b)	–	–	–	–
Saponaria lutea (b)	–	–	–	–
Saponaria officinalis (b)	–	–	–	–
Saponaria ocymoides (b)	–	–	–	–
Saponaria pumilla (b)	–	–	–	–
Saponaria vaccaria (b)	–	–	–	–
Silene acaulis (b)	4.14	404.1	+ +	–
Silene alba (b)	–	–	–	–
Silene alpestris (b)	–	2.28	–	–
Silene antirrhina (*i*)	nd	87.2	+ +	–
Silene apetala (*i*)	nd	148.8	+ +	–
Silene armeria (b)	–	–	–	–
Silene asterias (b)	–	–	–	–
Silene bellidifolia (a)	13.4	641.8	+ + +	–
Silene bellidioides (a)	–	–	–	–
Silene burchellii (a)	177.4	7321	+ + + +	–
Silene campanula (g)	0.38	7.96	+	–
Silene caroliniana (g)	0.08	–	–	–
Silene clandestina (a)	17.9	2311	+ + +	–
Silene coeli-rosa (b)	–	–	–	–
Silene colorata (g)	–	424.0	+ + +	–
Silene colorata (*i*)	nd	261.3	+ + +	–
Silene conica (b)	–	–	–	–
Silene coronaria (*i*)	nd	137.8	+ + (+)	–
Silene cretica (*i*)	nd	49.1	+ +	–
Silene dioica (f)	–	–	–	–
Silene dioica (b)	–	–	–	–
Silene disticha (*i*)	nd	109.5	+ +	–
Silene echinata (*i*)	nd	101.4	+ +	–
Silene fridvaldskyana (a)	1.06	160.0	+ + +	–
Silene gallica (*i*)	nd	109.8	+ +	–
Silene gallica ssp *quinquevulera* (g)	3.24	539.8	+ + (+)	–
Silene italica (a)	50.5	1086	+ + +	–
Silene keiskei (a)	2.46	200.0	+ +	–
Silene laciniata (a)	48.0	2000	+ + +	–
Silene latifolia (a)	–	0.39	–	–
Silene linicola (*i*)	nd	1485	+ + + (+)	–
Silene maritima (b)	–	–	–	–
Silene mellifera (*i*)	nd	107.5	+ + +	–
Silene multiflora (a)	–	–	–	–
Silene noctiflora (a)	–	–	–	–
Silene nutans (a)	14.0	1147	+ + +	–
Silene otites ssp. *hungarica* (a)	403.0	2688	+ + +	C–
Silene pendula (f)	–	–	–	–
Silene pendula (b)	–	–	–	–
Silene portensis (*i*)	nd	86.2	C + +	C–
Silene pusilla (e)	0.75	4.4	+	–
Silene regia (b)	34.9	1395	+ + +	–
Silene regia (a)	52.5	2364	C + +	C–
Silene roemeri (*i*)	nd	199.7	+ + (+)	–
Silene rupestris (a)	–	–	–	–
Silene saxifraga (a)	30.0	250.0	+ +	–
Silene scabriflora	nd	483.5	+ + (+)	–
Silene schafta (b)	3.24	585.7	+ + +	–
Silene sendtneri (*i*)	nd	267.1	+ + +	–
Silene squamigera (*i*)	nd	16.08	+	–
Silene tatarica (*i*)	nd	108.4	+ +	–
Silene thessalonica (a)	76.0	1013	+ + (+)	–
Silene undulata (a)	0.15	3.27	+	–
Silene uniflora (a)	–	–	–	–
Silene vallesia (a)	–	0.14	–	–
Silene vallesia (b)	–	–	–	–

(*continued on next page*)

Table 2 (*continued*)

Species[a]	RIA[b]		Bioassay[c]	
	Black	DBL-1	Agonist	Antagonist
Silene virginica (b)	91.5	237.9	+ +	–
Silene viridiflora (*i*)	nd	659.0	+ + +	–
Silene vulgaris (b)	–	–	–	–
Silene zawadskii (a)	–	–	–	–
Spergularia media (e)	–	–	–	–
Spergularia rubra (b)	–	–	–	–
Stellaria holostea (b)	–	–	–	–
Stellaria media (b)	–	–	–	–
Vaccaria elisabethae (b)	–	1.0	–	–
Vaccaria pyrimidalis (g)	–	–	–	–
Viscaria alpina (b)	–	–	–	–

[a] Source: (a) B & T World Seeds; (b) Chilterns Seeds, Ulverston, Cumbria, U.K.; (c) Ness Botanical Gardens, Wirral, U.K.; (d) Plant World, Newton Abbot, Devon, U.K.; (e) Seed Guild; (f) Suttons Seeds; (g) Thompson & Morgan; (h) Unwins Seeds; (i) Siberian Botanical Garden, Tomsk, Russia.

[b] RIA results expressed in μg ecdysone equivalent/g; NL: non-linear response; nd: not determined.

[c] Bioassay results (20 μl aliquots); – not active, + active as neat extract, + + active as 10-fold dilution, + + + active as 100-fold dilution, + + + + active as 1000-fold dilution. C = cytotoxic at relevant dilution.

phytoecdysteroids are negative, with the exception of the genus *Sagina*, where four species have been found to be ecdysteroid-positive. The predominantly edysteroid-positive genera of *Lychnis*, *Petrocoptis* and *Silene* are in the Silenoideae. However, other genera in the Silenoideae appear to be ecdysteroid-negative: *Agrostemma*, *Drypis*, *Gypsophila*, *Petrorhagia* and *Dianthus*. Very few genera in the Paronychioideae have been investigated for ecdysteroid content; only three species in the genera *Spergula* (two positive species and one negative) and two in *Spergularia* (both negative) have been examined.

2.2. Relationship to morphology-based classification

In further consideration of the chemotaxonomic significance, we shall focus on the *Silene*/*Lychnis* complex. Chowdhuri (1957) grouped 443 species of *Silene* into 44 sections, some of which were further divided into subsections. Ecdysteroid data exist for 97 of the species which Chowdhuri recognised. Some sections are essentially ecdysteroid-negative: Gastrolychnis, Inflatae, Compactae, Melandriformes, Coniomorpha. Some sections consist of almost only ecdysteroid-positive species: Paniculate, Occidentales, Siphonomorpha, Chloranthae, Tataricae, Graminifoliae, Otites, Fruticulosae and Silene. Other sections contain both ecdysteroid-positive and ecdysteroid-negative species: Sclerocalycinae, Chloranthae, Suffruticosae, Auriculatae, Macranthae, Lasiocalycinae, Scorpioideae. Too few of the named species have been tested for other sections to be certain into which category they fall. It would be necessary to obtain data for many more of the named species, but even then the morphology-based taxonomic divisions remain uncertain.

2.3. Relationship to molecular taxonomy

Oxelman and Lidén (1995) considered the phylogenetic organisation of 64 species of *Silene*/*Lychnis*/*Petrocoptis*, as inferred from the sequences of the internal transcribed spacer (ITS) regions and the intervening 5.8S gene of the nuclear ribosomal DNA. The derived phylogenetic tree divides into five major branches, of which the largest (Group V: the *Silene* group) divides into two major sub-groups and these divide further into sub-sub-groups. The molecular tree (Fig. 1) bears limited resemblance to the traditional morphology-based taxonomical organisations. Branches I (Eudianthe) and III (Steris group) are ecdysteroid-negative. Groups II (Petrocoptis), IV (*Lychnis* group) and Va are mainly ecdysteroid-positive. Group V is predominantly ecdysteroid-negative, which may be indicative of secondary loss of ecdysteroid accumulation after the branch point to *Cucubalus baccifer*, which is ecdysteroid-positive.

Oxelman et al. (1997) extended their study of the Sileneae to analysis of the chloroplast rps16 intron, and the resultant phylogenetic relationships supported the conclusions from the ITS sequences, and the authors have used the combined ITS and rps16 intron data in order to revise the classification of the tribe Sileneae (Oxelman et al., 2000).

2.4. Distribution of ecdysteroids in individual plants

The presence of ecdysteroids throughout individual plants (2–12 per species) of several species of *Silene* was assessed in plants of different ages (38–300 days). The following species, for which the seeds are all ecdysteroid-negative, were found to be essentially ecdysteroid-negative for other plant parts: *S. alba*, *S. armeria*, *S. coeli-rosa*, *S. pendula*, *S. vulgaris* and *S. zawadski*. However, even in these species low levels of ecdysteroids (< 3.5 μg ecdysone equivalents/g d.w. with the DBL-1 antiserum) were detected in some plant parts (consistently in flowers and leaves of *S. armeria*, but erratically in *S. coeli-rosa*, *S. vulgaris* and *S. zawadski*). In *S. armeria*, older flowering plants (> 43 days) all possessed detectable ecdysteroids in the flowers and leaves, but younger (non-flowering) plants (22 and 38 days) did not possess detectable ecdysteroid levels in any plant part. There was no observable relationship to age (or developmental status) for the other species. Seeds of *S. multiflora* are ecdysteroid-negative, but other plant parts contain low, but definite, levels of ecdysteroids (1–17 μg/g).

Plants of the following species were consistently ecdysteroid-positive: *S. bellidifolia*, *S. colorata*, *S. latifolia*, *S. otites hungarica*, *S. pusilla*, *S. saxifraga* and

Table 3
Distribution of ecdysteroids in the Caryophyllaceae

Genera and species	Ecdysteroids	References
Acanthophyllum (48 species)		
A. gypsophyloides	–	Saatov et al. (unpublished)
Agrostemma (9 species)		
A. bicolor	–	Zibareva et al. (unpublished)
A. githago	–	Dinan et al. (unpublished)
A. gracilis	–	Zibareva et al. (unpublished)
Allochrusa (6 species)		
Alsinidendron (3 species)		
Ankyropetalum (1species)		
Arenaria (several hundred species)		
A. aggregata	–	Zibareva et al. (unpublished)
A. balearica	–	Dinan et al. (unpublished)
A. erinacea	–	Dinan et al. (unpublished)
A. montana	–	Dinan et al. (unpublished)
A. obtusiloba	–	Dinan et al. (unpublished)
A. pundens	–	Zibareva et al. (unpublished)
Bolanthus (1 species)		
Brachystemma (2 species)		
Brewerina (1 species)		
Bufonia (10 species)		
Cerastium (several hundred species)		
C. alpinum	–	Dinan et al. (unpublished)
C. arvense	–	Revina et al. (1988)
C. biebersteinii	–	Dinan et al. (unpublished)
C. boissieri	–	Dinan et al. (unpublished)
C. candidissimum	–	Zibareva et al. (unpublished)
C. cerastoides	–	Volodin et al. (unpublished)
C. cespitosum	–	Volodin et al. (unpublished)
C. davuricum	–	Revina et al. (1988)
C. holosteoides	–/(+)	Revina et al. (1988)
C. pauciflorum	–	Revina et al. (1988)
C. tomentosum	–	Dinan et al. (unpublished)
C. triviale	–	Zibareva et al. (unpublished)
Cerdia (2 species)		
Colobanthus (22 species)		
Cucubalus (16 species)		
C. baccifer	+/–	Cheng et al. (2001)/Zibareva et al. (unpublished)
Cyanthophylla (? Species)		
Dianthus (several hundred species)		
D. amurensis	–	Dinan et al. (unpublished)
D. arenarius	–	Dinan et al. (unpublished)
D. armeria	–	Dinan et al. (unpublished)
D.×arvenensis	–	Dinan et al. (unpublished)
D. barbatus	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
D. callizonus	–	Dinan et al. (unpublished)
D. carthusianorum	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
D. caryophyllus	–	Zibareva et al. (unpublished); Dinan et al. (unpublished); Blackford and Dinan (1997b)
D. chinensis	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
D. deltoides	–/(+)	Dinan et al. (unpublished)/Zibareva et al. (unpublished); Volodin et al. (unpublished)
D. fragrans	–	Zibareva et al. (unpublished)
D. furcatus ssp. *furcatus*	–	Dinan et al. (unpublished)
D. gratianopolitanus	–	Dinan et al. (unpublished)
D. hoeltzeri	+	Saatov et al. (1990b); Saatov et al. (1999)
D. hungaricus	–	Dinan et al. (unpublished)
D. kitaibelii	–	Dinan et al. (unpublished)
D. knappii	–	Dinan et al. (unpublished)
D. masmenaeus	–	Zibareva et al. (unpublished)
D. monspessulanus	–	Zibareva et al. (unpublished)
D. petraeus ssp. *noaeanus*	–	Dinan et al. (unpublished)
D. petraeus ssp. *petraeus*	–	Dinan et al. (unpublished)
D. plumarius	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)

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Table 3 (*continued*)

Genera and species	Ecdysteroids	References
D. seguieri	–	Dinan et al. (unpublished)
D. shinanensis	–	Dinan et al. (unpublished)
D. sylvestris	–	Zibareva et al. (unpublished)
D. subacaulis	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
D. superbus	–/(+)	Zibareva et al. (unpublished); Revina et al. (1988); Volodin et al. (unpublished)
D. versicolor	–	Revina et al. (1988)
D. waldsteinii	–	Zibareva et al. (unpublished)
Diaphanoptera (1 species)		
Drymaria (92 species)		
Drypis (1 species)		
D. spinosa	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
Gypsophila (117 species)		
G. acutifolia	–	Zibareva et al. (unpublished)
G. altissima	–	Zibareva et al. (unpublished); Revina et al. (1988)
G. cephalotes	–	Revina et al. (1988)
G. elegans	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
G. libanotica	–	Zibareva et al. (unpublished)
G. muralis	–	Zibareva et al. (unpublished)
G. pacifica	–	Dinan et al. (unpublished)
G. paniculata	–	Zibareva et al. (unpublished); Dinan et al. (unpublished); Revina et al (1988)
G. patrinii	–	Revina et al. (1988)
G. perfoliata	+	Imai et al. (1969b); Matsuoka et al. (1969)
G. pilosa	–	Zibareva et al. (unpublished)
G. repens	–	Dinan et al. (unpublished)
G. sericea	–	Revina et al. (1988)
G. silenoides	–	Zibareva et al. (unpublished)
G. trichotoma	–	Zibareva et al. (unpublished)
Habrosia (1 species)		
Hockenya (? species)		
H. peploides	–	Zibareva et al. (unpublished)
Holosteum (11 species)		
H. umbellatum	–	Zibareva et al. (unpublished)
Krauseola (2 species)		
Lepyrodiclis (6 species)		
Loeflingia (9 species)		
Lychnis (89 species)		
L. alpina	–	Dinan et al. (unpublished); Zibareva et al. (1995)
L. apetala	–	Dinan et al. (unpublished)
L.×arkwrightii	+	Dinan et al. (unpublished); Zibareva et al. (1995)
L. chalcedonica	+	Saatov et al. (unpublished); Zibareva et al. (unpublished); Dinan et al. (unpublished); Revina et al. (1988); Imai et al. (1969b); Matsuoka et al. (1969); Zibareva et al. (1995); Imai et al. (1969a); Zibareva and Sviridova (1989); Zibareva et al. (1991a)
L. cognata	+	Zibareva et al. (1995)
L. compacta	–	Zibareva et al. (1995)
L. coreacea	+	Saatov et al. (unpublished)
L. coronaria	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Abubakirov (1984)
L. coronaria occulata	+	Dinan et al. (unpublished)
L. flos-cuculi	+	Saatov et al. (unpublished); Dinan et al. (unpublished); Revina et al. (1988); Volodin et al. (unpublished)]; Girault et al. (1990)
L. flos-cuculi nana	+	Dinan et al. (unpublished)
L. flos-jovis	+	Zibareva et al. (unpublished); Dinan et al. (unpublished)
L. fulgens	+	Saatov et al. (unpublished); Zibareva et al. (1995); Abubakirov (1984); Baltaev et al. (1986)
L. gracilis	–	Zibareva et al. (1995)
L.×haageana	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva et al. (1995)
L. kiusiana	+	Zibareva et al. (unpublished)
L. miqueliana	+	Dinan et al. (unpublished); Imai et al. (1969b); Matsuoka et al. (1969); Imai et al. (1969a); Zibareva et al. (unpublished)
L. plena	+	Dinan et al. (unpublished)
L. samojedorum	+	Volodin et al. (unpublished)
L. sibirica	+	Zibareva et al. (1995)
L. villosula	+	Zibareva et al. (1995)

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Table 3 (*continued*)

Genera and species	Ecdysteroids	References
L. viscaria	+/–	Dinan et al. (unpublished)
L. wilfordii	+	Dinan et al. (unpublished); Zibareva et al. (1995); Zibareva et al. (1991a)
L. yunnanensis	–	Dinan et al. (unpublished)
Mesostemma (5 species)		
Microphyes (5 species)		
Minuartia (ca. 120 species)		
M. baldaccii	–	Zibareva et al. (unpublished)
M. biflora	–	Revina et al. (1988)
M. campestris	–	Zibareva et al. (unpublished)
M. fastigiata	–	Zibareva et al. (unpublished)
M. hybrida	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
M. kashmirica	–	Zibareva et al. (unpublished)
M. laricifolia	–	Dinan et al. (unpublished)
M. macrocarpa	(+)	Volodin et al. (2002)
M. pinifolia	–	Zibareva et al. (unpublished)
M. setacea	–	Zibareva et al. (unpublished)
M. verna	–	Dinan et al. (unpublished); Revina et al. (1988)
Moehringia (13 species)		
M. lateriflora	–	Revina et al. (1988); Volodin et al. (unpublished)
M. umbrosa	–	Revina et al. (1988)
Moenchia (5 species)		
M. mantica	–	Zibareva et al. (unpublished)
Myosoton (1 species)		
M. aquaticum	–	Dinan et al. (unpublished)
Ochotonophila (? species)		
Ortegia (? species)		
O. hispanica	–	Zibareva et al. (unpublished)
Pentastemonondiscus (1 species)		
Petrocoptis (2 species)		
P. glaucifolia	+	Zibareva et al. (unpublished); Dinan et al. (unpublished)
P. hispanica	+	Dinan et al. (unpublished)
P. pyrenaica	+	Zibareva et al. (unpublished); Dinan et al. (unpublished)
Petrorhagia (15 species)		
P. nanteuilli	–	Zibareva et al. (unpublished)
P. prolifera	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
P. saxifraga	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
Phyrna (? species)		
Pinosia (1 species)		
Pirinia (? species)		
Pleioneura (5 species)		
Polycarpaea (34 species)		
P. smithii	–	Zibareva et al. (unpublished)
Polycarpon (20 species)		
P. tetraphyllum	–	Zibareva et al. (unpublished)
Polytepalum (1 species)		
Pseudostellaria (14 species)		
Pycnophyllopsis (2 species)		
Pycnophyllum (28 species)		
Robbairea (1 species)		
Sagina (ca. 80 species)		
S. apetala	+	Zibareva et al. (unpublished)
S. ciliata	+	Zibareva et al. (unpublished)
S. maritima	+	Zibareva et al. (unpublished)
S. procumbens	+	Zibareva et al. (unpublished); Volodin et al. (unpublished)
S. saginoides	–	Zibareva et al. (unpublished)
S. subulata	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
Sanctambrosia (1 species)		
Saponaria (23 species)		
S. bellidifolia	+	Zibareva et al. (unpublished)
S. caespitosa	–	Zibareva et al. (unpublished)
S. calabrica	–/+	Dinan et al. (unpublished)/Zibareva et al. (unpublished)
S. cerastoides	–	Zibareva et al. (unpublished)

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Table 3 (*continued*)

Genera and species	Ecdysteroids	References
S. cypria	–	Zibareva et al. (unpublished)
S. haussknechtii	–	Zibareva et al. (unpublished)
S. lutea	–	Dinan et al. (unpublished)
S. ocymoides	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. officinalis	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. pumilla	–	Dinan et al. (unpublished)
S. sicula	–	Zibareva et al. (unpublished)
S. vaccaria	–	Dinan et al. (unpublished)
Schiedea (32 species)		
Scleranthopsis (1 species)		
Silene (>700 species)		
S. acaulis	+	Dinan et al. (unpublished); Volodin et al. (unpublished); Zibareva (2000); Zibareva (1995)
S. acutifolia	–	Zibareva et al. (unpublished)
S. aegyptica	–	Zibareva et al. (unpublished)
S. alba	–	Dinan et al. (unpublished); Volodin et al. (unpublished); Zibareva (2000)
S. alpestris	–	Dinan et al. (unpublished); Zibareva (2000)
S. altaica	+	Zibareva et al. (unpublished); Zibareva (2000); Zibareva (1999); Bathori et al. (1995)
S. antirrhina	+	Zibareva et al. (unpublished); Meng et al. (2001)
S. apetala	+	Zibareva et al. (unpublished)
S. armeria	–/+	Dinan et al. (unpublished); Zibareva (1997)/Zibareva (2000); Bathori et al. (1995)
S. asterias	–	Dinan et al. (unpublished); Zibareva (2000)
S. bashkirorum	+	Zibareva (2000)
S. bellidifolia	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva (2000)
S. bellidioides	–	Dinan et al. (unpublished)
S. boryi	–	Zibareva (2000)
S. brachypoda	+	Zibareva (2000)
S. brahuica	+	Saatov et al. (unpublished); Saatov et al. (1999); Abubakirov (1984); Bathori et al. (1995); Abubakirov (1982); Saatov et al. (1981); Saatov et al. (1982a); Saatov et al. (1982b); Saatov et al. (1984a); Saatov et al. (1984b); Saatov et al. (1986b); Dzhukharova et al. (1991); Saatov et al. (1993); Dzhukharova et al. (1993); Dzhukharova et al. (1994a); Dzhukharova et al. (1994b); Sadikov and Saatov (1998); Sadikov and Saatov (1999); Sadikov et al. (2000)
S. bupleroides	–	Zibareva (2000)
S. burchellii	+	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. campanula	–	Dinan et al. (unpublished); Zibareva (2000)
S. campanulata	+	Zibareva (1999)
S. caramanica	+	Zibareva (1999)
S. caroliniana	–	Dinan et al. (unpublished)
S. catholica	+	Zibareva et al. (unpublished); Zibareva (2000); Zibareva (1999); Bathori et al. (1995)
S. caucasica	+	Zibareva et al. (unpublished)
S. chamarensis	+	Revina et al. (1988); Saatov et al. (1993)
S. chlorantha	+	Zibareva et al. (unpublished); Zibareva (2000); Zibareva (1995); Sviridova et al. (1995)
S. chlorifolia	+	Zibareva et al. (unpublished); Zibareva (2000); Meng et al. (2001)
S. ciliata	+	Zibareva et al. (unpublished); Zibareva (2000); Bathori et al. (1995)
S. ciliata var. *graefferi*	+	Zibareva (2000)
S. clandestina	+	Dinan et al. (unpublished)
S. claviformis	+	Saatov et al. (unpublished)
S. coeli-rosa	–/+	Dinan et al. (unpublished); Zibareva (2000); Bathori et al. (1995)
S. colorata	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva (2000); Zibareva (1995); Zibareva and Yeryomina (1996)
S. colorata ssp. *trichocalycina*	+	Zibareva (2000)
S. compacta	–	Zibareva (2000)
S. conica	–	Dinan et al. (unpublished); Zibareva (2000); Zibareva (1997)
S. conoidea	–	Zibareva (2000)
S. coronaria	+	Zibareva et al. (unpublished); Zibareva (1999)
S. cretica	+	Zibareva et al. (unpublished); Zibareva (1997); Zibareva and Yeryomina (1996); Zibareva et al. (1997a)
S. cucubalus	–/+	Zibareva et al. (unpublished); Volodin et al. (unpublished)/Dinan et al. (unpublished)
S. damboldtiana	+	Zibareva (1999)
S. densiflora	+	Zibareva (2000); Sviridova et al. (1995)
S. dichotoma	–	Zibareva (2000)

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Table 3 (*continued*)

Genera and species	Ecdysteroids	References
S. dinarica	–	Zibareva (2000)
S. dioica	–/+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva (2000)/Zibareva (1997); Saatov et al. (1993); Girault et al. (1996)
S. disticha	+	Zibareva et al. (unpublished); Zibareva (1997); Zibareva (1995); Zibareva and Yeryomina (1996)
S. divaricata	–/(+)	Zibareva (2000)/Zibareva et al. (unpublished)
S. dubia	–	Zibareva et al. (unpublished)
S. echinata	+	Zibareva et al. (unpublished)
S. elegans	+	Zibareva et al. (unpublished)
S. exscapa	–	Zibareva et al. (unpublished)
S. fabaria	–	Zibareva (2000)
S. fabariodes	–	Zibareva (2000)
S. firma	+	Zibareva et al. (unpublished) Zibareva (1999)
S. flavescens	+	Zibareva (1999); Bathori et al. (1995)
S. foetida	–	Zibareva et al. (unpublished)
S. fortunei	+	Gaidi et al. (2002)
S. frivaldszkyana	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva (1997); Sviridova et al. (1995); Zibareva et al. (1997b)
S. fruticosa	+	Zibareva et al. (unpublished); Zibareva et al. (1997a)
S. fuscata	–	Zibareva (2000)
S. gallica	+	Zibareva et al. (unpublished); Bathori et al. (1995); Bergamasco and Horn (1983)
S. gallica var. *quinquevulnera*	+	Dinan et al. (unpublished); Zibareva (2000); Zibareva (1999)
S. gigantea	+	Zibareva et al. (unpublished); Zibareva (2000); Zibareva (1999)
S. giraldi	–	Zibareva et al. (unpublished)
S. goulimyi	+	Zibareva (2000)
S. graminifolia	+	Zibareva et al. (unpublished); Revina et al. (1988); Zibareva (1997); Saatov et al. (1993)
S. gyntensis	+	Saatov et al. (unpublished)
S. hifacensis	+	Zibareva (2000)
S. hupanica	–	Zibareva et al. (unpublished)
S. inaperta	–	Zibareva (2000)
S. italica	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva (1997); Sviridova et al. (1995); Zibareva et al. (1997a); Meng et al. (2001)
S. italica ssp. *nemoralis*	+	Bathori et al. (2000c); Bathori et al. (2002a); Bathori et al. (2002b); Bathori et al. (2002c); Pongracz et al. (2003)
S. jennissensis	+	Zibareva et al. (unpublished); Revina et al. (1988); Zibareva (1997) Saatov et al. (1993); Zibareva et al. (1997a)
S. keiskei	+	Dinan et al. (unpublished)
S. laciniata	+	Dinan et al. (unpublished)
S. laeta	–	Zibareva (2000)
S. lagunensis	+	Dinan et al. (unpublished)
S. latifolia	–/+	Zibareva et al. (unpublished); Dinan et al. (unpublished)/Abubakirov (1984); Bathori et al. (1995); Abubakirov (1982); Saatov et al. (1993)
S. linicola	+	Saatov et al. (unpublished); Zibareva et al. (unpublished); Zibareva (1997); Zibareva & Yeryomina (1996); Zibareva et al. (1997a)
S. littorea	–	Zibareva et al. (unpublished)
S. longicalycina	+	Abubakirov (1984); Bathori et al. (1995); Abubakirov (1982); Saatov et al. (1993)
S. longicilia	+	Zibareva et al. (unpublished)
S. macrorhiza	–	Zibareva et al. (unpublished)
S. maritima	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. mellifera	+	Blackford & Dinan (1997b); Zibareva et al. (1997b)
S. micropetala	+	Bathori et al. (1995); Zibareva (1997); Saatov et al. (1993); Zibareva and Yeryomina (1996)
S. molissima	+	Zibareva et al. (unpublished)
S. multicaulis	–/+	Zibareva (2000)/Bathori et al. (1995)
S. multiflora	–/+	Dinan et al. (unpublished)/Revina et al. (1988)
S. muscipula	+/–	Dinan et al. (unpublished)/Zibareva et al. (unpublished)
S. nemoralis	+	Sviridova et al. (1995)
S. noctiflora	–	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva (2000)
S. nocturna	–	Zibareva et al. (unpublished)
S. nutans	+	Dinan et al. (unpublished); Revina et al. (1988); Girault et al. (1990); Zibareva (2000); Bathori et al. (1995); Zibareva (1997); Saatov et al. (1993); Sviridova et al. (1995); Zibareva et al. (1997a); Bathori et al. (1986a,b); Bathori et al. (1987); Baltaev et al. (1985a); Baltaev et al. (1985b); Raynor et al. (1989); Ramazanov et al. (1997); Wilson and Morden (1999)

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Table 3 (*continued*)

Genera and species	Ecdysteroids	References
S. oligantha	+	Zibareva et al. (unpublished)
S. olympica	–	Zibareva et al. (unpublished)
S. orphanidis	–	Zibareva (2000)
S. otites	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Girault et al. (1990); Zibareva (2000); Bathori et al. (1995); Saatov et al. (1993); Zibareva et al. (1997a); Girault et al. (1996); Bathori et al. (1986a,b); Raynor et al. (1989); Wilson & Morden (1999); Bathory et al. (1986); Bathori et al. (1986b); Bathori et al. (1988); Wilson et al. (1990); Bathori et al. (1997); Wilson et al. (1998); Wilson et al. (1999); Bathori et al. (1999)
S. otites ssp. *hungarica*	+	Dinan et al. (unpublished); Bathori et al. (1987)
S. otites parviflorus	+	Zibareva et al. (unpublished); Zibareva (2000)
S. paradoxa	+	Zibareva et al. (unpublished); Zibareva (2000)
S. parnassica	(+)/+	Zibareva et al. (unpublished)/Zibareva et al. (1997a)
S. patula	+	Zibareva (2000)
S. paucifolia	+	Volodin et al. (unpublished)
S. pendula	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. persica	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. polaris	–	Zibareva (1997)
S. portensis	+	Zibareva et al. (unpublished); Zibareva (2000); Zibareva (1995); Meng et al. (2001)
S. praemixta	+	Saatov et al. (unpublished); Abubakirov (1984); Abubakirov (1982); Saatov et al. (1993); Saatov et al. (1979a); Saatov et al. (1979b); Abubakirov (1980); Saatov et al. (1985)
S. pratensis	–	Zibareva et al. (unpublished)
S. procumbens	–	Zibareva (2000)
S. pseudotites	+	Zibareva et al. (unpublished); Zibareva (2000); Zibareva (1999); Meng et al. (2001)
S. pseudovelutina	+	Zibareva et al. (unpublished); Zibareva (2000)
S. pusilla	–/(+)	Zibareva (2000); Dinan et al. (unpublished)
S. pygmaea	+	Zibareva et al. (unpublished); Zibareva (1999); Bathori et al. (1995)
S. quadrifida	–	Zibareva et al. (unpublished)
S. quentensis	+	Saatov et al. (unpublished)
S. radicosa	+	Zibareva et al. (unpublished); Zibareva (2000); Meng et al. (2001)
S. regia	+	Dinan et al. (unpublished); Meng et al. (2001)
S. reichenbachii	+	Zibareva (2000)
S. repens	+	Revina et al. (1988); Zibareva (2000); Saatov et al. (1993)
S. requienii	–	Zibareva (2000)
S. roemeri	+	Zibareva et al. (unpublished); Zibareva (2000)
S. rubella	+	Zibareva (2000)
S. rupestris	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. ruprechtii	–	Zibareva et al. (unpublished)
S. saxifraga	+	Zibareva et al. (unpublished); Dinan et al. (unpublished)
S. scabriflora	+	Zibareva et al. (unpublished)
S. scabrifolia	+	Saatov et al. (1999); Saatov et al. (1993); Saatov et al. (1986a); Saatov et al. (1986c); Saatov et al. (1987a); Saatov et al. (1987b); Saatov et al. (1990a,b)
S. schafta	+	Dinan et al. (unpublished); Saatov et al. (1993)
S. schmuckeri	+	Zibareva (2000); Zibareva (1999)]
S. schumacheri	+	Bathori et al. (1995)
S. schwarzenbergeri	–	Dinan et al. (unpublished); Zibareva (2000)
S. secundiflora	+	Zibareva (1997); Zibareva and Yeryomina (1996)
S. sedoides	–	Zibareva et al. (unpublished)
S. sendtneri	+	Zibareva et al. (unpublished); Zibareva (2000); Bathori et al. (1995); Zibareva (1995)
S. sericea	+	Zibareva et al. (unpublished)
S. setisperma	–	Zibareva et al. (unpublished)
S. sieberi	+	Zibareva (2000)
S. sobolevskajae	+	Revina et al. (1988); Saatov et al. (1993); Raynor et al. (1989)
S. squamigera	+	Zibareva et al. (unpublished); Baltaev et al. (1987)
S. spergulifolia	+	Zibareva et al. (unpublished)
S. subconica	–	Zibareva et al. (unpublished)
S. succulenta	–	Zibareva (2000)
S. supina	+	Saatov et al. (unpublished); Ramazanov et al. (1997)
S. tatarica	+	Zibareva et al. (unpublished); Girault et al. (1990); Bathori et al. (1995); Zibareva (1997); Dzhukharova et al. (1991); Zibareva and Yeryomina (1996); Baltaev et al. (1987); Baltaev (1998)
S. thessalonica	+	Zibareva et al. (unpublished); Dinan et al. (unpublished); Zibareva et al. (1997b)
S. tomentella	+	Ramazanov et al. (1995); Ramazanov et al. (1996)

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Table 3 (*continued*)

Genera and species	Ecdysteroids	References
S. turgida	–	Revina et al. (1988)
S. turkestanica	+	Zibareva et al. (unpublished)
S. undulata	(+)/–	Dinan et al. (unpublished)/Zibareva et al. (unpublished)
S. uniflora	–	Dinan et al. (unpublished)
S. vallesia	–	Dinan et al. (unpublished); Zibareva (2000)
S. virginica	+	Dinan et al. (unpublished)
S. viridiflora	+	Saatov et al. (unpublished); Zibareva et al. (unpublished); Zibareva (1997); Zibareva (1995); Zibareva et al. (1997b); Ramazanov et al. (1997)
S. viscosa	–	Zibareva et al. (unpublished); Volodin et al. (unpublished)
S. vulgaris	–	Dinan et al. (unpublished); Zibareva (2000); Zibareva (1997)
S. waldsteinii	–	Zibareva (2000)
S. wallichiana	+	Saatov et al. (unpublished); Saatov et al. (1990b); Abubakirov (1984); Bathori et al. (1995); Abubakirov (1982); Saatov et al. (1993); Saatov et al. (1987a,b,c); Saatov et al. (1988)
S. zawadskii	–/+	Zibareva (2000)/Saatov et al. (unpublished); Dinan et al. (unpublished) Bathori et al. (1995)
Spergula (31 species)		
S. arvensis	+/–	Volodin et al. (2002)/Zibareva et al. (unpublished)
S. media	–	Zibareva et al. (unpublished)
S. sativa	+	Revina et al. (1988)
S. rubra	–	Volodin et al. (unpublished)
Spergularia (66 species)		
S. bocconei	–	Zibareva et al. (unpublished)
S. media	–	Dinan et al. (unpublished)
S. rubra	–	Dinan et al. (unpublished)
S. rupicola	–	Zibareva et al. (unpublished)
Stellaria (several hundred species)		
S. bungeana	–	Revina et al. (1988)
S. crassifolia	–	Volodin et al. (unpublished)
S. hebecalyx-		
S. holostea	–/+	Dinan et al. (unpublished); Revina et al. (1988)
S. media	–	Dinan et al. (unpublished)
S. nemorum	–/(+)	Volodin et al. (unpublished)
S. palustris	–/(+)	Volodin et al. (unpublished)
S. umbellata	–	Revina et al. (1988)
Stipulicida (2 species)		
Thurya (1 species)		
Thylacospermum (2 species)		
Uebelinia (6 species)		
Vaccaria (5 species)		
V. dioicum	+	Saatov et al. (unpublished)
V. diurnum	–	Zibareva et al. (unpublished)
V. elisabethae	–	Dinan et al. (unpublished)
V. hispanica	–	Zibareva et al. (unpublished)
V. pyrimidalis	–	Zibareva et al. (unpublished); Dinan et al. (unpublished)
Velezia (2 species)		
V. rigida	–	Zibareva et al. (unpublished)
V. quadridentata	–	Zibareva et al. (unpublished)
Viscaria (3 species)		
V alpina	–	Dinan et al. (unpublished)
V. vulgaris	–	Volodin et al. (unpublished)
Wilhelmsia (2 species)		

Genera in the Caryophyllaceae are according to Brummit (1992). The approximate number of species in each of the 66 genera was obtained from w3-TROPICOS.

S. thessalonica. The seeds of these species vary considerably in their ecdysteroid content, with those of *S. latifolia* (0.39 μg/g) and *S. pusilla* (4.4 μg/g) containing low levels, while the seeds of *S. bellidifolia* (642 μg/g), *S. colorata* (424 μg/g), *S. otites hungarica* (2688 μg/g), *S. saxifraga* (250 μg/g) and *S. thessalonica* (1013 μg/g) contain substantial amounts. In plants of *S. latifolia*, highest levels of RIA-positive material were found in roots (497–3283 μg/g), with much lower levels associated with leaves (2–65 μg/g), stem (34–107 μg/g),

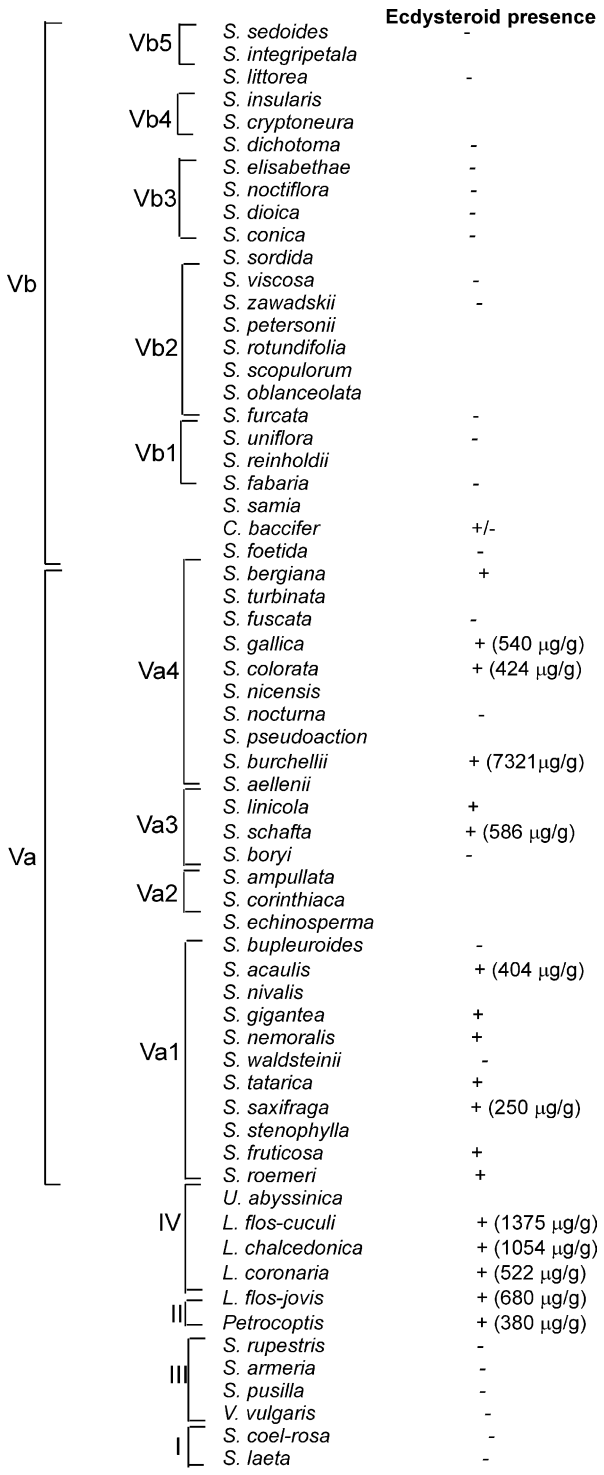

Fig. 1. Relationship between the occurrence of ecdysteroids in seeds and the taxonomic relationships within the *Sileneae* (as taken from Oxelman and Lidén, 1995). Ecdysteroid presence and absence are indicated by + and −, respectively. Where known, the ecdysteroid levels, as determined by radioimmunoassay with the DBL-1 antiserum, are included. Genera abbreviations: S; *Silene*, C; *Cucubalus*, U; *Uebelinia*, L; *Lychnis*, V; *Viscaria*.

crown (105–165 μg/g) and flowers (4–16 μg/g). Similarly, in *S. pusilla*, highest levels were associated with the roots (182–436 μg/g), with lower levels in the leaves (5–86 μg/g) and stems (13–119 μg/g). All plant parts of *S. bellidifolia* contain substantial amounts of RIA-positive material: leaves (858–5844 μg/g), roots (1662–3133 μg/g), crown (745–2201 μg/g) and stems (863 μg/g). In *S. colorata*, roots (335–7222 μg/g) and flowers (126–5297 μg/g) accumulate the highest levels of ecdysteroids with leaves (17–1059 μg/g) and stems (22–1778 μg/g) generally containing lower levels. It is apparent from the ranges of these concentrations that levels vary considerably from plant to plant for *S. colorata*. This variation is partially associated with the age of the plant, since ecdysteroid levels in stems and leaves are substantially higher in older plants (106 days) than younger plants (28 or 43 days). Young (22 days) plants of *S. otites hungarica* contain relatively low levels of ecdysteroids (54 μg/g in the leaves and 1957 μg/g in the roots), but levels are much higher in older (101 days) plants: roots (2780–4807 μg/g), leaves (2478–9419 μg/g), stems (4198–4352 μg/g) and crown (2541–3703 μg/g). The roots of *S. saxifraga* possess the highest ecdysteroid levels in this species (1175–2291 μg/g), with moderate levels in the leaves (398–784 μg/g) and stems (313–994 μg/g). The highest ecdysteroid levels are also found in roots of *S. thessalonica* (4864–12565 μg/g), but levels in leaves (526–3239 μg/g) and crowns (637–3004 μg/g) are also high.

3. Future directions

The present study provides an outline examination of the Caryophyllaceae for phytoecdysteroids. It is apparent that the combination of ecdysteroid analysis with molecular phylogenetic analysis is the most promising way forward, extending the analysis both within and beyond the tribe *Sileneae*. On the basis of the current study we shall be able to carry out a targeted assessment of informative species and genera in order to clarify more extensively the taxonomic distribution of ecdysteroids in this Family. We focussed on analysis of seeds for reasons of comparability; because of the known variations in ecdysteroid content with plant part and developmental stage. However, it is clear that some species for which the seeds are ecdysteroid-negative accumulate ecdysteroids after germination. Thus, it will be important to analyse both seeds and vegetative material for ecdysteroid content. Further, the identities of the major and minor ecdysteroids vary between species and possibly also between different parts of the plant and at different stages of development. We have initiated studies on this aspect in order to determine whether ecdysteroid profiles may have chemotaxonomic significance. Ecdysteroid levels and profiles may also vary in relation to geographical location and ecological environment, and

it will be important to determine to what extent such variations are genetically regulated. This is an aspect which has so far received only limited attention (Volodin et al., 2002), and a consistent and coherent examination of this with respect to one family, i.e. the Caryophyllaceae, would be highly informative.

4. Experimental

4.1. Phytoecdysteroid database

The literature from 1966 (when the first reports of phytoecdysteroids appeared) to the present was extensively surveyed and a database of all the plant species which have been examined for the presence or absence of ecdysteroids compiled. The data for previously published species in the Caryophyllaceae reported in this article have been extracted from that database.

4.2. Plant material

Seeds were purchased either from Chiltern Seeds Ltd. (Ulverston, Cumbria, U.K.), B&T World Seeds Sarl (Olonzac, France) or Thompson & Morgan (Ipswich, Suffolk, U.K.), or were generously donated by Ness Botanical Gardens, Wirral, U.K. *S. alba*, *S. armeria*, *S. bellidifolia*, *S. coeli-rosa*, *S. colorata*, *S. latifolia*, *S. multiflora*, *S. otites hungarica*, *S. pendula*, *S. pusilla*, *S. saxifraga*, *S. thessalonica*, *S. vulgaris* and *S. zawadski* plants were grown in the glass-houses of the University of Exeter. After harvesting at known ages after sowing, individual plant heights were measured and the plants were then dissected into their constituent parts (leaves, stem, root, crown and flowers), which were individually weighed and freeze-dried to constant dry weight.

4.3. Preparation of plant material and extraction

Plant material was extracted according to a previously described method (Dinan, 1995). Briefly, seeds and other plant parts were ground in a pestle and mortar and samples (25 mg) were extracted with MeOH (3×1 ml) at 55 °C for 3 h. The combined extracts were diluted with water (1.3 ml) and partitioned against hexane (2 × 2 ml). The aqueous methanol phase was used for RIA, bioassays, HPLC and TLC. Other plant parts were freeze-dried for 4 days and then processed as described for the seeds.

4.4. Bioassays for ecdysteroid agonist and antagonist activities

Bioassays, using the microplate-based *Drosophila melanogaster* B_{II} cell line, were performed as described previously (Clément et al., 1993). The concentration of 20-hydroxyecdysone used in the antagonist assay was 5×10^{-8} M.

4.5. Radioimmunoassays

Radioimmunoassays using ecdysteroid-specific antisera were carried out using a previously described method (Dinan, 1992). The antisera used were DBL-1 and Black (generously donated by Professor Jan Koolman, Univesität Marburg, F.R.G.). The cross-reactivities of these antisera for a number of ecdysteroids have been determined (Dinan, 1995).

5. Uncited References

Zibareva and Yeryomina, 2002 is not cited.

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