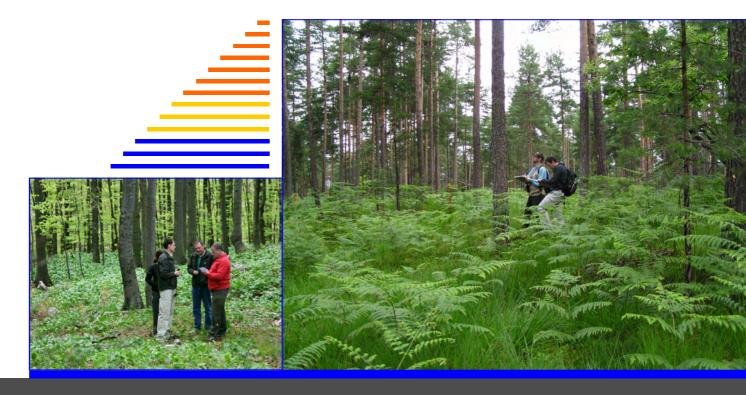


Ministrstvo za kmetijstvo, gozdarstvo in prehrano, Dunajska 58, Ljubljana



Ministrstvo za okolje in prostor Dunajska 48, Ljubljana

#### Gozdarski Inštitut Slovenije Večna pot 2



# Poročilo o projektni nalogi FutMon Life+

1. mejnik

Ljubljana, 29. junij 2009



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### Poročilo o projektni nalogi FutMon Life+

Mejnik 1, junij 2009

Naročnik : MKGP, MOP, EU

Ljubljana, 29. junij 2009

#### Kazalo

1	Р	olletno poročilo o izvajanju posameznih akcij	3
	1.1 repi	FutMon Life+ aktivnost L1 in L2a - Izdelava mreže za veliko prostorski rezentativni monitoring (2009-2010)	4
	1.2	Intenzivni monitoring (IM1 FutMon LIFE+)	6
	1.3	Vitalnost drevja (D1 FutMon LIFE+; demonstracijska naloga)	.10
	1.4 dem	Kroženje hranil in kritični vnosi v gozdne ekosisteme (D2 FutMon LIFE+; nonstracijska naloga)	.13
		Kroženje vode v gozdnih ekosistemih (D3 FutMon LIFE+; demonstracijska oga)	.15
		Kakovost, strokovna presoja in ocena spremljanja depozitov (C1-DEP-22 Mon LIFE+)	.18
	1.7	Upravljanje projekta (M7 FutMon LIFE+)	.22
	1.8	M8 (FutMon LIFE+)	.24
2	Р	oročilo o krožnih analizah (foliarne)	.25
3	Р	oročilo o posodobitvi navodil za izvajanje aktivnosti IM1	.26
4 (ł		oročilo o aktivnostih na področju QA //bfw.ac.at/rz/bfwcms.web?dok=7887)	.27
5	Р	oročilo o fenološki delavnici v Sloveniji	.28
6	Р	oročilo o delavnicah za skrbnike ploskev IM1	.33
7	Р	oročilo o harmonizaciji inventurnih sistemov ICP in NFI (L1)	.36
	P	riloge 1-8	

#### 1 Polletno poročilo o izvajanju posameznih akcij

Na osnovi pogodbe o sofinanciranju projekta LIFE07ENV/D/000218, »Further development and implementation of an EU-level forest monitoring system – FutMon«, (MOP 2511-08-600085 in MKGP 2311-09-000083) ter EU smo na GIS pripravili poročilo o delu za obdobje od 1. januarja do 30. junija 2009. Poročilo obsega opis dejavnosti za akcije v prvem poletju poteka naloge in so določene v projektni prijavi iz 4. alineje 1. člena:

- polletno poročilo o izvajanju akcij L1, L2, D1, IM1, D2, D3, M7, M8, C1depo,
- poročilo o krožnih analizah (foliarne),
- poročilo o posodobitvi navodil za izvajanje aktivnosti IM1,
- poročilo o aktivnostih na področju QA,
- poročilo o fenološki delavnici v Sloveniji,
- poročilo o delavnicah za skrbnike ploskev IM1 in
- poročilo o harmonizaciji inventurnih sistemov ICP in NFI (L1).

V prvi polovici l. 2009 smo uspeli dokončati vse formalnosti glede podpisa pogodbe z koordinatorjem projektne naloge z inštitutom« Johann Heinrich von Thünen Institut (vTI)«, Institut für Weltforstwirtschaft, iz Hamburga, Nemčija. V prvih šestih mesecih so potekale številne razprave glede metodologije dela, ki predstavlja del ciljev projektne (oblikovanje novih navodil za delo po posameznih že obstoječih ter tudi novih aktivnosti, meritev). Vzporedno z delom potekajo številne delavnice, prenosi znanja tako med odgovornimi raziskovalci za posamezna področja kot tudi v posameznih državah (skrbniki ploskev in naprav). Nakupi opreme so zaradi oblikovanja metodologij delno zaostajajo, vendar ne več kot mesec ali dva, tako, da pričakujemo da se to ne bo pokazalo na ravni leta (prvega leta raziskav). Zaradi narave projekta, katerega cilj je uvajanje novih indikatorjev spremljanja stanja gozdov in preizkušanja novih pristopov k vse-evropskemu monitoringu gozdov se morajo izvajajo določene terminske kot tudi finančne korekcije, vendar vse znotraj načrtovanih aktivnosti in v okviru »Splošnih določb« finančnega okoljskega mehanizma Life+.

## 1.1 FutMon Life+ aktivnost L1 in L2a - Izdelava mreže za veliko prostorski reprezentativni monitoring (2009-2010)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1.1. 2009 -31.12.2010 Vodja: M. Kovač Sodelavci GIS: G. Kušar, A. Japelj, M. Skudnik, S. Fajon, A. Ferreira, J. Žlogar, D. Jurc Ostali sodelavci:

#### Namen in cilj raziskave:

Namen tega sklopa projekta je razviti metodologijo za združitev nacionalnih gozdnih inventur z inventuro ICP-Forest in izdelati premostitvene funkcij. V okviru faze L1 se bo v okviru mednarodnega sodelovanja izdelal evropska mreža. V okviru L2 modula bo teklo testiranje kazalcev obveznih po ICP Forest navodilih in izračun funkcij. V letu 2009 so cilji naslednji:

- Izdelava evropske mreže, zasnova modela za združitev obeh monitoringov in testiranje na ploskvah 16x16 km;
- Testiranje novih kazalcev za pojasnjevanje stanja krošenj na mreži 16x16 km.

#### Načrt aktivnosti:

- izdelava »mreže«, za evropsko mrežo,
- priprava metodologije za združitev monitoringov,
- dopolnitev obstoječega nacionalnega priročnika za snemanje na ploskvah s potrebami projekta,
- organizacija internega seminarja in izvedba snemanj,
- vnos podatkov v podatkovno bazo,
- obdelava podatkov.

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

V prvi polovici leta 2009 so bile za modul L1 opravljene naslednje aktivnosti:

• Preverjena je bila gostota vzorčne mreže in razporeditev vzorčnih ploskev ter korektnost izbire ploskev (gozd, negozd). Vzorčna mreža 16x16 km ostaja enaka kot doslej in ustreza evropskim zahtevam, za periodično (5-10 let) zanesljivejšo velikoprostorsko inventuro pa obstaja gostejša (4x4 km) vzorčna mreža.

• V teku je preverjanje statistične moči preizkusa (*statistical* power) glede na gostoto vzorčne mreže za izbrane parametre monitoringa. Statistična moč preizkusa se nanaša na število potrebnih ponovitev (gostoto vzorčnih ploskev), da se statistično zazna razlika v ocenah sprememb parametrov med leti z gotovo (dano) zanesljivostjo. Rezultat takšne analize omogoča, koliko vzorčnih ploskev je v naslednjem letu potrebno posneti, da se zazna razlika v ocenah parametra za 3 % glede na prejšnje leto. Za vzorčno mrežo 4x4 km za parameter lesne zaloge dreves znaša moč preizkusa približno 200 ploskev namesto 700. Za vzorčno mrežo 16x16 izračunavanje statistične moči preizkusa še poteka. Preverjeno je že, da Slovenija za korekten integralen monitoring gozdov in gozdnih ekosistemov ter spremljanje stanja in razvoja potrebuje vzorčno mrežo 4x4 km (periodična snemanja), za nadzor kritičnih sprememb pa zadostujejo vsakoletna snemanja na redkejši 16x16 km vzorčna mreža.

• Razvite so bile premostitvene funkcije za povezavo podatkov preteklih snemanj z novim snemanji (kontinuiteta časovnih vrst) za vse parametre razen za poškodbe drevja zaradi biotskih vzrokov. Za slednje priprave premostitvenih funkcij še potekajo.

V okviru L2a modula poteka testiranje protokolov, dodelava navodil oz. priročnika in priprava terenskih navodil. V teku so tudi priprave za izvedbo internega seminarja (prvi teden julija) in izvedbe snemanj (julij, avgust).

#### 1.2 Intenzivni monitoring (IM1 FutMon LIFE+)

#### Naročnik: EU DG. ENV., MKGP, MOP

*Šifra:* LIFE07 ENV/D/000218

Trajanje naloge: 1. 1. 2009 - 31. 12. 2010

Vodja: P. Simončič

Sodelavci GIS: M. Rupel , M. Ferlan, M. Kovač, M. Skudnik, A. Japelj, T. Levanič, R. Krajnc, L. Kutnar, P. Simončič, M. Urbančič, A. Verlič,

D. Žlindra, M. Špenko, I. Truden, N. Filipič, , M. Huibers, D. Jurc, N. Ogris, M. Jurc, U. Vilhar

Ostali sodelavci: F. Batič in K. Eler (BF odd. agr.), skrbniki ploskev (20 + 2; ZGS), B. Zupančič, A. Planinšek (ARSO) , T. Vovk

#### Namen in cilj raziskave:

Namen naloge je izbor ploskev, izbor opazovanj in meritev in spremljanje znakov in lastnosti intenzivnega monitoring (IM) gozdov. Za ta namen se izvajajo dela na t.i. osnovnih ploskvah IM v obdobju 2009 – 2010. Vrednotenje rezultatov podatkov iz vseh IM ploskev v projektu v državah članicah EU bo potekalo koordinirano v nalogah oz. akcijah FutMon LIFE+ projekta in sicer v akciji C1 – trees 30 (NWD), C1-Fol-10 (FI) in v akciji A1-1 (DE).

Nacionalne rezultate bomo vrednotili delno v nalogi IM1 in v ustreznih C1 aktivnostih.

**Načrt aktivnosti:**1. Letni popis stanja krošenj v skladu s 2 poglavjem navodil za izvajanje IM »ICP Forest« (<u>http://www.icpforests</u>. org/Manual.htm) skupaj z oceno mortalitete in sečnjo (sanitarna...) drevja;

2. Ocena rasti drevja (1x v dveletnem obdobju) v skladu s 5. poglavjem navodil za izvajanje IM »ICP Forest«;

3. Izvedba ocene preskrbljenosti drevja hranili (1x) skladno s 4. poglavjem navodil »ICP Forest«;

4. Ocena pritalne vegetacije (1x) skladno s 4. poglavjem navodil »ICP Forest«;

5. Kontinuirano spremljanje depozicije na 7 ploskvah IM, skladno s 6. poglavjem navodil »ICP Forest«;

6. Spremljanje kakovosti zraka v skladu z 10a. poglavjem navodil »ICP Forest«;

7. Tla – če še ni bilo izvedeno se izvedele obvezne analize iz 3. poglavja navodil »ICP Forest« (za Slo 1-2 ploskvi in nekaj parametrov, ki jih še nismo določili);

8. Metrologija – v skladu z 7. poglavjem navodil »ICP Forest« (padavine, T in vlaga zraka, globalno obsevanje, hitrost in smer vetra);

Podatki se posredujejo EC preko koordinatorja projekta. V povezavi z aktivnostmi IM1 bodo morale biti izvedene tudi ustrezne krožne analize za zagotavljanje ustrezne kvalitete dela laboratorijev in sodelavcev na terenu.

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

V Sloveniji potekajo aktivnosti intenzivnega monitoringa (IM1) na desetih ploskvah. V prejšnjih letih so aktivnosti potekale na 11 ploskvah, vendar je bilo manjše število aktivnosti, ki se izvajajo na vseh desetih ploskvah.

Ploskve, ki so aktivne v letošnjem letu so: Pokljuka, Fondek (Trnovski gozd), Gropajski bori (Sežana), Brdo, Borovec (pri Kočevski Reki), Kladje (Pohorje), Lontovž (Kum), Gorica (Draga), Krakovski gozd in Murska Šuma (prikaz na spletni strani GIS: <u>http://www.gozdis.si/monitoring/raven\_2.htm</u>).

Na 7 ploskvah poteka stalno spremljanje depozita, kar je bilo dogovorjeno z neposrednimi pogajanji med vodilnim partnerjem, EU in GIS. Ker se je predhodno depozit spremljalo le na 5 ploskvah (na ploskvah Fondek, Brdo, Borovec, Lontovž in Murska Šuma), je bilo potrebno dodatno opremiti še dve ploskvi. Na novo smo opremili ploskev Gropajski bori pri Sežani in Kladje II na Pohorju. Na ploskvi Pohorje nam je postavitev vzorčevalnikov za spremljanje depozitov otežila zima, ki je bila precej snežena in zato tudi dolga. Tako smo lahko ploskev za spremljanje depozita opremili šele v maju, od takrat pa spremljanje poteka nemoteno. Na ploskvi se poleg depozita spremlja tudi tok po deblu z ustreznimi tobogani. Na novo je bilo potrebno izvesti individualne tečaje za skrbnika na ploskvi Gropajski bori (v začetku leta 2009) in tudi za skrbnika na ploskvi Pohorje (v maju 2009), ki že lahko samostojno opravljajo svoje delo.

Nekaj težav nam je povzročilo pomanjkanje opreme za spremljanje depozita, vendar smo težavo sedaj že rešili (dobava novih 5 litrskih posod za žlebiče).

Vzorce z IM1 ploskev se z lokalnih mest ZGS prenesejo in zbirajo v Laboratoriju za gozdno ekologijo (LGE) Gozdarskega inštituta Slovenije, kjer poteka reden postopek analiz obveznih parametrov za FutMon (oz. ICP Forest, Expert Panel on Deposition Measurements; http://www.icp-forests.org/EPdepo.htm) projekt. Analize potekajo nemoteno razen analiza kationov, kjer imamo zaradi pomanjkanjem sredstev na GIS za materialne stroške težave z nakupom rezervnih delov za IC (npr.: kolone za določanje kationov). Rezultati se po logičnih kontrolah shranjujejo v podatkovno bazo LGE/GIS, na to v skupno bazo GIS za potrebe monitoringa gozdov (druga polovica l. 2009), iz katere bodo s pomočjo aplikacij pripravljena poročila za oblikovanje skupne EU FutMon podatkovne baze (pošiljanje koordinatorju projektne naloge vTI v Hamburg).

**Kakovost zraka** se spremlja v skladu z navodili iz 10a poglavja ICP Forest navodil. Kakovost se spremlja na vseh 10 ploskvah in sicer s pasivnimi vzorčevalniki. Vsi skrbniki so bili poučeni o izvajanju meritev, o menjavi vzorčevalnikov, o nujni uporabi rokavic,.... Kakovost zraka se na večini ploskev spremlja od 25. marca naprej. Izjema so tri ploskve in sicer ploskev Gorica, kjer se je začelo spremljati z 8.4., ploskev Pohorje, kjer se je začelo z 20.5. in ploskev Pokljuka, kjer se je začelo spremljati s 3.6., čemur je vzrok dolga zima in dostopnost ploskev. Ploskev Pohorje in Pokljuka ležita na višji nadmorski višini, kjer se tudi vegetacijska doba začne kasneje. Vzorčevalniki so izpostavljeni 14 dni, nato jih skrbniki zamenjajo z novimi (neizpostavljenimi), izpostavljene pa pošljejo v Laboratorij za gozdno ekologijo Gozdarskega inštituta Slovenije. Tu se dozimetri v kontrolirani atmosferi odprejo in pripravijo na analizo, tako da naredimo vodno ekstrakcijo filtrov skupaj s centrifugiranjem. Sledi analiza ekstrakta z ionskim kromatografom (Metrohm).

**Meteorologija**: Dobavitelji posameznih sklopov meteoroloških postaj so bili izbrani do konca aprila 2009. Izbira najugodnejšega ponudnika je potekala na podlagi zbranih predračunov. Vsa oprema bo nabavljena do konca junija 2009. Na dve izbrani ploskvi (Brdo in Gropajski bori) sta bili začasno montirani dve odsluženi meteorološki postaji proizvajalca Davis Inc. Postaji sta pričeli s snemanjem podatkov v sredini maja 2009. Na vse ostale ploskve se bo do konca nabavnega roka (junij 2009) postavilo 2-4m visoka stojala in betonskimi temelji. Vse skupaj bo omogočalo lažjo in zanesljivejšo montažo meteoroloških postaj in tudi kasnejšo kontrolo in servisiranje senzorjev.

V poletnih mesecih bodo potekal **popis stanja krošenj skupaj z** ugotavljanjem **povzročiteljev poškodb sestojev** (opis simptomov, določitev povzročitelja in količinska ocena poškodbe), **popis pritalne vegetacije, odvzem vzorcev listja drevja**, v jeseni odvzem vzorcev iglic drevja, v obdobju mirovanja vegetacije (2009/2010) pa bodo izvedene meritve za **oceno rasti drevja**. Na potekajo tudi redna fenološka opazovanja drevja. Na eni ploskvi Gropajski bori bodo v jeseni odvzeti vzorci **gozdnih tal**. partly carry out , optional in programe\* FutMon Life+

not in vFutMonLife+

\*: if execution is reasonable due to the content (national founds)

SFI, P. Simoncic -1 D1 2009-\_010 IM1 2009- 2010 D2 2009-2010 D3 2009-2010 Številka nutrients ground. air qualit. LAI litterfall nutrient · activity IM1 pilot action crown soils growth deposit meteo Daljin crown phenology growth soil solut folia soil pF for so stand T soil ploskve vegetat. znavanj extend.\* extend. ground. analysius moisture deposits vegetat. inten. 1-2x/year Pogostost yearly 2 years: 10 years 5 years: 5 years: cont cont cont. cont. month. cont. 1-2x/ year yearly cont cont. 1x / project cont. cont. 2009 2009/10 2009 loskev 22\_1 Pokljuka ~ 22\_2 D1, D3 Trnovski \* 4 ~ ~ ~ ~ ozd 22\_3 Sežana D1, D3 1 1 1 1 1 4 1 1 1 1 1  $\checkmark$ 1 1  $\checkmark$ 1 ~ 1 1 ~ - -- -- -- -- $\checkmark$ 22\_4 Brdo D1, D2, D3 ~ ~ ~ ~ 4 1 ~ ~ ~ ~ ~ ~ ~  $\checkmark$ ~ ~  $\checkmark$ ~ ~ ~ ~ ~ ~ ~ 22\_5 Borovec D1, D2, D3 1 1 1 1 1 1 ✓ ~  $\sqrt{\sqrt{}}$ ~ 1 1 ~ ✓ ✓ ~ 1 ~  $\checkmark$ ✓  $\checkmark$  $\checkmark$ ~ 1  $\checkmark$ ≁ ≁ 22\_6 Pohorie D1, D3 1 ~ 4 1 1 1 1 ~ 1 ~ ¥ ? 7 <del>ški Kozj</del>i ++ 22\_8 Zasavje 1 D1, D3 ~ ~ ~~~ ~ 1 1 ~ 1 4 .... .... - -... - -. . - -... - -. . - -- -- -- -- -. . 22\_9 . oški potok ~ ~ 1 -~ 4 22\_10 Krakovski 4 - -~ ~ ~ zd 22\_11 Murska ~ D1, D3 ~ ~ 11 ~ ~ ~ 4 1 ~ ~ ~ ... - -~  $\checkmark$  $\checkmark$ ~ -~ 1 ~ ~ Šuma at all plots Legend: \*: C.C.& forest health in project FutMon Life+ 1 plot is excluded from FutMon Life+ + no activity in FutMon Life+ is going on but not in offcial programe\* FutMon Life+

#### 1.3 Vitalnost drevja (D1 FutMon LIFE+; demonstracijska naloga)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1. 1. 2009 - 31. 12. 2010 Vodja: M. Kovač Sodelavci GIS: M. Skudnik, D. Jurc, N. Ogris, M. Jurc, M. Rupel , T. Levanič, R. Krajnc, P. Simončič, M. Urbančič, D. Žlindra, M. Špenko Ostali sodelavci: ZGS (22 sodelavcev), ARSO, BF odd. agr., T. Vovk

#### Namen in cilj raziskave:

Namen demonstracijske naloge D1 je priprava integralnih indikatorjev za oceno vitalnosti drevja in priprava predloga operativnega spremljanja vitalnosti drevja v okviru evropskega monitoringa gozdov, ki bo temeljilo na sodobnih znanstvenih dognanjih. V okviru demonstracijske naloge D1 »Vitalnost drevja« je predviden integralen pristop k ocenjevanju vitalnosti, stanja drevja na ploskvah IM1, kar naj bi bilo različno od programov spremljanja stanja gozdov v preteklosti (npr. Forest Focus 2003-2006, snemanje osutosti in porumenelosti), poleg tega naj bi pridobili dodatne podatke o procesih kot so alokacija ogljika, rastna dinamika, pomlajevanje obravnavanih sestojev, odziv drevja na različne strese (npr. časovne odstopanja od povprečij pojavljanja fenoloških faz kot indikator stresa).

#### Načrt aktivnosti:

Aktivnosti naloge D1 se bodo v Sloveniji izvajale na 6 ploskvah IM. Metode določene v ICP Forest navodilih poglavja 2 kot so struktura sestoja, sečnja, mortaliteta, osutost, cvetenje, morfologija krošenj, ocena vzrokov poškodb. Dodatno se bodo izvajale meritve rasti drevja po ICP navodilih, poglavje 5 (»Forest Growth«), kontinuirane meritve rasti z ročnimi dendrometri D1; meritve opada opisane v ICP navodilih, poglavju 11 (»litterfall«), posebna pozornost bo posvečena listju/iglicam in plodovom. Izvedena bodo natančnejša fenološka opazovanja kjer bomo na izbranem objektu izvedli poskusna snemanja z digitalnim fotoaparatom. Narejene bodo meritve *Leaf Area Index-a* (indeks listne površine) (v nadaljevanju LAI) z napravo LI-COR 2000 oz. primerjalno z drugimi metodami (sodelovanje z BF, Odd. za gozd.).

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

Poteka priprava predlogov indikatorjev za operativno snemanje vitalnosti drevja. Na šestih ploskvah IM so se pričele izvajati metode določene v ICP Forest navodilih poglavja 2. Obstoječi nacionalni priročnik za snemanje na ploskvah se dopolnjuje s poglavjem o ugotavljanjem povzročiteljev poškodb na drevesu. Ocenjevanje osutosti krošnje se bo dopolnilo z določitvijo znanih povzročiteljev osutosti. Tako se bo v popisu v letu 2009 poleg ostalih parametrov ocenila tudi lokacija poškodbe na drevesu, opisali se bodo opaženi simptomi poškodbe in določila se bo kategorija povzročitelja poškodbe ter povzročitelj.

Dodatno so se za namene izvajanja meritev rasti drevja po ICP navodilih, poglavje 5 (*»Forest Growth«*) na izbranih drevesih namestili dendrometri na podlagi katerih se izvajajo neprekinjene, kontinuirane meritve debelinske rasti drevja.

**Fenološki popisi** se v Sloveniji izvajajo na vseh D1 ploskvah. Popisi se izvajajo na ravni drevesa. Na ploskvah je bilo v letu 2004 izbranih 20 dreves, ki so se večinoma ohranila od predhodnih opazovanj. Po eno drevo je zaradi različnih vzrokov (strela, snegolom) odpadlo na ploskvi Pokljuka, Pohorje in Gropajski bori. Dreves nismo nadomeščali z novimi, saj je na ploskev potrebno opazovati od 10 do 20 dreves.

Popisovalci fenoloških faz so ostali večinoma isti kot v preteklih letih, edino na ploskvi Gropajski bori v Sežani je eden izmed skrbnikov prenehal z delom, tako da ga je nadomestil nov skrbnik. Skrbniki s ploskev Gropajski bori in se je udeležil tudi fenološke delavnice v Lipici in je sodeloval pri terenskem interkalibracijskem tečaju na črnem boru in bukvi.

V letošnjem letu smo pred začetkom vegetacijske dobe opravili pogovor z vsemi skrbniki ploskev, kjer smo jim predstavili novosti glede letošnjega fenoloških popisov. Opozorili so bili, da je med kritičnimi fazami potrebno obiskati ploskve vsaj enkrat na teden, bolje pa, če se snemanje opravi večkrat tedensko ali tudi dnevno. Izven kritičnih faz je število opazovanj enako kot preteklosti (na 2 – 4 tedne). Poleg tega so bili informirani glede pošiljanja obrazcev, ki je vsaj enkrat na mesec. V začetku aprila so večinoma začeli z opazovanjem (na ploskvi Kum v sredini aprila, na ploskvah Pokljuka in Pohorje pa v maju – vse tri ploskve ležijo na višji nadmorski višini). Skrbniki so upoštevali nova navodila, tako da so nekateri opravljali popise tudi večkrat tedensko, obrazce pa tudi redno pošiljajo. Tudi vnos v podatkovno bazo za fenološke popise je reden.

Od 5.- 7. maja 2009 je v Lipici potekala mednarodna fenološka delavnica FutMon (Combined Field Course on Phenology and LAI), kjer so bile sprejete novosti, ki so združene v dodatku k priročniku na internetni strani projektne naloge FutMon (http://futmon.org/).

Glavna novost pri fenoloških aktivnosti je uporaba kamer, vsaka država podpisnica/partner naj bi v teh dveh letih vzpostavila snemanje fenologije na vsaj eni ploskvi s pomočjo kamere/r.



Fotografija 1: Demonstracija delovanja fenologije s pomočjo kamere na fenološka delavnica FutMon v mesecu maju 2009 v Sloveniji (Foto: M. Čater)

V juniju (11. junij 2009) je v Lipici in na ploskvi Gropajski bori potekala delavnica za skrbnike ploskev (Gozdu škodljivi biotski dejavniki). Na terenskem delu so bile predstavljene (T. Brišnik) novosti pri izvajanju fenoloških popisov. Poleg snemanja s kamerami je bilo opozorjeno na spremenjeno točkovanje intenzivnosti fenološkega pojava (odganjanje, jesensko rumenjenje ter odpadanje listov) ter na možnost količinskega ocenjevanje cvetenja. Skrbnikom je bil posredovan nekoliko prirejen obrazec za popise. Popisovalcem - skrbnikom ploskev so bile predstavljene tudi najpogostejše napake pri njihovem delu v zadnjih letih.

V letu 2009 se morajo na vseh ploskvah za D1 (6 ploskev) postaviti lovilci za spremljanje opada. Material za postavitev lovilcev opada je nabavljen, postavitev lovilcev se predvideva v juliju oz avgusta in pred jesenskim odpadanjem listja.

## 1.4 Kroženje hranil in kritični vnosi v gozdne ekosisteme (D2 FutMon LIFE+; demonstracijska naloga)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1. 1. 2009 - 31. 12. 2010 Vodja: P. Simončič Sodelavci GIS: M. Ferlan, T. Brišnik, M. Rupel , M. Kobal, M. Čater, L. Kutnar, A. Verlič, U. Vilhar, D. Žlindra, M. Špenko Ostali sodelavci: K. Eler (BF odd. agr.), skrbniki ploskev (ZGS), ARSO, T. Vovk

#### Namen in cilj raziskave:

Namen demonstracijske naloge D2, da se na omejenem številu ploskev IM1 (za SLO 3 objekti, predlog) preveri in razvije tiste metode spremljanja stanja gozdov, ki so potrebne za izboljšanje ocene kroženja hranil za izbrane gozdne ekosisteme oz. ploskve ter ocene kritičnih obremenitev teh sestojev z izbranimi onesnažili; v tem primeru gre za oceno rizika za gozdne ekosisteme glede na vnos (potencialni in izmerjen) dušika, ozona (O<sub>3</sub>), POP, kovin in povezovanja s scenariji podnebnih sprememb. Zbiranje podatkov bo potekalo v nalogi D2 in evaluacija rezultatov pa je vključena v aktivnosti naloge C1-Fol-10 (Fi), projekta FutMon Life+.

#### Načrt aktivnosti:

Naloga bo potekala na ploskvah IM kjer se izvajajo aktivnosti naloge IM1 in dodatno: 1. spremljanje opada na 2(3) ploskvah IM skladno z 11. poglavjem navodil »ICP Forest«(http://www.icpforests.org/Manual.htm);

2. spremljanje talne raztopine na 2(3) ploskvah IM skladno s 3. poglavjem navodil »ICP Forest«;

3. intenzivnejše izvajanje spremljanje preskrbljenosti drevja hranili skladno z navodili, ki jih bo pripravljena v

akciji C1-Fol-10 (Fi) v začetku l. 2009 in bodo uporabljena v drugi polovici l. 2009;

4. ocena vsebnosti hranil v pritalni vegetaciji na osnovi novih navodil, ki jih bo pripravili strokovnjaki v akciji C1- Fol-10 (Fi), izvedba je načrtovana za drugo polovico l. 2009.

Za izvedbo naloge je potrebno v l. 2009 postaviti vzorčevalnike opada (litterbags), izvajati vzorčenja opada glede na

navodila (11. poglavje navodil »ICP Forest«). Izvesti je potrebno ustrezne izračune za kritične vnose in ocene kroženja hranil na izbranih ploskvah (v Sloveniji na 2 oz. 3 od 10). Rezultati naloge bo koordinator projekta (vTI, Hamburg) posredoval EC, DG ENV..

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

V letu 2009 se morajo na vseh ploskvah za D2 (2 ploskvi) postaviti lovilci za spremljanje opada, kot tudi v predhodni akciji D1. Material za postavitev lovilcev opada je nabavljen, postavitev lovilcev se predvideva v juliju oz avgusta in pred jesenskim odpadanjem listja. Poleg opada se bo v poletnih mesecih ob popisu vegetacije vzorčila tudi pritalna vegetacija, glede načina vzorčenja in metode pa so v prvi polovici leta potekale intenzivne razprave. Dodatno se bodo v okviru akcije D2 izvedle še intenzivnejše analize mineralne prehrane drevja. Kot v akcijah D1 in D3 so je predvidena uporaba rezultatov meritev LAI, indeksa listne površine.

Spremljanje **talne raztopine** se v Sloveniji izvaja na dveh ploskvah in sicer v sestoju rdečega bora na Brdu pri Kranju in v bukovem sestoju na Borovcu pri Kočevski Reki. Vzorčenje poteka vsakih 14 dni. Talno raztopino vzorčimo z lizimetri v obliki manjšega valja iz poroznega materiala. Na slovenskih ploskvah se uporablja lizimetre s podtlakom 0,6 bara, ki so jih pripravili na nizozemskem inštitutu Alterra oz. z lizimetri P80. Lizimetri so vgrajeni na treh lokacijah v blažilnem območju ploskve. Na vsaki lokaciji so trije lizimetri vgrajeni tik pod organskim horizontom, trije na globini 20 cm in trije na globini 40 cm pod površino tal. S plastičnimi cevkami so povezani s steklenicami (0,5 l), v katerih se pred vsakim vzorčenjem vzpostavi podtlak 0,6 bara.

Z lizimetri s podtlakom vzorčimo tisto talno raztopino, ki ne steče prosto skozi tla, temveč zastaja v porah tal, pri čemer potekajo reakcije med tlemi in raztopino.

## 1.5 Kroženje vode v gozdnih ekosistemih (D3 FutMon LIFE+; demonstracijska naloga)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1. 1. 2009 - 31. 12. 2010 Vodja: U. Vilhar Sodelavci GIS: M. Rupel, T. Brišnik, Š. Fajon, M. Ferlan, M. Čater, M. Kobal, M. Urbančič, P. Simončič, A. Verlič, N. Filipič, D. Žlindra Ostali sodelavci: K. Eler (BF odd. agr.), J. Diaci (BF, odd. za gozd...), skrbniki ploskev (ZGS), ARSO

#### Namen in cilj raziskave:

Namen demonstracijske naloge D2, da se na omejenem številu ploskev IM1 (za SLO smo predlagali 6 ploskev) razvije in vpelje vse potrebno za modeliranje kroženja vode in izračun vodne bilance. Rezultati naloge bodo posredovani akciji C1 Met – 29. Preskrba z vodo (preko gozdnih tal) je eden izmed ključnih dejavnikov ki vpliva na stanje, vitalnost drevja in posledično stanje sestoja. V preteklosti se na ploskvah IM ni izvajalo meritev za izračun vodne bilance.

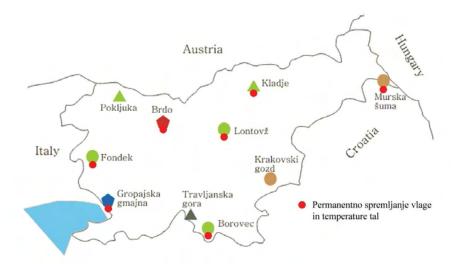
Na osnovi meritev bodo testirani modeli za izračun / oceno vodne bilance; tok vode ocenjen z modeli bo služil tudi oceno toka hranil v gozdnih tleh (navezava na D2). Potrebno bo določiti razpoložljivost vode in indikatorje stresa suše za obravnavane ploskve IM, tudi v povezavi z rezultati naloge D1 (rast drevja, stanje krošenj, pojav bolezni idr).,

#### Načrt aktivnosti:

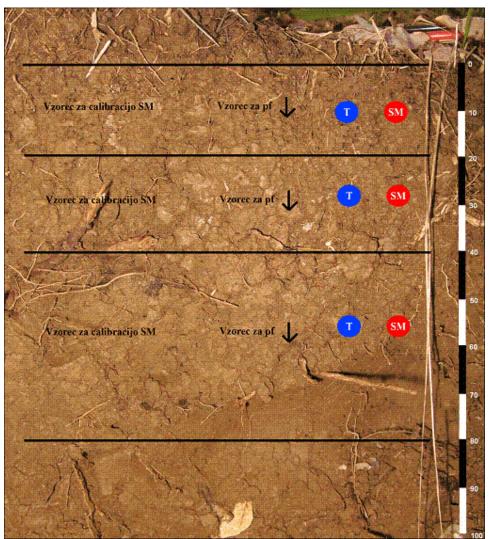
V okviru naloge se bo na 6 ploskvah IM izvedlo še meritve volumske vlage tal (TDR meritve), matrični potencial, določitev pF krivulje za tla, padavine na ravni sestoja (navezava na nalogo IM1), meritve temperature tal in ocena listnega indeksa (LAI). Opcijsko se bodo (v nekaterih državah članicah EU) izvajale meritve hidravlične prevodnosti tal in analize korenin.

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

Na sedmih izbranih ploskvah (Murska Šuma, Lontovž, Borovec, Fondek, Kladje, Brdo, Gropajski bori) so bile določene lokacije talnih profilov za namen spremljanje talnega profila volumske vlage in temperature tal v sestojih. Ker so tla v sestojih razmeroma heterogena smo se odločili za tri (3) ponovitve. V vsaki od teh ponovitev (v vsakem profilu) se bodo izvajale meritve volumske vlage na tleh na treh globinah v kolikor bo to dopuščala globina tal in sicer na 10cm, 30 cm, 60cm. Vzporedno, na istih globinah, bodo tekle tudi meritve talne temperature z dodatkom meritve na površini tal (0cm). V maju in juniju 2009 je potekala intenzivna nabava opreme, ki bo končana do konca junija 2009. Sprotne aktivnosti na ploskvah se odvijajo v obliki priprav talnih profilov na ploskvah.



Skica 1: Prikaz ploskev na katerih bodo potekale meritve volumske vlage tal in temperature tal z namenom pridobivanja vhodnih podatkov za izračun ocene vodne bilance za sestoje.



Fotografija 2: Prikaz talnega profila z mesti instalacije senzorjev za merjenje T in vlage tal (Foto: M. Ferlan)

V vsakem profilu se bo na prej navedenih globinah vzorčilo tudi neporušene vzorce tal, ki bodo služili za določanje vodno-zračnih lastnosti tal. Rezultati te analize bodo pomagali pri preučevanju vodnega cikla na ploskvah. V laboratoriju podvržemo z vodo nasičene vzorce več stopnjam nadtlaka in iz mase še zadržane vode v vzorcu sklepamo na silo, s katero tla zadržujejo vodo v svojih porah in s tem tudi, kako so dovzetna za stres suše.

Do sedaj so talni profili izkopani na ploskvi Gropajski bori, Brdo in Kladje, drugi pa bodo v mesecu juliju.

#### 1.6 Kakovost, strokovna presoja in ocena spremljanja depozitov (C1-DEP-22 FutMon LIFE+)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1. 1. 2009 - 31. 12. 2010 Vodja: D. Žlindra Sodelavci GIS: M. Špenko, M. Huibers, N. Filipič, I. Truden, M. Rupel, T. Brišnik, P. Simončič, A. Verlič novi Ostali sodelavci: K. Eler (BF odd. agr.), skrbniki ploskev (ZGS), ARSO

#### Namen in cilj raziskave:

Cilji akcije C1-Dep-22(SI) so izboljšanje, harmonizacija in razvoj metod za spremljanje depozitov. Vključevala bo nadaljnji razvoj 6. poglavja priročnika ICP Forests "Deposition" in koordinacijo primerjave vseh tipov vzorčevalnika za depozite (do konca leta 2010). Tako pridobljeni podatki bodo ovrednoteni, služili pa bodo tudi podpori akcijski skupini "D2" (akcija C1-Fol1-10(FI)).

Akcija je povezana s ploskvami, kjer se bo izvajala akcija "IM1". V okviru akcije "IM1" se bo na eni ploskvi vsake države postavilo, poleg že obstoječih, set standardiziranih vzorčevalnikov (32) sestojnih depozitov in set standardiziranih vzorčevalnikov (3) depozitov na prostem za periodo enega leta. Podatki bodo poslani vodilnemu partnerju.

Akcija vključuje prispevke k ovrednotenju demonstracijske akcije "Nutrient cycling and Critical Loads" (akcijska skupina "D2") na področju depozitov in kritičnih vnosov do konca leta 2010 (vodenje skozi akcijo C1-Fol1-10(FI).

Akcija vključuje podporo vodilnega partnerja pri razvoju kontrole ustreznosti, primernosti in enoličnosti podatkov za njihovo potrditev (validacijo).

#### Načrt aktivnosti:

Vsem državam bomo pomagali pri pridobitvi in načinu inštalacije standardiziranih vzorčevalnikov za depozite v sestoju (dodatno: za depozite na prostem). En set bomo kot referenčni set postavili na izbrani ploskvi IM1. Vzorčenje bo potekalo v enakem časovnih presledkih kot vzorčenje ostalih vzorčevalnikov za depozit. Enako bodo izvajane kvantitativne in kvalitativne analize dobljenih raztopin. Rezultati bodo podlaga za oceno hranilnih tokov, toka ogljika in drugih tokov. Rezultati bodo služili tudi oceni kritičnih vnosov in presežkov kritičnih vnosov na ploskvah.

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

V Hamburgu (12. - 15. junij 2009) je bil predstavnik GIS kot vodja akcije ter pomočnik vodje »ICP Forest Expert Panel on Deposition« prisoten na sestanku strokovne skupine za depozite (»Expert Panel on Deposition«) na prvem skupnem FutMon projektnem delovnem celotedenskem sestanku (Kick-off meeting). Ne delavnici je potekala razprava o aktivnostih akcije IM1, predvsem o usklajevanju, harmoniziranega meritev sestojnih padavin ter o tipu vzorčevalnika za sestojne padavine. Na tem mestu je pomembno poudariti, da je bil kot referenčni vzorčevalnik izbran vzorčevalnik slovenskega proizvajalca ROTO, narejenega na osnovi priporočil strokovnjakov GIS, sodelavcev Alterre (Nizozemska) ter predlogov strokovne skupine EPoD.

Aktivnosti, povezane z depoziti bodo imele težišče v akciji IM1/C1-DEP-22(SI), prispevali pa bodo tudi k akciji D2 in D3. Največ razprave je bilo na temo standardiziranih vzorčevalnikov za akcijo C1-DEP-22(SI), ki se bo izvajala na eni od ploskev IM1 na državo (**Priloga** <u>Depo HH minutes 2009.pdf</u>). Odločili smo se, da bomo:

- 1.) izvedli meritve 30 vzorčevalnikov sestojnih depozitov in 3 vzorčevalnikov na prostem, vzorce pa bi za kvalitativno in kvantitativno določitev združevali. in analizirali vsak vzorec posebej,
- 2.) vzorčevalnike razporedili po navodilih priročnika, čeprav se raziskovalne ploskve od države do države spreminjajo (oblika, površina, ...). Sistem razporeditve naj bi bil sistematično- naključen,
- 3.) vzorčili po nacionalnem sistemu (periode),
- 4.) združevanje vzorcev po sistemu 1 vzorec sestojnih usedlin in en vzorec usedlin na prostem. Navodila za združevanje bomo poslali kasneje.
- 5.) načrtovali vzorčevalnik, ki bo narejen po navodilih priročnika (Vzorčevalnik je sestavljen iz lija in zbirne posode. Material, iz katerega sta narejena, naj bi bil HDPE. Vzorčevalna površina naj bi bila vodoravna in zgornji del lija navpična. Površina vzorčevalnika mora biti gladka. Inertno sito / mrežica z odprtinami 1 mm naj bo vstavljena prosto na vrh vratu vzorčevalnika. Zbirna posoda mora biti v temnem in na hladnem.) Moral bi imeti obroč proti ptičem. Premer bo 16 cm in ne 20. Kapaciteta zbirne posode je 4 litre in nameščena v tla, če je mogoče. Cev, ki služi za povezavo med lijem in zbirno posodo, naj bi bila iz črnega PE. Višina vzorčevalnika bo 1 m nad površino, velja tako za vzorčevalnike v sestoju kot za vzorčevalnike na prostem. Ograjenost ploskve naj bo po nacionalnem sistemu.
- 6.) čistili vzorčevalnike po nacionalnem sistemu delovanja.
- 7.) naročili opremo od 1.3.09 do 31.5.2009.

Roki in odgovorni:

- za začetek proizvodnje: začetek marca (D. Žlindra, GIS),
- izdelovanje vzorčevalnikov: sredina aprila 2009 (D. Žlindra, GIS),
- postavitev vzorčevalnikov: 31. maj 2009 (partnerji projekta FutMon),

- začetek vzorčenja: 1. junij 2009 (partnerji projekta FutMon),
- konec vzorčenja: 1. junij 2010 (partnerji projekta FutMon).

Na sestanku, delavnici v januarju v Hamburgu EPD ni bil dorečen sistema obroča za zaščito pred pticam (kontaminacija depozita s iztrebki) in inštalacije vzorčevalnika ter še nekaj pomembnih podrobnosti. Zato smo po začetni okrožnici <u>»Invitation«, »Guidelines« in »Harmonized sampler« poslali v okrožnico še »Harmonized sampler 2«,</u> kjer poskušamo dobiti mnenja in informacije vseh udeležencev akcije (vse v prilogi + dopis\_mrežice.docx in ).

Velika večina sodelujočih je vzorčevalne lije in menzure že naročila, čeprav niso še vsi dobili dobavljene. Zaradi tega in ker še nismo uskladili oblike vzorčevalnika na terenu (ali je lij neposredno pritrjen na menzuro, ali je vmes cev, da je lahko menzura v tleh), se bo uradni začetek vzorčenja zavlekel iz predvidenega 03.06.2009 na kasnejši datum (julij ali avgust).

Ploskev v Sloveniji, kjer bo potekala vzporedna primerjava nacionalnih in harmoniziranih vzorčevalnikov, je ploskev 4 (Brdo pri Kranju). Dne 2.6.2009 smo v t.i. buferno cono ploskve v sestoju poleg dosedanjih vzorčevalnikov inštaliranih dodatnih 30 harmoniziranih vzorčevalnikov (3 harmonizirani na 1 nacionalnega) in še trije (poleg treh nacionalnih) na odprtem.



Fotografija 3: končni zgled vzorčevalnika z obročem proti ptičem (foto: D. Žlindra)



Fotografija 4: postavitev vzorčevalnikov na ploskvi Brdo (Foto: D. Žlindra)



Fotografija 5: postavitev vzorčevalnikov na prostem (Foto: D. Žlindra)

Vzorčevanje je steklo 17.6.2009. Potekalo bo po dosedanjem sistemu, kjer se vzorca iz prve in druge 14-dnevne periode združita v enega.

#### 1.7 Upravljanje projekta (M7 FutMon LIFE+)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1. 1. 2009 - 31. 12. 2010 Vodja: P. Simončič Sodelavci GIS: M. Kovač, Brišnik, A. Verlič, D. Žlindra, S. Kristan, N. Milenković Ostali sodelavci: vTI (Hamburg, Nemčija)

#### Namen in cilj raziskave:

Namen akcije je upravljanje projekta na nacionalni ravni s financami, osebjem, z mrežo ploskev, laboratorijskimi analizami, kontrolo kvalitete aktivnosti. Delo s podatkovno bazo projekta z vsemi podsklopi je del naloge M8. Prav tako tudi diseminacija, širjenje znanja in informacij rezultatov naloge.

#### Načrt aktivnosti:

Finančni vođenje naloge bo izveđeno v skladu z nacionalno zakonodajo in relevantno EU zakonodajo; Uredba (ES) št. 614/2007 Evropskega parlamenta in Sveta, o finančnem instrumentu za okolje, LIFE+ z dne 23. maja 2007 in v skladu z pogodbo ter splošno določbo EU Life + (*http://ec.europa.eu/environment/life/toolkit/ pmtools/lifeplus/cp.htm*) in dogovorom z vodilnim partnerjem, EU DG ENV in nacionalnimi sofinancerji. V skladu s planom bodo pripravljeni letni poročili o aktivnostih, ki bodo posredovana vodilnemu partnerju vTI iz Hamburga in nacionalnim sofinancerjem. V aktivnosti M7 je sodelovanje na združenih srečanjih strokovnjakov (Combined Expert Meetings), statusnih delavnicah in srečanjih vodij laboratorijev, ki sodelujejo v projektu. Sodelovanje z uporabniki znanj (javne predstavitve, sodelovanje na javnih tribunah...), na znanstvenih seminarjih in pripravo prispevkov za medije bodo del aktivnosti naloge M8, deloma pa M7.

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

V prvi polovici l. 2009 so potekale aktivnosti pri urejanju številnih formalnosti glede podpisa pogodbe z koordinatorjem projektne naloge z inštitutom »vTI« iz Hamburga iz Nemčije. Urejali smo dokumentacijo in usklajevali dogovor (pogodbo) z nacionalnimi financerji naloge (MKGP in MOP).

Med EU projektnimi partnerji so potekale razprave glede metodologije dela, ki predstavlja del ciljev projektne (oblikovanje novih navodil za delo po posameznih že obstoječih ter tudi novih aktivnosti, meritev). V Sloveniji je in poteka (trajna naloga) urejanje razmer za delo za skrbnike ploskev ob hkratnem izobraževanju tako skrbnikov kot delavcev GIS. Vzporedno z delom potekajo številne delavnice in prenosi znanja. Nakupi opreme so zaradi oblikovanja metodologij delno zaostajajo, vendar ne več kot mesec ali dva, tako, da pričakujemo da se to ne bo pokazalo na ravni leta (prvega leta raziskav). Zaradi narave projekta, katerega cilj je uvajanje novih indikatorjev spremljanja stanja gozdov in preizkušanja novih pristopov k vseevropskemu monitoringu gozdov se morajo izvajajo določene terminske kot tudi finančne korekcije, vendar vse znotraj načrtovanih aktivnosti in v okviru »Splošnih določb« finančnega okoljskega mehanizma Life+. Te korekcije bomo izvedli takoj po oddaji poročila za nacionalne financerje MKGP in MOP.

Predstavniki GIS so sodelovali na delovnih sestankih svojih strokovnih skupin:

- a) Daniel Žlindra (depoziti, delavnica za Life+),
- b) Marko Kovač (osutost/D1, tla, delavnica za Life+),
- c) Tom Levanič (meteorologija, rast D1, delavnica za Life+),
- d) Primož Simončič (mineralna prehrana in D2, fenologija, tla, delavnica za Life+): začetne delavnice projekta FutMon v Hamburgu, januar 2009).
- e) Primož Simončič in Mitja Ferlan (delavnici za D3, meritve vlage tal in fizikalnih lastnosti tal, marec 2009, Freising, Nemčija).
- f) Lado Kutnar (EG Ground vegtation, Rim)
- g) Marko Kovač (akcija L1 in L2, Firence, Italija)
- h) Gal Kušar (Task Force ICP Forest in FutMon sestanek, St. Petrsburg, Rusija)
- i) Organizacija fenološke delavnice FutMon Combined Field Course on Phenology and LAI, od 5.- 7. maja 2009 v Lipici (sodelavci GIS in ZGS).

#### 1.8 M8 (FutMon LIFE+)

FutMon Life+ aktivnost M8 - Prenos rezultatov in obveščanje javnosti na nacionalni ravni (2009-2010)

Naročnik: EU DG. ENV., MKGP, MOP Šifra: LIFE07 ENV/D/000218 Trajanje naloge: 1.1. 2009 -31.12.2010 Vodja: M. Kovač Sodelavci GIS: G. Kušar, A. Japelj, M. Skudnik, J. Žlogar, P. Simončič , T. Levanič, D. Žlindra, T. Brišnik Ostali sodelavci: P. Ogrinc, M. Mlinar

#### Namen in cilj raziskave:

Namen tega sklopa projekta je razviti sistem zajemanja, hranjenja, logičnega kontroliranja in posredovanja podatkov za vse module projekta.

V letu 2009 so cilji naslednji:

- Zasnova organizacije podatkov projekta in načinov vnosov, logične kontrole, testiranje programa.
- Tekoče vnašanje podatkov.

#### Načrt aktivnosti:

- Podatkovna organizacija projekta (zasnova povezav med moduli)
- Definiranje mask in logičnih kontrol
- Testiranje programov
- Definiranje protokola za vnos, pregledovanje, popravljanje podatkov,
- Vnašanje podatkov za vse module
- Sodelovanje z uporabniki znanja, prenos znanja, predstavitve rezultatov javnosti.

#### Polletno (1.1.2009-30.6.2009) poročilo aktivnosti:

V prvi polovici leta se je zasnovalo novo strukturo podatkovne zbirke, definiralo maske in logične kontrole. Definiralo se je protokole za vnos, pregledovanje in popravljanje podatkov. Z logičnim kontrolami se je preverilo in popravilo staro podatkovno zbirko. Testiralo se je tudi programe za izračune vrednosti parametrov snemanja. Pripravili so se vpisni listi za ugotavljanje povzročiteljev poškodb in z namenom lažjega vnosa v bazo podatkov so se za vse nove indikatorje, ki se bodo popisovali na terenu, pripravili šifranti z numeričnimi kodami.

#### 2 POROČILO O KROŽNIH ANALIZAH (FOLIARNE)

S projektom FutMon je prišlo v ospredje vprašanje zanesljivosti in pravilnosti podatkov, generiranih tekom projekta. V ta namen se je in se bo v letu 2009 zvrstilo 6 krožnih analiz za različne matrikse vzorcev: tla (kemijski parametri), tla (potencial zadrževanja vode), iglice/listi, vode, ozon (pasivni vzorčevalniki). Na sestanku v Hamburgu je bilo domenjeno, da po končanem krožnem testu vsak laboratorij prejme poročilo o kvalifikaciji (qualification report). Da se posamezen laboratorij kvalificira za analizo določenega parametra, mora biti vsaj 50% vseh rezultatov za posamezen parameter v sprejemljivih mejah, ki so navedene, in vnaprej določene, v dokumentu delovne skupine za kvaliteto. Manjkajoči podatki se pri krožnih testih obravnavajo kot napačen rezultat.

Dva krožna testa sta že zaključena t.j. test s foliarnimi in vodnimi vzorci. Trije so v teku (oba testa s talnimi vzorci in ozon).

V prvem krožnem testu (foliarni vzorci) se je Laboratorij za gozdno ekologijo (LGE, koda v projektu FutMon je **F27**) Gozdarskega inštituta Slovenije kvalificiral za vse obvezne parametre: N, S, P, K, Ca, Mg in za opcijskega: C (**Priloga** <u>11 WRT foli.pdf</u>).

Enako se je LGE GIS kvalificiral za vse obvezne parametre vzorcev vod: pH, elektroprevodnost, alkaliteta, natrij (Na), amonij (NH<sub>4</sub>), kalij (K), kalcij (Ca), magnezij (Mg), klorid (Cl), nitrat (NO<sub>3</sub>), sulfat (SO<sub>4</sub>), celokupni dušik (Tot N), raztopljen organski ogljik (DOC) (**Priloga** <u>WRT2009 results.xls</u>).

#### 3 Poročilo o posodobitvi navodil za izvajanje aktivnosti IM1

Glede na dogovor , ki je bil dosežene na prvem projektnem sestanku in delavnicah (januar, Hamburg, FutMon kick off meeting) so bili pripravljeni t. i. protokoli za delo na terenu. Njihov namen je da se opredeli cilje oz. naloge ki se morajo izvajati glede na projektno dokumentacijo in dogovor med projektnimi partnerji in koordinatorjem projekta FutMon (vTI, Hamburg) in ne presegajo zavez pogodbe.

Nekateri protokoli se ta trenutek v delovni obliki in so dosegljivi na spletnih straneh projekta FutMon, vendar le s pomočjo uporabniškega imena in gesla.

Končne verzije protokolov za terensko delo, ki so bile dokončane do 27. maja 2009, so (priloga 8):

- a) FutMon QA/QC Guide for Laboratory Work (1st version);
- b) Field protocol on continuous measures of forest growth, Action Group D1: Tree vitality and adaptation;
- c) FUTMON FIELD PROTOCOL PHENOLOGY (D1), V1.1; last update 19th May 2009;
- d) Field protocol on permanent and continuous measures of forest growth –
   Expert Panel Crown Condition and Assessment Damage Causes, Tree Vitality (D1), FutMon Field Protocol;
- a) Expert Panel Crown Condition and Assessment Damage Causes Tree Vitality (D1) FutMon Field Protocol ;
- b) FutMon (Life+) field protocol: Sampling procedure for evaluation of nutrient budgets in vegetation in FutMon intensive monitoring plots and more intensive foliage surveys (D2), V 1.0; last update 15th May 2009;
- c) Litterfall sampling and analysis, FutMon (Life+) Field Protocol 2009V1; last update 14th May 2009, IM1 recommended, mandatory on D1 and D2 Demonstration Project plots;
- d) Field\_prot\_pheno\_V1\_15May09.doc page 1/6 1 FUTMON FIELD PROTOCOL PHENOLOGY (D1), V1.0; last update 15th May 2009;
- e) field\_prot\_SoilWater\_v1\_150509.doc page 1 / 12FutMon Field Protocol, Determination of the soil water retention characteristic.,

#### 4 Poročilo o aktivnostih na področju QA (<u>http://bfw.ac.at/rz/bfwcms.web?dok=7887</u>)

Prav poseben poudarek v projektu FutMon je namenjen področju zagotavljanja in ohranjanja kvalitete na vseh področjih dela v projektu, od terenskega dela do dela v laboratoriju, preverjanje rezultatov in analiza podatkov (**Priloga DraftFutMonFieldProtocolQualLabs.doc**). Z uvajanjem zagotavljanja kvalitete želimo v projektu FutMon uvesti sledljivost in pravilnosti podatkov (popolnoma novo področje), generiranih tekom projekta. V ta namen bo v vsaki datoteki sporočanih rezultatov tudi zaznamek, ali se je laboratorij kvalificiral za analizo tega parametra ali ne. Kvalifikacija pomeni, da mora biti vsaj 50 % vrednosti za posamezen parameter, analiziran v krožnem testu, znotraj sprejemljivih mej. Od januarskega srečanja v Hamburgu je bila uvedena novost, da se manjkajoči podatki obravnavajo kot napačen rezultat, kar prizadene laboratorije, ki so se z neporočanjem 'sumljivih' vrednosti izogibali slabih splošnih rezultatov. (**Priloga MinutesWGQAOCHamburg2009.doc**)

Poleg univerzalnih metod nadzora dela (vzporedna analiza kontrolnih vzorcev, uporaba kontrolnih kart, ...) uporabljamo tudi specifične indikatorje kakovosti (za vode npr. ionsko ravnovesje, bilanca dušika, razmerje natrij:klorid, izračunana/izmerjena elektroprevodnost). Veliko podobnih indikatorjev se lahko uporablja tudi v primeru talnih vzorcev (npr.: celokupna vsebnost > vsebnost, določena z zlatotopko > vsebnost, dobljena z ekstrakcijo z barijevim kloridom). Na ta način tudi smiselno preverjamo dobljene vrednosti posameznih parametrov in se ne opiramo samo na primerjalne vrednosti kontrolnih vzorcev.

Za lažjo in hitrejšo komunikacijo med laboratoriji projekta FutMon so kode posameznih laboratorijev prosto dostopne ostalim laboratorijem, z namenom, da v laboratorij s težavami poišče nasvet ali rešitev pri laboratoriju, ki je geografsko blizu in ima dober rezultat v krožnem testu. Laboratorijem z zelo slabimi rezultati pa je v okviru FutMon projekta dostopna pomoč izkušenih in dobro delujočih laboratorijev.

#### 5 Poročilo o fenološki delavnici v Sloveniji

V Lipici je od 5. do 7. maja potekala skupna FutMon delavnica za fenologijo in LAI. Na njej je bilo prisotnih 29 strokovnjakov iz 16 držav.

Prvi dan je bil seminarski in je potekal v Lipici v hotelu Maestoso. Pri fenološkem delu sta bili predstavljeni obe obliki spremljanja in sicer že preizkušena metoda spremljanja fenologije s pomočjo daljnogleda kot tudi novejša oblika spremljanja preko kamere. Za posnetke posnete s kamero je bil izveden tudi kratek test. Delavnica se je nato nadaljevala s predavanjem o meritvah LAI.

Drugi dan je bil terenski dan, ki se je začel z ogledom ploskve Gropajski bori pri Sežani in prikazom merjenja LAI.

Ekskurzija se je nadaljevala s prikazom spremljanja fenologije na ploskvi s črnim borom.



Fotografija: 6 Spremljanje fenologije na ploskvi s črnim borom (foto: A. Verlič)

Nadaljnje fenološko opazovanje je potekalo na višinskim profilu (450 - 1150 m) vse do pod vrhom Nanosa, kjer se je zaradi nadmorske višine odganjanje bukve šele pričelo. Drevesa so bila precej različna v razvitosti (od razreda 1 do razreda 5). Na tej ploskvi je bilo označenih 15 dreves bukve in vsak izmed udeležencev jih je najprej ocenjeval individualno, nato pa smo vsa drevesa ocenili še skupinsko in zraven poskušali uskladiti morebitna različna mnenja. Opazile so se razlike v ocenjevanju razvitosti ozelenjevanja, poleg tega pa tudi v opažanju cvetenja bukve.



Fotografija 7: Ocenjevanje (interkalibracijski test) fenologije pri bukvi pod Nanosom (foto: L. Kutnar)

Med ekskurzijo so bila na dveh ploskvah (ploskev Gropajski bori s črnim borom in ploskev pod Nanosom z bukvijo) prikazana merjenja LAI z različnimi metodami, pogovor je tekel o razlikah med metodami .



Fotografija 8: Demonstracija meritev svetlobe v bukovem sestoju (foto: M. Urbančič)

Razgovor je tekel tudi o problemih glede vremenskih razmer (potrebuje se difuzna svetloba) in problemih z dovolj veliko referenčno ploskvijo na prostem (v bližini).

Naslednji dan je pri fenološkem delu sledila analiza reševanja vprašalnika. Ugotovili smo, da je pri snemanju prišlo do kar precejšnjih odstopanj, saj so bili precejkrat zasedeni skoraj vsi možni razredi. Po kalibraciji oči bi bil rezultat zagotovo boljši.

Na delavnici so se poudarile novosti pri opazovanju fenologije v sklopu projekta FutMon.

Prva izmed njih je zagotovo izvajanje fenološkega popisa s pomočjo kamere. Zaradi izpolnjevanja kriterijev pogodbe FutMon, naj bi vsaka izmed sodelujočih držav testirala kamero na vsaj eni ploskvi.

Še naprej bodo veljale smernice ICP Forests Manual (Phenological Observation), vendar so dodane nekatere spremembe:

- Še vedno sta možni tako ekstenzivna na ravni ploskve kot tudi intenzivna opazovanja – na ravni drevesa, vendar je, kadar je možno, priporočeno uporabljati intenzivna opazovanja (v Sloveniji imamo le snemanja na ravni drevesa).
- 2. Spremenjeni so nekateri obrazci (PHE, PHI in PLP).
- 3. Minimalna frekvenca opazovanja med kritičnimi fazami pri klasičnem opazovanju je enkrat na teden, optimalno je vsakodnevno spremljanje. Nujni sta dve fazi in sicer ozelenjevanje ter jesensko obarvanje.
- 4. Pri uporabi kamere je nujno vsakodnevno spremljanje in sicer naj bi kamera posnela vsaj dva posnetka na dan za drevo (zaradi različne svetlobe), nato pa naj bi se trajno shranil boljši izmed obeh posnetkov. Pri uporabi kamere so obvezne vse faze fenologije.
- 5. Uporaba kamer je namenjena za odmaknjene ploskve, kjer z nakupom relativno drage opreme vseeno prihranimo glede na potovalne stroške.
- 6. Če se med snemanjem opazi biotske spremembe na opazovanih drevesih, jih je potrebno zabeležiti in sicer v obrazca PHI oz PHE. Natančnejše preiskave, opise in oddaja podatkov glede opazovanih poškodb je potrebno opraviti glede na navodila ICP Forests o poškodbah gozda. Če je potrebno, mora na ploskev priti trenirano osebje in sicer najkasneje 4 tedne po prijavi poškodbe.
- 7. Zaradi možnosti spremljanja fenologije preko kamere je potrebno v popis izbranih dreves vključiti tudi vertikalno smer pogleda, torej ali se drevo gleda od spodaj (1), na višini krošnje (2) ali od zgoraj (3).
- 8. Med popisom je potrebno označiti tudi metodo opazovanja:
  - 1. Terensko opazovanje
  - 2. Digitalna kamera
  - 3. Tako terensko opazovanje kot tudi kamera

- 9. Sprememba je v točkovanju intenzivnosti fenološkega pojava (zelenjenja, jesenskega rumenjenja in odpadanja listja) in sicer se zaradi problemov s številko 0, zdaj točkuje od 1 do 5 (v Sloveniji smo to že uporabljali) in sicer:
  - 1. < od 1%
  - 2. = 1 33%
  - 3.  $\geq 33 66\%$
  - 4.  $\geq 66 99\%$
  - 5. ≥99%
- 10. Sprememba je nastala tudi pri točkovanju intenzitete cvetenja, vendar je dodatno ocenjevanje opcijsko (sistem ocenjevanja deloma velja tudi za poškodbe):
  - 6= cvetenje oz poškodbe niso prisotne

#### 7 = cvetenje oz poškodbe so prisotne

- 7.1 = cvetenje je posamično (opcijsko ocenjevanje)
- 7.2 = cvetenje je zmerno (opcijsko ocenjevanje)
- 7.3 = cvetenje je močno (opcijsko ocenjevanje)

Uporaba kamer prinaša prednosti in slabosti. Prednost uporabe kamer je zagotovo v stalnem (kontinuiranem) spremljanju (tudi na odmaknjenih lokacijah), v možnosti medsebojne primerjave več ploskev, iste ploskve v različnih letih ali primerjave med državami oz regijami. Ocenjevanja so lahko narejena, ko je na voljo osebje (ni nujno, da so opravljene točno določen dan), poleg tega je lažje določiti začetek pojava poškodb.

Slabosti kamer so v visokih investicijskih stroških, potrebi po baterijah, možnosti tehničnih okvar in vandalizma ter slabem videnju v gostejših (iglastih) gozdovih.

Pri uporabi kamer je potrebno zagotoviti dovolj kvalitetno slike, da se lahko opravi ocenjevanje fenologije po protokolu, poleg tega mora slika zadostiti tudi morebitnemu ocenjevanju poškodb. Na ploskev mora biti spremljanih vsaj 10 dreves.

Kamere morajo biti odporne proti zunanjim (vremenskim) vplivom, slike morajo biti visoke ločljivosti (minimalna zahteva je 6Mpix z 300 pix/cm) – tudi pri polnem zoomu. Kamera potrebuje svoj spomin ali biti priključena na hranilnik podatkov (datalogger), ki mora biti shranjen na prostoru, ki je zaščiten pred vremenskimi vplivi. Napajanje je možno preko baterij, sončnih celic ali priključka na elektriko. Delovanje kamere je potrebno preveriti vsakič, ko je nekdo prisoten na ploskvi (ob vsakem obisku).

Lokacija kamere: Najboljše je, če je kamera pritrjena na drog, ki sega nad krošnje. Da bi kamera lahko snemala več dreves, mora biti gibljiva in imeti možnost programiranja, tako da lahko ob določenih časih snema vedno iste pozicije dreves. Lokacija kamere naj bo izbrana tako, da bo na čim večjem področju lahko posnela čim večje število posameznih dreves. Kamera je lahko locirana tudi pod krošnjami dreves, vendar je tako možnost snemanja večjih dreves precej omejena, kar se da kompenzirati z večjim številom kamer. Kamera mora posneti celoten zgornji del krošnje.

Slike morajo biti posnete večkrat dnevno (vsaj dvakrat) zaradi spremembe pogojev svetlobe. Podatki morajo biti zaradi njihovega zavarovanja pobrani vsaj enkrat na dva meseca. Kamera je lahko povezana tudi na mrežo, tako da so lahko opazovanja narejena tudi od doma. Vseeno je pametno, če so slike shranjene tudi na ploskvi (varnostna kopija).

Slike različnih ploskev naj bi analizirala ena oseba, kar je obvezno, ko gre za ploskve z istimi drevesnimi vrstami. Tako je izločen vpliv različnih opazovalcev. Za popis se uporabljajo enake kode kot za terenska opazovanja. Analizira se le eno opazovanje dnevno.

Slike morajo biti shranjene, saj so tako lahko uporabljene za interkalibracijo in za primerjavo med državami. Med kritičnimi fazami mora biti za posamezno drevo shranjena vsaj ena slika dnevno, za ostalo sezono pa je dovolj ena slika na teden. Slike morajo biti dostopne tudi ostalim partnerjem projekta (lahko tudi v FutMon podatkovnem centru).

#### 6 Poročilo o delavnicah za skrbnike ploskev IM1

V četrtek, 11. 6. 2009 je v Lipici potekala združena delavnica za skrbnike ploskev naloge FutMon in za predstavnike območnih enot Zavoda za gozdove, zadolženih za varstvo gozdov.



Fotografija 8: Skupna delavnica FutMon v Lipici (foto: A. Verlič)

Prvi dopoldanski del (Popis škodljivih biotskih dejavnikov v okviru popisa stanja gozdov (FutMon Life+), je bil namenjen obnovitvi znanj o škodljivih organizmih na gozdnem drevju. Prvo predavanje s poudarkom na boleznih je vodil Doc. Dr. Dušan Jurc z Gozdarskega inštituta. Predstavil je simptome bolezni, ki prizadenejo naše gozdove.

Drugi del predavanja o gozdnih škodljivcih je vodila Prof. Dr. Maja Jurc z Biotehniške fakultete, Oddelka za gozdarstvo in obnovljive gozdne vire. Poudarek je bil na prepoznavanju poškodb, ki jih povzročajo različni škodljivci, večinoma žuželke.

Po skupnem dopoldanskem delu smo se razdelili v dve skupini. Prva skupina (Posebni nadzor škodljivih organizmov) je ostala v hotelu in poslušala naslednja predavanja:

- a) Marija Kolšek vloga ZGS v posebnih nadzorih,
- b) Prof. dr. Maja Jurc kostanjeva šiškarica (*Dryocosmus kuriphilus*), kitajski in azijski kozliček (*Anplophora chinensis, A. glabripennis*)

- c) Dr. Nikica Ogris fitoftorna sušica vej (*Phytophthota ramorum* in *P. kernoviae*), javorov rak (*Eutypella parasitica*)
- d) Barbara Piškur borov smolasti rak (Gibberella circinata)
- e) Tine Hauptman jesenov ožig (*Hymenoscyphus albidus, Chalara fraxinea*)
- f) Doc. dr. Dušan Jurc šarka (Plum pox potyvirus), hrušev ožig (*Erwinia amylovora*).

Druga skupina v kateri so bili skrbniki ploskev je odšla na teren in sicer na ploskev Gropajski bori pri Sežani.



Fotografija 9: Nadaljevanje delavnice na terenu (Foto: D. Žlindra)

Tu so potekale predstavitve izbranih aktivnosti, ki potekajo v okviru projektne naloge FutMon Life+:

- a) Košiček B., OE Sežana, ZGS: uvodna predstavitev območja
- b) Brišnik T. fenološka opazovanja drevja
- c) Ferlan M. meteorološke meritve in meritve vlage tal
- g) Čater M. predstavitev meritev svetlobnih razmer
- h) Levanič T. predstavitev programa dendrometrijskih meritev
- i) Jurc D. popis škodljivih biotskih dejavnikov v okviru popisa stanja gozdov (prikaz v praksi)
- j) Zaključek in dogovor o nadaljnjem sodelovanju GIS / ZGS.



Fotografija 10: Predstavitev meritev vlažnosti tal (Foto: A. Verlič)

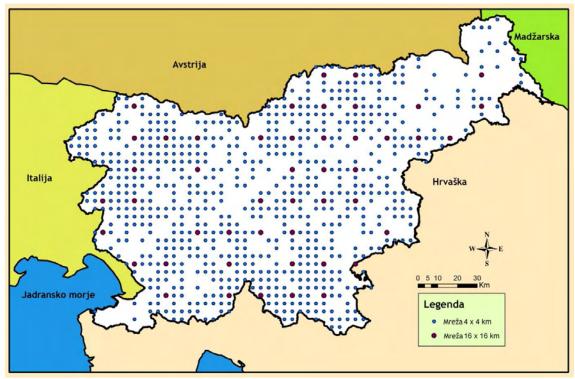
V predstavitvah so bile prikazane meritve, ki bodo v letošnjem in drugem letu potekale na ploskvah in predstavljena oprema, ki je na novo postavljena na ploskvah. Skrbniki so bili naprošeni, da ob vsakem obisku pregledajo naprave ter sporočijo očitne nepravilnosti (npr. podrte ali huje poškodovane opreme). Pri aktivnostih, ki so potekale tudi v prejšnjih letih so bile posredovane novosti ter opozorila na napake prejšnjih let in izboljšave starejših postopkov.

Skupne delavnice se je udeležilo 63 udeležencev iz 14 območnih enot Zavoda za gozdove Slovenije, iz centralne enote ZGS, iz Gozdarskega inštituta Slovenije in predstavnik JGZ Brdo (skrbnik ploskve). Od tega je bilo 23 udeležencev druge skupine (skrbniki ploskev in sodelavci z Gozdarskega inštituta).

## 7 Poročilo o harmonizaciji inventurnih sistemov ICP in NFI (L1)

V geografskem informacijskem sistemu se je izdelal t.i. »mreže« teoretičnih točk, ki so razporejene preko Slovenije na vzorčni mreži 16x16 km. Kot izhodišče prve točke na vzorčni mreži se je določila točka, ki se nahaja 50 metrov vzhodno od štirih M6 ploskev, ki so bile uporabljene za monitoring osutosti in poškodovanosti gozdov (ICP Forest). V programskem okolju ArcGIS (modul ArcMap 9.3) se je glede na atributne podatke karte rabe tal MKGP in glede na stanje vidno na digitalnih ortofoto posnetkih, preverilo ali so te točke locirane v gozdu ali izven njega. Pri določitvi gozdnega prostora na DOF-ih se je upoštevala definicija gozda kot ga določa Zakon o spremembah in dopolnitvah Zakona o gozdovih (Ur. l. RS št. 110/2007). S tem se je ugotovilo teoretično število vzorčnih ploskev, ki bi jih bilo potrebno zajeti v sistem velikoprostorske gozdne inventure.

V prvi polovici leta 2009 je bila preverjena gostota vzorčne mreže in razporeditev vzorčnih ploskev ter korektnost izbire ploskev (gozd, negozd). Vzorčna mreža 16x16 km ostaja enaka kot doslej in ustreza evropskim zahtevam, za periodično (5-10 let) zanesljivejšo velikoprostorsko inventuro pa obstaja gostejša (4x4 km) vzorčna mreža. V teku je preverjanje statistične moči preizkusa (statistical power) glede na gostoto vzorčne mreže za izbrane parametre monitoringa. Statistična moč preizkusa se nanaša na število potrebnih ponovitev (gostoto vzorčnih ploskev), da se statistično zazna razlika v ocenah sprememb parametrov med leti z gotovo (dano) zanesljivostjo. To praktično pove, koliko vzorčnih ploskev je v naslednjem letu potrebno posneti, da se zazna razlika v ocenah parametra za 3 % glede na prejšnje leto. Za vzorčno mrežo 4x4 km za parameter lesne zaloge dreves znaša moč preizkusa približno 200 ploskev namesto 700. Za vzorčno mrežo 16x16 izračunavanje statistične moči preizkusa še poteka. Preverjeno je že, da Slovenija za korekten integralen monitoring gozdov in gozdnih ekosistemov ter spremljanje stanja in razvoja potrebuje vzorčno mrežo 4x4 km (periodična snemanja), za nadzor kritičnih sprememb pa zadostujejo vsakoletna snemanja na redkejši 16x16 km vzorčna mreža. Razvite so bile premostitvene funkcije za povezavo podatkov preteklih snemanj z novim snemanji (kontinuiteta časovnih vrst) za vse parametre razen za poškodbe drevja zaradi biotskih vzrokov. Za slednje priprave premostitvenih funkcij še potekajo.



Slika : Razporeditev ploskev vzorčne mreže 4 x 4 km in 16 x 16 km.

## Delavnica »FutMon Status«, 26-27.5.2009. St. Petersburg, Rusija

## Poudarki iz delavnice:

- uvodna predstavitev projekta (začel leta 2009, vključno z 2010),
- Slovenija (22) je poslala vso zahtevano dokumentacijo,
- za ploskve IM1 potrebno na terenu postavit table, da gre za FutMon Life+,
- predstavitev revizije sistema monitoringa in sinergije z ICP Forest,
- predstavitev statusa in napredka projekta po posameznih akcijah (D1, D2, D3 in C1),
- upravljanje projekta (zahteve, naloge, poročila, roki,...),
- upravljane baze podatkov,
- aktivnosti v monitoringu,
- zagotavljanje kakovosti (QAQC),
- analiza podatkov in poročila,
- diskusija glede tega, kaj je obvezno in kaj priporočljivo meriti ter kje (na katerih ploskvah),
- pohvala Sloveniji za izvedbo seminarja o fenologiji v Lipici.

Dokumenti delavnice so dostopni na spletnih straneh: (http:// www.futmon.org/lock\_internal/St.%20Petersburg.htm) Delavnici "<u>NFI related activities in the Life+ - FutMon project</u>" in "<u>Strategies for</u> <u>the integration of large scale forest monitoring networks</u>" 19-20.3.2009, Firence, Italija

## Poudarki iz prve delavnice:

- predstavitev FutMon akcije C1-NFI
- izbor ključnih parametrov snemanj glede na harmonizacijo znotraj projekta Cost E43 in informacijski potreb za tekoče projekte in poročanja (JRC Framework project, EEA forest type classification to MCPFE, Kyoto reporting),
- načrtovanje terenskih meritev,
- podatkovne zbirke in oblike poročil.

## Poudarki iz druge delavnice:

- predstavitev FutMon akcije C1-HarmonLS.
- povezave med projekti,
- poročanja po državah glede stanja sistemov in povezav med NFI in ICP vzorčnim ploskvami in mrežami; sedanje stanje, prihodnja integracija, koordinacija, standardizacija aktivnosti,
- zaključki in prihodnje delo,

Dokumenti z delavnice so dostopni na : http://www.aisf.it/futmon/

Priloge

## **Draft Qualification Report**

11th Needle/Leaf Interlaboratory Comparison Test 2008/2009

Daniel Zlindra Slovenian Forestry Institute Laboratory for Forest Ecology Vecna pot 2 SI-1000-Ljubljana Slovenia FutMon-Beneficiary: 022 Laboratory code: 13 (new code: F27)

Parameter	analysed	passed	Percentage of correct results	Remarks	Requalification passed
N	yes	yes	100		
S	yes	yes	75		
P	yes	yes	100		
Ca	yes	yes	100		
Mg	yes	yes	50		
K	yes	yes	100		
Zn					
Mn					
Fe					
Cu					
Pb					
В					
Cd					
С	yes	yes	50		

passed: 50% of the results per element or more are within the tolerable limits not passed: less than 50% of the results per element are within the tolerable limits

18.02.2009

Alfred First

Alfred Fürst Forest Foliar Co-ordinating Centre

FutMon/ICP Forests Combined Expert Meeting, 12 - 16 January 2009

Meeting of the Expert Panel on Meteorology Field measurements for water budget modelling (Action D3) Meeting 13 Hamburg, Germany, Thursday 15-16 January 2009

## **Draft Minutes**

Chair: Stephan Raspe

**Participants:** Richard Fischer (vTI); Vít Šrámek (CZ); Karl Gartner (AT); Mayte Minaya (ES); Erwin Ulrich (FR); Matthias Dobbertin (CH); Zuzana Sitkova (SLO); Radu Cenusa (RO); Tom Levanic (SI); Miklós Manninger (HU); Matt Wilkinson (UK); Albertas Kasperavicius (LT); Pawel Lech (PL); Bruno Petricione (IT); Panagiotis Michopoulos (GR) (partly)

#### Agenda: see Annex 1

#### **Results:**

## 1) Opening and focus of the meeting

- a Importance of meteorology and hydrology under FutMon increases
- b D actions only on IM1 plots with full set of surveys, this means that information on meteorology will be mandatory for D actions plots
- c Former optional surveys will be mandatory for D3
  - Soil volumetric water content
  - Matrix potential
  - Soil temperature
  - Stand precipitation

#### 2) Field measurements of the D3 project

- a Objectives of action D3
  - to demonstrate the feasibility of more intensive soil moisture measurements
  - D3 will only deal with data collection
  - data are needed for the development and implementation of water budget modells on intensive monitoring plots
- b Surveys spec. necessary for D3 action
  - Soil volumetric water content
  - Matrix potential
  - Stand precipitation
  - Soil temperature

- c Relevance of parameters
  - Soil volumetric water content
    - \* Soil moisture
    - \* Easy to measure over the whole range
    - \* Validation of models
    - \* Together with matrix potential  $\rightarrow$  field pF
    - \* Measured water availability
  - Matrix potential
    - \* Suction power of the soil  $\rightarrow$  water flow
    - \* Limited range (< 800 1000 hPa)
    - \* Validation of models
    - \* Together with soil water content  $\rightarrow$  field pF
  - Stand precipitation
    - \* Real input into the soil  $\rightarrow$  water flow
    - \* Calibration and validation of models
    - \* Together with open field precipitation  $\rightarrow$  interception
  - Soil temperature
    - \* Physical condition of water
    - \* Physiological status of roots
    - \* Validation of models
    - \* Easily to measure

## 3) Status of meteo measurements on Level II plots

Austria: current: 10% of Level II plots with meteo incl. Soil moisture measurement; planning for FutMon 6 D3 plots

Poland: planning: from 12 FutMon plots 6 with meteo

Italy: FutMon 22 plots, 13 core plots. External meteo and soil moisture data

United Kingdom: 10 Level II plots, 8 meteo stations, outside

Czech Republic: 14 Level II plots; 12 meteo stations (4 incl. wind); 10 soil moisture plots

Hungary: 8 FutMon plots with 5 meteo stations which covers all 8 plots; 2 plots with soil moisture measurement

Spain: 30 IM1 plots; 14 meteo stations

Romania: 12 Level II plots; 4 D3 plots, no meteo stations, but possibility to by meteo data for 2-3 plots from national weather service

- Slovenia: 6 Level Ii plots; 2 meteo stations (1 tower), wind measurement in 2 meter hight, soil moisture measurement with Ecco probe and Gypsen block
- Lithuania: no meteo measurement, planned to by data from national weather service for 2 3 stations, reduced parameter set possible?
- Slovacia: At present only 2 from all 7 meteo plots at open field are working favourably, in accordance with a list of mandatory meteorological variables, except one plot where wind variables measurement is not at 10 m height, but only at 2 m. Because of long distance of Level II plots from weather stations we usually do not use

external data from Slovak Hydrometeorological Institute (SHMI). In case of need it is possible to require the raw meteorological data but access is not free (feebased services)

- France: currently only 13 plots with automatic stations (T, RH, P and only 4 of them with wind speed, wind direction and global radiation) for the other stations we can buy meteo data from the official weather service, but very often the stations are farer than our owns ones from the plots in the forest.
- Mecklenburg-Western Pomerania: 2 FutMon plots, all with meteo, stand precipitation from depo survey
- Bavaria: 10 FutMon IM1 plots all with meteo; 6 D3 plots with soil moisture measurements since 1998
- Rhineland-Palatinate: 3 FutMon IM1 plots all with meteo measurements at open field plots, measurement or stand precipitation via depo measurements.

## 4) Requirements on soil moisture measurements

- soil water content (WC) should be measured
- matrix potential will be calculated by measured WC and measured pF curves (see meeting 12 Combined meeting of the Expert Panels on Soil and Meteorology)
- the field sampling scheme was already discussed in meeting 12 (see minutes of the "Combined Meeting of Expert Panels on Soil and Meteorology"). It was assigned that minimum number of spatial replicates (profiles) should be 3 and in each profile sensors should be located in 3-4 depth intervals (0-20; 20-40, 40-80 cm and if thickness of forest floor was > 5 cm also in the forest floor).
- daily values required; it is recommended to measure at least every 6 hours (4 times a day)
- frequency of measurements must be reported
- calibration of sensors needed, but laborious → submission 2010 of not calibrated data (from 2009) + a specific mark; after calibration the corrected data will be resubmitted

## 5) Requirements on soil temperature measurements

- should be measured at the same depth as moisture measurement
- for new instrumentations PT100 standard is recommended
- sensors should be located near to WC sensors
- at least one sensor per plot and depth is required
- frequency of measurement should be at least the same as WC measurement
- Mean, Min, Max and frequency should be submitted

## 6) Requirements on stand precipitation measurement

- use of depo survey will be sufficient
- frequency of measurements should be at least every second week (biweekly)
- but, daily values are needed. Therefore daily stand precipitation must be calculated from weekly or biweekly sums
- calculated values must mark and methods reported
- number of samples should be reported

## 7) Revision of ICP Forests sub-manual VII Meteorological Measurements

- a Management of plots without meteo stations
  - Daily values are needed
  - could be modelled
  - For plots not included in D3 wind is not necessary
- b Data submission
  - In FutMon daily gapless values should be submitted
  - Higher time resolution has to be clarified later; is problematic due to data validation and gap filling procedures

## 8) Conclusions of the meeting and planning for the future

- a A table of references will be collected
  - Equipment of ICP Forests community should be listed to support beneficiaries in new instrumentations
  - Chairman will prepare a table and send it to all participants to fill in their experience with meteo and hydrological equipment
  - Deadline end of February
- b A soil moisture workshop will be organised
  - Soil moisture measurements and equipment
  - Soil physical measurements
  - Companies who sell equipment and external experts will join
  - Planned for end of March
  - Location will be Freising /Germany
- c To do list
  - "Field protocol" for meteorological and hydrological field measurements has to be established until March 2009
  - Table of references has to be established until end of February 2009
  - Soil moisture measurement workshop will be held in March 2009
  - Installation of new plots during 2009
  - Field measurements 2009 (new plots after instrumentation) and 2010
  - Submission of data from 2009 up to 15.09.2010
  - Submission of data from 2010 up to 15.09.2011

27.01.09

Stephan Raspe

#### European Union / United Nations Economic Commission for Europe International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects on Forests

#### Meeting of the Working Group QA/QC in Labs

January 13<sup>th</sup> 2009, Hamburg, Germany

#### Minutes (Combined FutMon and ICP-Forests Meeting)

#### 0. Introduction

Nils König welcomed the members of the Working Group in Hamburg. He introduced Anna Kowalska as co-chairperson (proposed at the Florence meeting in 2008, to be approved at the Task Force Meeting in St. Petersburg 2009) to the participants and presented the agenda of the meeting.

#### 1. Ring tests (water, soil, foliage, soil physics):

A proposed timetable for the ring tests has been revised.

Four ringtests are planned for 2009: soil, water, foliar, and soil physics. The participation in the ring tests is mandatory for all laboratories which are analysing samples within the FutMon project.

Responsibles for sample preparation and sending, data evaluation, and elaboration of the reports: A. Fürst (foliar ring test), B. de Vos and N. Cools (soil and soil physics ring tests), K. and J. Derome (sample preparation and sending water ring test), R. Mosello (evaluation and report water ring test) confirmed the preparedness. A data input module used in previous foliar ring tests has been installed and is ready to use in the forthcoming ring tests (except soil physics) as corroborated by A. Fürst.

A timetable for soil physics has been proposed by FSCC representatives and accepted. Sending water samples, originally planned for February 2009 had been moved to March by necessity to ascertain full list of laboratories that will perform analyses in frame of the FutMon project. vTl will mail next week (19-24.01.2009) a request to all beneficiary NFC's to send back as soon as possible all information about their laboratories. After completing the list of prospective participants by K. Derome and A. Fürst invitation will be sent at the beginning of February. Water testing laboratories will also get a reminder on complying with QA checks. The deadline for registration has been set at the end of February. For the next foliar ring test registration will be appointed at some future date. Also decision on data submission deadline in 2010 will be taken in future time.

The reports for foliar and soil ring tests are going to have the same content as previously. Data of water ring test will be elaborated according to ISO standard. All reports will contain the table with visual designation of performance of the lab for each parameter.

Draft reports with the results of the 4 ring tests will be sent to the participants in September 2009 and discussed at the Meeting of the Heads of the Labs to be held in Warsaw in October 2009. (see attached the minutes of an informal meeting for the preparation of the water ring test: annex 1)

#### Timetable ringtest 2009 (FutMon) and meetings

Month	1	2	3	4	5	6	7	8	9	10	11	12	Jan 2010
Soil			sending samples			results from Participants			evaluation of data, draft report			final report	

Water		sending samples		results from Participants			evaluation of data, draft report		final report	
Foliage	results from Participants	evaluation of data, draft report	final report			sending samples (next ringtest)				results from Participants (next test)
Soil physics		sending samples			results from Participants		evaluation of data, draft report			
WG QA/QC	Combined Meeting Hamburg									
Heads of the Labs								Meeting Warsaw: Discussion of the 4 ringtest results (yellow)		

#### 2. benchmark of the ring tests

Benchmarking of the ring tests has been discussed.

After the ring test each participant will receive a qualification report. The form of the report proposed by A. Fürst and N. König has been accepted to use in future ring tests. It has been decided to qualify the results of each parameter, if 50% or more of the results for this parameter for all the samples of the ring test are within the tolerable limits (listed in the quality check paper of the WG). Missing data (mandatory parameter not analysed) is a reason for the lack of qualification. High within-lab variation and high limit of quantification for a parameter will be remarked in the qualification report (see annex 2) and in future can be incorporated in the criteria for qualification.

The proposal for a requalification procedure has been accepted. It will consist in reanalysis of the ring test samples, report to the WG QA/QC with the new results together with the original reports of the instruments and information about weight factors, dilution factors etc. and information about the reasons for the unsatisfactory results during the ring test. Alternatively: assistance program for the lab with bad ring test results is launched; then reanalysis of the ring test samples, report to the WG QA/QC with the new results together with the original reports of the instruments and information about weight factors, dilution factors etc. and information about the reasons for the lab with bad ring test results together with the original reports of the instruments and information about weight factors, dilution factors etc. and information about the reasons for the bad results during the ring test. Requalification report is drawn up after positive decision of the persons in charge for the different ring tests (in case of doubt: WG QA/QC) about the report from the lab.

Stability of the samples used for reanalysis will be checked by the persons in charge for the preparation of the ring tests samples, in case of water samples also from a German (N. König) and an Italien (R. Mosello) laboratory.

#### 3. Opening of the lab codes

After meeting of the heads of the labs in Hamburg 2008 a letter to labs was sent to ask them to release their lab ID within this group. In case they do so, they can benefit from the information present in a technical info database on laboratory equipment, instruments etc. In case they do not, they won't have access to the codes of the other laboratories. *It is intended that this information will be posted on a password protected section of the ICP Forests website and will be made available to all participating laboratories, their heads and the WG QA/QC in Laboratories only. On the next Task Force Meeting in St. Petersburg in May 2009 WG QA/QC in Laboratories will inform all NFC's about decision of the laboratories for the codes. It was decided to abstain from a password protected section of the website for the codes.* 

registration procedure of the next ring tests by marking their agreement. List of released codes will be sent to the heads of the labs along with the report as a hardcopy.

#### 4. helping program for labs with bad ring test results

Assistance program for lab with bad ring test results will be continued within the frame of FutMon. In the final proposal of the project 10 visits of laboratories under C1 actions are possible. Currently one laboratory requested for help. First contact questionnaire has been sent; the WG group is still awaiting the response.

Assistance to the labs can be also given through free exchange of knowledge via google group WG QA/QC in Labs, established in 2008. Laboratories will be informed about this possibility as well.

#### 5. revision of the quality check paper to become submanual

In May 2008 a paper "Quality Assurance and Control in Laboratories - a review of possible quality checks and other forms of assistance" was published on the ICP-Forests web-page (<u>http://www.icp-forests.org/DocsQualLab/QualCheckMay2008.pdf</u>). The Quality Committee decided that this paper should be the basis of a new ICP Forests submanual "Quality Assurance and Control in Laboratories". Therefore the document needs some revision (e.g. restructuring, adding a chapter on quality indicators) to become submanual. It was decided that a small group (K. Derome, N. Cools, N. Clarke, T. Jakovljevic, P. O'Dea) under the leadership of A. Kowalska shall elaborate a proposal for the new submanual. It was decided to use actual version of the paper with some amendments as the draft field protocol for the FutMon project.

#### 6. discussion of the quality indicators

After a short discussion about the proposed quality indicators at the EPD meeting in Rovaniemi, Finland, three quality indicators have been chosen:

1. Percentage of the results of the ring tests within tolerable limits for each ring test.

Percentage of the results of the ring tests of repeatability below 10% (not for water ring tests).
 Mean percentage of parameters for which laboratories use control charts.

First two of them can be inferred from results of the ring tests. Third must be obtained from laboratories (as e.g. an answer submitted with the ring test results or from the quality report forms, see topic 9).

#### 7. Use of the quality checks in practice

Numerous quality checks for integrity of data are listed in the document "Quality Assurance and Control in Laboratories - a review of possible quality checks and other forms of assistance". Use of them is strongly recommended to labs to assure quality of data. The laboratories are in charge of the data quality.

A link between data and their quality must be maintained in the database.

The possibility of integrating of quality checks into data reports has been discussed. The information on data if passed or not ion balance might be integrated in a new form (quality form); that needs to be discussed with the database manager (see topic 9).

The reason of missing data, as e.g. small sample volume, contamination, may be also encoded and combined into data reports.

#### 8. Detection/Quantification limits

N.König presented the method of assessment the LOQ widely used in German laboratories, whereas the presentation by G. Tartari will possibly be shown at the next meeting of the heads of the labs.

It was decided to report and use only the quantification limit, not the detection limit.

Data below quantification limit are marked in database by "-1" and it will not be changed, but the value of method/matrix limit of quantification must be linked to these data.

Oliver Granke proposed to remove the section on missing data and values below LOQ from deposition, foliar and soil submanuals and gather them in the quality submanual with definitions and description of how to report these data.

#### 9. Discussion about the data submission formats with vTI (O. Granke)

Oliver Granke from vTI presented current construction of the database records. Additional parameters proposed by WG QA/QC can be easily integrated in the database, provided that existing units are not changed and number of digits is invariable with floating decimal point. New quality forms will be constructed, separately for deposition, foliar, soil and soil solution data.

The following information/quality parameters were proposed to be included in the new forms:

- beneficiary/country code
- year
- plot No
- LOQ for each parameter
- detection method (coded like in ring test reports) for each parameter,
- ring test No
- lab code
- ranking of lab in the ring test (% of results within tolerable limits)

- mean and standard deviation for each parameter from control charts

Data to the forms come from labs internal quality control and from ring test results; both types of information are available for the labs, therefore labs will fill in the new forms.

Data submission forms should combine both: mandatory and optional parameters. Database has not got any given data completness limits, if necessary, completness of results can be marked in extra added column in %.

O. Granke will elaborate the new forms and will circulate them among WG members for comments. Forms will be submitted to the Task Force Meeting in May 2009 for approval, then presented in October 2009 at the meeting of the heads of the labs.

A new working group "data management" (with data managers of the beneficiaries) was proposed. The WG will constitute if decision is reached at the meeting of data managers in September 2009.

#### 10. Second meeting of the heads of the labs (Warsaw, October 2009)

Anna Kowalska proposed 12.-13. or 19.-20. October for the meeting.

At the first meeting of the heads of the labs in Hamburg it was decided to ask participants for presentations about the following analytical problems:

- Problems with digestion methods for plant material (microwave and other systems)
- Problems with Aqua Regia digestion for soils (microwave and other systems)
- problems with ICP and AAS measurements in extracts and digested solutions
- problems with DOC and TN measurements in water samples
- comparison of results from different instruments

Results of the ring tests as well as consequences of non-qualified results, data submission, presentation of the google group WG QA/QC in Labs, and some analytical problems will be raised at the sessions.

The preliminary topics for the agenda has been accepted as below:

Торіс	Presentation(s)	Time needed
Report of the ringtest results (soil)	N.Cools	1:15
Report of the ringtest results (foliar)	A.Fuerst	1:00
Report of the ringtest results (water)	R.Mosello	1:15
Report of the ringtest results (soil physics)	N.Cools	0:45
Information about qualification/requalification	N.Koenig	0:20
Assistance program for labs	N.Koenig	0:10

Data submission: new form for quality information	O.Granke/A.Kowalska	0:30
Information about the opening of the lab code	A.Fuerst	0:05
Presentation and discussion of analytical problems proposed by the participants (see list above)		2:00
Detection/quantification limits – determination and use	G.Tartari	0:25
Problems with digestion methods for plant material	A.Fuerst	0:20
Problems with Aqua Regia digestion for soils	Participants	1:00
Google Group/web page	N.Koenig	0:30
FSCC reference sample	N.Cools	0:20
Other business		0:30

8-10 weeks before the meeting a letter will be sent to the possible participants by N. König with a request for presentations about analytical problems and proposals of other topics.

#### 11. Discussion about the maximum sample storage period

N. Cools outlined the requirements of ISO standard 18512 related to current practice in 20 countries/ 25 institutions. Most of the countries store samples for long-term monitoring, with a limited experience of change of properties with time. Minimum-maximum requirements for storing conditions should be set, controlled and reported (DAR-Q).

N. Koenig presented results of a few years tests of standard materials. Some measured values had been changing over the observed period. Some of the elements are more than other vulnerable to changes (e.g. exchangeable Mn, Fe and H, pH).

In foliar material changes although appear, but they are less frequent than in soils. Chemical composition of water is highly susceptible to alteration, especially at pH >4,5. Some information on the storage of water samples can be found in Analytical Info Sheets at: http://www.icp-forests.org/WGgual\_lab.htm.

Changes of chemistry of samples in time are not easy, if at all possible to avoid, therefore storage conditions have to be controlled and documented.

#### 12. Miscellaneous

a. A. Fuerst proposed new tolerable limits for low concentration (e.g. non-foliar litter, branches), basing on the results from last ring test. New limits are broader than for normal, higher concentration in foliage.

Element	Tolerable deviation from the mean (+%)	for concentrations below	
Sulphur	20	0.5 mg/g	
Phosphorous	15	0.5 mg/g	
Magnesium	15	0.5 mg/g	
Zinc	20	20 µg/g	
Manganese	20	20 µg/g	
Iron	30	20 µg/g	

Lead	40	0.5 µg/g
Boron	30	5 µg/g
Nitrogen	15	5 mg/g
Potassium	15	1 mg/g

b. Last foliage ring test (11<sup>th</sup>) first results will be published on the internet web-site on Monday, 19 Jan.

First evaluation revealed some analytical problems: results of C, Mg, Ca got worse and in sample containing branches. A digestion method problem might have occurred, as indicate the results of Fe and Cu.

c. The date of the next WG meeting will be decided bei vTI (combined meeting).

20.01.2009 A.Kowalska / N. König

annex 1:

#### Informal meeting Derome Kirsti, Nils Koenig, Rosario Mosello Hamburg, 14 January 2009

# Intercomparison exercises, with attention to the atmospheric deposition and soil water exercise

The intercomparison exercise dealing with atmospheric deposition and soil water, in the framework of the FutMon project, will be carried out following as strictly as possible the ISO/IEC rules, e.g.

#### IUPAC Technical report 1/2006

The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories

#### ILAC - G13:2007

ILAC guidelines for the Competence of Providers of Proficiency Testing Schemes ISO/IEC CD 17043:2008

Conformity Assessement – General requirements for proficiency testing.

The most important differences with the two previous intercomparisons (WRT 2002 and 2005) are:

1) the need to previously detail to the participants all the steps and methods involved in the intercomparison and

2) the lack of screening on the digitation and units of the submitted results (particular relevance for nitrate and alkalinity, often mailed with units different from those required).

Samples will be prepared from METLA (Derome), which will perform **preliminary analyses** to assure the needed and agreed range of concentrations and the **homogeneity test** among bottles.

**Stability of the solutions** will be tested for a period of six months from METLA, CNR ISE and Nordwestdeutsche Forstliche Versuchsanstalt. ICP Forests laboratories not directly participating in the FutMon project will be as well involved in the intercomparison.

With the help of vTI Hamburg a list of all FutMon labs will be collected until 6<sup>th</sup> Feb. 09 and then combined with the list of ICP Forests labs and others.

From this complete list Alfred Fürst will prepare 3 lists of the labs for the soil, water and plant ring test with the lab-codes and passwords for the registration. He will send these 3 lists to the persons in charge for the 3 ring tests.

Four documents will prepare and accompany the intercomparison exercises:

1) General invitation letter dealing with the three intercomparisons (soil, plant and water), written by Nils König (and Alfred Fürst).

2) Detailed letters specific for each of the three intercomparisons from the three persons in charge for water, soil and leaves tests. In the letter information must be given about lab codes and passwords for the pre-registration, using the software prepared by Fürst.

3) Detailed letters to the registrated laboratories specific for each of the three intercomparisons from the three persons in charge for water, soil and plant tests, giving all the technical information on the process and methods used and warnings for the correct submission of data (e.g. attention to the units, any correction of the results forbidden, how to mail the results, etc). Minute to Alfred, Nils, Kirsti, John, Bruno, Nathalie.

4) Accompanying letter of each type of intercomparison, giving details on the range of expected concentration, timing for the analyses and other practical details concerning the analyses and the mailing of data. (Prepared by the person/institute in charge of the preparation and mailing of the results).

Copies of these letters should be available both in the Fürst's web page and in the ICP Forests QA/QC web page, where a specific space for the intercomparisons should be present, to disseminate the different documents, including expected results, draft and final reports, etc. (König).

The persons/labs in charge of the data elaboration will prepare, together with the summary of results and the draft report, a "Qualification report" for each laboratory, where a summary of the performances will be published. The report will document, for each parameter, if analysed or not, if passed to the test, and notes (see annex). Not analysed mandatory variables will be considered as "not passed". This qualification report will be mailed as well to the National Focal Centres.

Laboratories can submit to the person in charge of the intercomparison exercise a "Requalification report", where he documents the reason of the error(s) and the improvements done to make reliable the considered analysis. If accepted the re-qualification will be documented and mailed to the laboratory and NFC. For atmospheric deposition/soil water this will be done by CNR-ISE with the help of METLA and Nordwestdeutsche Forstliche Versuchsanstalt.

Draft report of the results will be circulated before the meeting of the head of laboratories in Warsaw (mid October), results will be presented and discussed in the meeting, and the points emerging from the discussion will be considered for the preparation of the final report.

FUTMON ICP FORESTS COMBINED EXPERT MEETING HAMBURG JANUARY 2009 Foliar and Litterfall Expert Meeting 15-16 January 2009

FutMon Project Action D2: Nutrient Cycling & Critical Loads Minutes

24 participants from 18 countries attended the meeting (Annex 1).

## First meeting day (Jan 15th 2009)

1. Chairman (A. Fürst) opened the meeting

- 2. Item 1: The meeting adopted the attached agenda (Annex 2).
- 3. Item 2: Chairman introduced briefly the Expert panels last year activity:
- 10th needle/leaf interlaboratory comparison test was finalised and published

• FutMon was accepted  $\mathcal{G}$  efforts for the quality improvement have some financial support during 2009-2010

4. Item 3 and 4: New information needs due to FutMon and manual updates.

The Chairman inform about the status of the manual updates. The normal update based on the guidelines from the QA Group (Marco Ferretti) is ongoing. No special changes are planned here only the analytical part should be moved to a separate laboratory manual. This updates should be finalized in the manual revision in 2010.

To react on the needs of the FutMon project (D2 "nutrient cycle") same changes are urgent especially for the new survey ground vegetation. We had to start with this survey 2009 to have real data end of 2010 and we need it then because of the needs of the D2 action. These changes should be made in so called "FutMon field protocols". The following timetable was given from VTI to the chairs of the Expert Panel:

- First draft end of January 2009
- Circulated 2th Febuary 2009
- Comments should be given till 13th Febuary 2009
- Second draft (including data forms) should be send to VTI till 27th Febuary 2009
- Final editing until 20th March 2009
- Final Manual should be adopted in the Task Force Meeting 2010
- Changes in the sub manual 4 (Sampling and analysis of needles and leaves) were adopted:

• Not only needle set 1 and 2 should be analyzed also older needle sets should be analyzed

• Each D2 partner can choose to pool the sample of the older needle sets or analysed them seperatly (only update in the field protokoll for FutMon not needed in the ICPManual) Minutes of the FutMon ICP Forests Combined Foliar and Litterfall Expert Meeting Hamburg January 2009

2

• Some smaller updates of the submission forms needed (drop out ringtest number, labcode) – they are not longer needed, because of the new QA/QC forms. Add code for the "leave\_type" of the "pooled sample".

For the "**Field protocol Sampling of ground vegetation**" the Draft which was send from Pasi Rautio mid of December 2008 was discussed.

The following comments were made by the participants:

Switzerland (Waldner): Different height between litterfall samplers and ground vegetation

sampling height. Could be a problem? Maybe harmonisation needed?

Italy (Matteucci, Petriccione): Because of the costs the sampling should be reduced to one or maximum two visits to the plot (Panel agree with this).

Take care sampling close to established features (e.g. litterfall traps); sampling personnel could have disturbed the ground vegetation.

Belgium (Roskams): For information on nutrient cycling processes we should separate woody parts from leaves for the analyses.

Germany (Dietrich): Exclude woody tree species higher than 0.5m from analysis

Germany (Dietrich): Will send information on a model for nutrient cycling and biomass calculation to Foliar EP group

Germany (Dietrich): Sampling design - Germany open to both random sampling and fixed sampling?

Italy (Petriccione): Only random sampling should be used (8 frames of 0.25m2 or 4 frames of 0.5 m2).

Panel agree with this: Random sampling should be done outside of the IM plot area, should be ten to twenty meters from the ground vegetation sample plot but as close as possible. The ground vegetation selected for the sample area should be similar to the plot.

Estonia (Laas): In case of not enough biomass could it possible to set a lower limit were we need no special analysis?

Panel agree with this: If there is not enough sample material for analysing after pooling No significant ground vegetation community.

Pooling, if required, should be done for those groups where the weight is not enough for chemical analyses, the other groups should be analysed separately.

Italy (Petriccione): Functional Group Grasses (sedges and rush), should be expanded to include the Graminae family and others.

Panel agree with this: Exclude wooden tree species above 0.5m from nutrient analyses Austria (Fürst): a maximum 70°C, for at least 48 hours harmonized to a maximum 80°C and 24 hours - Element concentrations and biomass should be reported as dry matter (105°C) for foliage, ground vegetation and litterfall.

## Minutes of the FutMon ICP Forests Combined Foliar and Litterfall Expert Meeting Hamburg January 2009

Austria (Fürst): Ground vegetation and Litterfall will be included in the Foliage Ringtest -Who is willing to produce a Ringtest sample? Email details to: alfred.fuerst@bfw.gv.at The Foliar Expert Panel agreed to supply the Expert Panel on Ground Vegetation and Forest Biodiversity with a draft copy of the ground vegetation field sampling protocol for comments respecting the deadlines outlined.

## Sampling and analysis of litterfall

Decomposition studies were made in Finland, Belgium and Switzerland. The panel agree to discuss the addendum to the litterfall manual in more detail till 2010 in the litterfall group. Priority is the creation of a field protocol and/or a manual update for sampling of litterfall in FutMon.

For this the following adaptation of the existing manual is needed:

Panel agree with this: Different litterfall collection traps are used in the countries. It is not possible to change this because each type has advantages and disadvantages. What is needed is information about the sampler type, sampler height and sample surface in m<sub>2</sub> and the number of samplers/plot. This information has to be included in the forms.

The manual point "Frequency of sampling" the following change should be made (Germany-

Dietrich): "It is recommended to collect litterfall bi-weekly or at least monthly."

Panel agree with this: A more frequent sampling (maybe needed for Phenology purpose) is not practical because of additional costs.

Panel agree with this: To fulfil the necessity of the D1 action litterfall on beech and oak plots will be separated, weighted and analyzed in different fractions:

- foliage
- wood (bark, branches, twigs)
- seeds & fruit,
- capsules,
- others

Separations in more fractions e.g. leaves of different tree species are possible.

The point why seeds & fruits should be separated from capsules for D1 should be discussed in the plenary session.

On all other plots the separation and analyse of pooled samples for foliage and non-foliage litter is enough.

For the Leaf Area measurement a camera system with fisheye could be used. It was discussed in the pre meeting with the chairs to buy an instrument and share it between the FutMon partners (R. Fischer) if the method is used. There will be a meeting Mai 2009 (Slovenia) about LAI measurements and methods. It was planned to set up a separate manual about LAI measurement and method(s). In this case the part for LAI measurement should be moved from the litterfall manual to this new manual.

Minutes of the FutMon ICP Forests Combined Foliar and Litterfall Expert Meeting Hamburg January 2009

## 4

Leaf area index calculation from litterfall samples were made in the past only from France (only for broadleaves) from Swiss and from Italy.

Panel agree with this: For the harmonization between the manual for sampling of needles and leaves and the litterfall manual the following points had to be changed:

• Na and Al will be deleted from the optional list

• Cd will be added in the optional list

- The units for Carbon will be changed to g/100g in the form
- $\bullet$  The units for Fe, Mn, Zn, Cu, Pb, B will be changed to  $\mu g/g$

• The unit for Cd is ng/g

One missing compartment for the D2 action is litterfall from ground vegetation. Swiss (Waldner) has experience with it, but the study was not published yet. They extrapolate the results of ground vegetation sampling from deciduous species.

5. Item 5: Quality issues.

The chairman gave an overview about the activities in the QA/QC-WG for labs.

Harmonisations between the ringtests in the different analytical groups were done.

- The same Webinterface (hosted by FFCC/BFW) will be used in all tests
- Use of tolerable limits (for low/high concentration) for the test evaluation
- Including QA/QC information as a separate table in the database
- In future there will be no feedback to the laboratories about wrong units in ringtests before ending the deadline
- Participants/NFCs/FutMon beneficiaries get a qualification report
- NFCs are responsible for data quality of the lab
- All labcodes will be opened for participants, ICP-Forests, EP, WG-QA/QC

The proposal for tolerable limits for low concentrations (e.g. non foliage litter) was adopted

from the Expert Panel.

The first results of the 11<sup>th</sup> Needle and Leaf Interlaboratory comparison were presented (Download from the FFCC webpage www.ffcc.at will available on the 19<sup>th</sup> of January 2009). One non foliar litterfall sample (pine branches) and three foliage samples (Pine, Maple, Spruce) were analysed. The results for nitrogen and carbon compared with the last test are getting worse (e.g. calibration problems with element analysers). Iron and Copper were also not so good. Better results - compared with the last test - for zinc, lead and cadmium were achieved.

For future ringtests FFCC needs test samples especially for ground vegetation and non foliage litterfall.

For (two) FutMon foliage laboratories a laboratory helping programme is possible – contact person for this programme is Nils König.

Minutes of the FutMon ICP Forests Combined Foliar and Litterfall Expert Meeting Hamburg January 2009

#### 5

The WG QA-QC for labs set up a Manual about Quality assurance and control in Laboratories (available on the ICP-Forests web page). In this manual are plausible ranges for foliage of different tree species (calculated from the level II database) available. Also for foliar litter there are some plausible ranges given (www.icp-forests.org webpage for WG QA/QC in labs).

## Second meeting day (Jan 16th 2009)

6. Item 6: Any other business:

List of the Foliage and Litterfall experts was updated.

Negotiations for a common scientific workshop with IUFRO (Turkey 2009) it was decided not to have a common meeting at this point. Nenad Potocic was a contact person between ICP EPs and IUFRO and gave here a short overview.

The panel agree to have: Back to back sessions in the next FutMon/ICP-Forests Combined Expert Meeting.

The minutes of the meeting were adopted.

Chairman A. Fürst closed the meeting

Expert Panel wants to warmly thank the organisers of the meeting, Martin Lorenz, Richard Fischer and Astrid Khalil in vTI.

Annex 1 (List of participants)

Annex 2 (Agenda of the meeting)

Minutes of the FutMon ICP Forests Combined Foliar and Litterfall Expert Meeting Hamburg January 2009

#### 6

#### Annex 1 (List of participants)

Fürst Alfred (Austria) Dietrich Hans-Peter (Germany) Jonard Mathieu (Belgium) Roskams Peter (Belgium) Potocic Nenard (Croatia) Lomsky Bohumir (Czech Republic) Sverrild Karen (Denmark) Ingerslev Morton (Denmark) Laas Indrek (Estonia) Ukonmaanaho Liisa (Finland) Merilä Päivi (Finland) Croisé Luc (France) Neville Pat (Ireland) Matteucci Giorgio (Italy) Fabbio Gianfranco (Italy) Bertini Giada (Italy) Petriccione Bruno (Italy) Timmermann Volkmar (Norway) Oneata Marian (Romania) Priwitzer Tibor (Slovakia) Simoncic Primoz (Slovenia) Garcia Paloma (Spain) Waldner Peter (Switzerland) Barsoum Nadia (UK) Minutes of the FutMon ICP Forests Combined Foliar and Litterfall Expert Meeting Hamburg January 2009 7

#### Annex 2 (Agenda of the meeting)

1) Opening, welcome and acceptance of the agenda

2) New information needs due to FutMon and corresponding demands for the EP and manuals. Other manual updates (Sampling from Needle and Leaves/Sampling of ground vegetation).

3) Discussion about the possible litterfall projects and manual amendments

4) Quality issues including ring-test results and coming ring-test

- 5) Any other business
- 6) Closing of the meeting

Protocol	Protocol-No.	otocol-No. Project Date				Bavaria n Forest Institute	LNF
	01/2009	_	MON Action D3-Soil sture workshop	20.03.2	009	motitute	
Workshop			·	L		Location	
See workshop	o programme					/1	KG-hall
						10:00	/25/2009
						End	
Chairman	Telefone	No.	Protocol	Telefoneo	Nr.	Invitation a by	and programme
Raspe	+49-81 71-492	-	Zimmermann	+49-81 71-491	-	Mail Ra 03/??/0	
Participants					Additi	onal mailing	list
Workshop participa	ants						
Progamme							
TOP 1: Talks Bulten TOP 2: Talk Nicolas TOP 3: Talk Keller TOP 4: Talk Campbe TOP 5: Talk Cools TOP 6: Talk v. Unold TOP 7: Talk Liu TOP 8: Talk Wessel- TOP 9: Talk Campbe TOP 9: Talk Campbe TOP 9: Talk Campbe TOP 9: Talk Renth TOP 10: Talk Ruth TOP 11: Talk Ruth TOP 12: Talk Sramel TOP 13: Talk Verme TOP 14: Talk Sverril TOP 15: Closing disc	l Bothe ll er e c iren d					Hint / Acti	
Result						Hint / Action	on
<ul> <li><b>TOP 1:</b></li> <li><i>Bulten: Field set of taking undisturbed soil samples,</i></li> <li><u>Discussion:</u></li> <li>Still difficult to take forest soils with high gravel content and roots present with this equipment, problem to take samples from dry soil, which should be taken in</li> </ul>							
a wet situation, recommendation starting from the can be cut at the c the soil core into							

#### disturbance

Bulten: pF values and measurement

#### Discussion:

Hydraulic contact with the soil core on the sand as well as the kaolin boxes is important (surface of the sand or kaolin is flexible and adapts to the soil core), measurement of pF=0 should be done with a pcynometer to get total porosity, according to Bulten it has to be done on a separate soil core since the soil core has to be dry to avoid interaction of humidity from the soil core with the mercury of the pycnometer, other experience from the university of Freiburg where field fresh samples were used in the pycnometer, weighing a completely saturated soil cores seems to be impossible

#### **TOP 2:**

Nicolas: pF measurements

#### Discussion:

Hydraulic contact of the soil cores on the ceramic pressure plates maintained by kaolin cover

Small soil cores are a trade-off between easier soil sampling and

representaiveness of the soil, larger soil cores (100 cm<sup>3</sup> or 250 cm<sup>3</sup> seem to be more representative),

Comparison between suction tables and pressure tables not yet performed, is going to be made in FUTMON

Open questions: How many profiles and repetitions per horizon are needed? How to handle organic layers in the sampling for soil physical measurements?

## **TOP 3:**

Keller: Hyprop system

#### Discussion:

Special tensiometers T5 with special ceramics with a boiling point above 8 bar, practial limit for measurements 3 bar, depending on the soil it takes 3-5 days to get all values, in addition time for saturating the soil core Problem to get micro tensiometers into a sample with gravel content, disturbance of the undisturbed soil core by the model: soil core is won with adapted mask taking into account the positions of the micro tensiometers Improvement of the method to consider an independent value of pF 4.2, but software yet cannot include independent values from the dry range for the fitting of the pF curve according to the different models Number of replicates depends on soil (recommendation 5-20) Comparison to suction plates see publications of Schindler or Durner

## **TOP 4:**

Campbell: WP4 system

#### Discussion:

Packing of the disturbed soil sample impacted by the individual operator, Reproducible?, up to .1 or .2 g/cm<sup>3</sup> to the original density possible

Very fast procedure (2 days) compared to suction plates (weeks)

#### **TOP 5:**

Cools: FUTMON protocol

#### Discussion:

The ring test should also include alternative methods from labs where available since planning of the FUTMON often includes these methods instead of ISO-methods

Additional set of soil cores needed

Pycnometer method for determining total porosity not included in ISO and yet not in field protocol

Soil sampling on the plots can be performed starting after the ring test in fall 2009

Time of storage of ring samples limited since biocides often contain tensides which change hydraulic properties, plants can even germinate at low storage temperatures (diverting experience here!)

Quality of the water concerning surface tension of water should be also be measured as well as temperature

#### **TOP 6:**

v.Unold: Tensiometers

#### Discussion:

Effect of salinity on the self-refilling tensiometer: due to the small pore size of the tensiometer salt ions will be excluded

For the self-refilling tensiometer the soil water is not de-gassed when it enters tensiometer

"Noise" of the signal of the pF-meter?

#### **TOP 7:**

Liu: Equitensiometer

#### Discussion:

Sodium concentration should be not too high since it affects electronics, eventually problem for salty soils in drier environment Different off-sets after a drying cycle

**TOP 8:** *Wessel-Bothe: pF-Meter* 

#### Discussion:

Contrasting comparison between pF-meter and tensiometer under different conditions lab/field(v.Unold, Brando), probably due to different consideration of hysteresis during the calibration process Neutral institution for testing?

#### **TOP 9:**

Campbell: Echo Sensors for matric potential, water content and soil temperature

#### Discussion:

If proper hydraulic contact is made for the MPS-1 sensor e.g. with homogenized soil then it makes more sense to measure soil matric potential instead of soil volumetric water content, since the latter is more prone in gravel-rich soils to introduce errors due to little contact with the surrounding soil Life span of ECHO-Probes is 3-5 years, more expensive sensors like soil moisture have the advantage that they have a longer time span due to the fact that no electonic parts are buried in the soil (up to 10 years)

#### **TOP 10:**

Pannatier: Calibration of EC-5 soil moisture probe

#### Discussion:

Deviation of the calibration curves from lab measured soils from the calibration relationship of Decagon, shift of 10 vol.% from the Decagon relation, might be dependent on dry bulk density (fit for alluvial soils), time consuming individual calibration process might be substituted by finding a relation towards dry bulk density since the gradients of the calibration curve were the same as for the Decagon relation

**TOP 11:** *Derome: Delta-T Profile Probe – PR2/6* 

#### Discussion:

How many depths? Constant depth steps versus genetic horizons Three mandatory depths in the protocol: 0-20, 20-40, 40-60 cm

Precision of the profile probe, since there is no direct contact to the soil? Information on Delta-T Website

## TOP 12:

Ruth: water content sensor with a flat sensitive sensor

## Discussion:

No commercial version of the sensor available, eventually interesting for measuring soil water content in the upper organic layer; problem of swelling and skrinking in the organic layer for all sensors! Difficulty to get the soil in close contact to the sensor due to large number of rods

## **TOP 13:**

Sramek: Gypsum block sensors and Campbell CS616 for soil volumetric content

#### Discussion:

Variation of the soil moisture content not only caused by the soil but also by the forest type.....

#### **TOP 14:**

Vermeiren: water balance experience in Flanders, usage of WatBal model

#### Discussion:

WatBal calculates total ET, no ET components

Manual readings of the Tektronix cable tester for TDR-measurements subjective, new methods?

Climatic data from national weather service will be used for FUTMON since dense network exists, no complex terrain in Belgium

#### **TOP 15:**

Sverrild: Soil water measuring and modelling with CoupModel in Denmark

#### Discussion:

Manual monthly event-related readings at various locations in Denmark, Cable tester profile for measuring soil moisture, type: Campbell, problem of contact to soil while measuring

#### **TOP 16:**

Closing discussion

Sampling for pF curve (mandatory if organic layers > 5 cm organic sample, sampling windows 0-20 cm, 20-40 cm, 40-80 cm) and soil moisture measuring depths should be the same, ranges are given to ensure that genetic horizons or other soil physical properties can be considered (special importance of Ahorizon for evapotranspiration), but also that texture information from BIOSOIL can be used, report the exact depth, zero plane is the upper border of the upper mineral horizon, minimum: 1 probe/depth range, importance for derivation of pedotransfer functions as well as gaining direct soil hydraulic parameters for water budget modelling Repetition for small-scale variability (0-20 cm: 3 replicates, 20-40 cm: 3 replicates, 40-80 cm: 3 replicates) Recommendation to take into account the canopy structure, plots in the middle between trees as well as the periphery (where possible) Frequency of soil moisture measurements: minimum requirement that when a

wider timescale than daily values are used a sufficient long, representative time series covering different moisture conditions could be used instead, strongly recommended to use daily values (Special case Denmark)

Calibration of sensors needed, but laborious, submission of not calibrated data + mark,

Technical recommendation of how to install the TDR-probes in an annex of the field protocol, contact to experienced installers of soil moisture equipment (Grimmeisen, Unold, UGT etc)

Further discussion by the Google group or mail

#### LIFE+ 2007, FUTMON

## Further Development and Implementation of an EU-level Forest Monitoring System Combined Field Course on Phenology and LAI

Actions: D1, D2, D3, C1-Tree-30(NWD), C1-Phen-10(FI), C1-Met-29(BY)

#### Minutes

Tuesday, May 5

The workshop was opened by Egbert Beuker.

Dr. Mirko Medved, director of the Slovenian Forest Institute welcomed the participants to Slovenia and Lipica, and wished all a success full workshop. Mr. Zivan Veselic presented an introduction to Slovenian forestry. Slovenia is one of the most densely forested countries in Europe.

The workshop started with the phenology assessments. All participants were provided with a memory stick that contained all the background material on phenology.

First a short introduction to the draft manual was given, focusing on the major points that differ from the current ICP Forests manual Chapter 9: "Phenological Observations". On request from the database the coding for the stage of the events was changed so that code 0 will no longer be used. Subsequently a short introduction to the use of digital cameras was given. The advantages of the use of cameras were presented, but also points of consideration were given. These will also be included into the field protocol. Cameras should be used when it is difficult or impossible to make the observations manually. It was decided that a minimum requirement for the quality of the pictures (minimum number of pixels) shall be included into the field protocol. Technical requirements for the cameras will not be given.

Life pictures from an on-line camera in Punkaharju, Finland were shown.

Pictures of autumn colouring and leaf fall in birch where shown and the analyses of the pictures was discussed. Subsequently participants of the course made themselves analyses of a series of pictures about autumn phenology in birch. It was experienced that it is not easy to assess pictures when one is not familiar with the species and with the history of the stand during the growing period. Analyzing pictures requires about the same training as making phenology observations in the field.

Concerning the assessment of damages it was decided that damage will be assessed, but submitted with the phenology data only on a no or yes basis. In case damage occurs further assessment of the damage should be made and submitted using the Damage forms following the guidelines of the ICP Forests manual chapter 2"Visual Assessment of the Crown Condition", Annex 2 "Assessment of damage causes".

The second item of the day was the leaf area index (LAI) measurements within the FutMon actions D1, D2 and D3. First the objectives of this part of the workshop were defined. The requirements of the several D actions on LAI measurements should be clarified. A second aim was to give an overview about existing methods for LAI measurement and a third point was to compare different methods and making first experiences during a field course. The requirements on LAI measurements were presented by Inge Dammann for D1, by John Derome for D2 and Stephan Raspe for D3. The following table gives a summary of their talks.

Definition total one-sided foliage area per unit ground surface area (Chen and Black 1991) Objectives Develop a new tree vitality parameter comparable with defoliation only a Supplementary role in estimating leaf biomass for nutrient cycles

• Parameterisation of water budget models (interception, transpiration, soil evaporation)

• Improvement of transfer functions

Resolution • relation to crown condition sites

- 40 scores /site
- annual values
- vegetation & non vegetation period
- A general value for the plot; no annual variation needed
- representative for plot(&species)
- min & max of the year
- annual values?

After this introductions into the different methods are given by experts:

Matjaz Cater: Light environment measurements and basic evaluation of images. it was a good introduction to indirect methods for light measurements in forests and their different results. He explained the general functioning of canopy analyzers like Li-Cor LAI2000 and of canopy analysis systems based on hemispherical image analysis (WinScanopy). Both systems produce a set of outcomes from which the LAI is probably the most difficult one. Therefore, he suggested to use better the "transparency" or "gap fraction" as an index for tree vitality than the LAI. Michael Leuchner: Methods for the indirect determination of the Leaf Area Index (LAI) in

forest canopies - LI-COR LAI-2000 Plant Canopy Analyzer. The introduction was clear and broad with a summary of advantages and disadvantages of the

LI-COR LAI-2000 system. Advantages are:

- fast measurements
- easy to handle (measurements and data handling)
- good repeatability
- good for determining relative changes in LAI (seasonal / interannual) Disadvantages are:

• stand has to be characterized well for good absolute values (clumping, woody area etc.)

- meteorological limitations (never perfect conditions)
- necessity of a clearing in proximity of the measurements
- several corrections add to total uncertainty

Christian Hertel: Methods for the indirect determination of the Leaf Area Index (LAI) in forest canopies - Hemispherical Images for LAI analysis (WinScanopy). The introduction was clear and broad with a summary of advantages and

disadvantages of the WinScanopy system. Advantages are:

- Visually useable dataset
- More than LAI-data (openess, PPFD, gap fraction, suntrack, clumping etc.)
- Useable on clear sky and overcast sky-conditions
- Disadvantages are:
- intensive postprocessing needed (no instantaneous read-out possible)
- photographical expertise necessary
- meteorological limitations
- with use of manual mode: subjective

John Derome: Planar mosaic photos.

Similar to hemispherical photography this techniques use a mosaic of "normal" digital photos. The evaluation is done by a specific black and white analysis soft ware. In Finland this method was used for all Biosoil plots.

Marius Teodosiu: The Trac-System.

The Trac-System is a method which is again a light intensity measurement system.

The big advantage of this system is simultaneous measurement of a correction factor needed for e.g. LI-COR LAI2000 method.

## Wednesday, May 6

During the second day of the workshop an excursion was organized to look at and practice spring phenology observations and different methods for LAI measurements. Due to the warm weather during spring unfortunately most of the spring phenology of forest trees in the region was already more or less completed. However, at a Black pine stand near Sezana there was a good discussion about the defining of flushing in pine. According to the manual flushing occurs when the separate green needles become visible. However, in black pine the needles are not yet clearly green when they become visible.

Also flowering of black pine was demonstrated. At a second stand of black pine the occurrence of damage was demonstrated.

After lunch Stephan Raspe demonstrated the use of a Speed Dome camera for phenology observations. The camera was located at the ground and looks with an optical zoom (36 fold) into the crown of the trees, similar to manual observations with binoculars.

At about 1100 m asl. an European beech stand was found with all the different stages of flushing still occurring. In this stand 15 trees were selected and the participants were asked to make the observations on flushing. After this the whole group discussed the flushing stage of each of the 15 trees. Again it became obvious how important proper training for the field staff is. A major outcome of the discussion was that the scoring classes for phenology will be revised in such way that 0% and 100% will be replaced by "less than 1%" and "more than 99%", respectively.

During the field course LAI measurements with different methods were demonstrated at two different sites (one in a pine stand and another in a beech stand). LI-COR LAI 2000 was demonstrated by Michael Leuchner. The handling of this system could be tested by all participants.

Problems with adequate weather conditions and suitable open field conditions for reference measurements were shown and discussed. The TRAC system was shown by Marius Teodosiu and some measurements were done. Hemispherical photography was demonstrated by Matjaz Cater and Christian Hertel (WinScanopy) and Martin Greve (simple fish eye photography and free ware evaluation). Differences between the methods were discussed and some photos were taken. The day was closed with a social diner at a nice vineyard near Vipava.

#### Thursday, May 7

First the results of the field exercise with the flushing of beech (attached) were presented and discussed. In cooperation with Volker Mues (the FutMon data centre) the final draft of the Field protocol was prepared including the submission forms according to the results of the discussions of the former days. It was also decided that pictures from automatic cameras will have to be send to the FutMon data centre. Guidelines will be presented soon. It was also decided that the intensity of flowering can be assessed as an optional parameter. The forms will be adjusted accordingly.

Concerning QA the need for sufficient training of the field staff was stressed once again. Control of the observations made manually by field staff is almost impossible.

In the second part of the day LAI evaluation and field protocol for LAI measurements were discussed. First the evaluation of data taken during the field course on Wednesday was demonstrated by the experts. Here the differences between the methods became obvious and the following requirements on data submission were discussed. This results in a first draft of a field protocol for LAI measurements, which was prepared during the night before by a small group (Volker Mues, Matjaz Cater, Inge Dammann, John Derome, Martin Greve, Michael

Leuchner, Christian Hertel, Stephan Raspe). Volker Mues will finish this draft before the St. Peterburg meeting with some assistance of the experts. The workshop was closed by Stephan Raspe with special thanks to the external experts Matjaz Cater, Martin Greve, Michael Leuchner and Christian Hertel).



## Expert Panel Biodiversity and Ground Vegetation

## Roma (Italy) - Wednesday, 22 April 2009

## 9 DRAFT MINUTES

The meeting was opened by the co-Chairman of the Expert Panel, Bruno Petriccione who also welcomed the participants to Rome. Pat Neville invited the participants to adopt the agenda, where the timing of some items was amended to suit departure times of participants.

Pat Neville invited the Expert Panel to confirm the joint chairmanship of himself and Bruno Petriccione for the Expert Panel which was duly ratified.

Bruno Petriccione invited the countries to outline their proposed approach to vegetation monitoring as required under LIFE+ project FutMon . Each country outlined their intentions accordingly. Turkey made a short presentation on the establishment of a monitoring network in their country.

The Expert Panel recommended, as far as possible, that countries should attempt to conduct their survey work under FutMon during 2009. However, it was also recognized that future opportunities to streamline and fully harmonise vegetation sampling assessment might be captured under future phases of the FutMon project. The issue of increasing sampling frequency versus the importance of sampling synchronization was discussed in detail. It was recommended to connect sampling frequency with the main drivers of biodiversity change in European forests.

Pat Neville invited the Expert Panel to consider the sampling protocol proposed for nutrient sampling of the ground vegetation community by the Expert Panel on Foliage. A great deal of concern was expressed from the Panel regarding the proposed sampling approach which removes the upper layer of vegetation. This is highly problematic in terms of secondary succession, aggravation of the seed bank and the introduction and spread of invasive species; all of which may compromise the results of future vegetation surveys. It was recommended that such sampling occur outside of the plot, in areas representative of the plot but no closer than 10m from the buffer zone. Estimates of biomass measured in a given species could be expanded to plot level through the use of the vegetation data set, which gives cover abundance estimates for all species. The height of the species is also important for biomass estimates and it was recommended that the height of the 5 most abundant vascular species be recorded in the sampling frame. This could be supported through the introduction of broad classes of visual height estimates from the common sampling area of 400m<sup>2</sup>. Finally, photos of the sampling frames make a useful record of the vegetation present.

The species code list was discussed in detail. At present seven different list exist, but one final Excel based list is nearly available through the efforts of Roberto Canullo, Franz Starlinger and Oliver Granke. It was agreed that new species or species codes could be sent bilaterally to Franz Starlinger. The Panel will look into the implementation of one species list.

The addition of a biodiversity component to the ICP Forests manual was discussed. As the ForestBiota project is now fully completed and recently published it was agreed to adopt this approach for a manual most suited for intensively monitored plots. Following the investigation of the results of the BioSoil project it is envisaged to adopt this approach as a manual for extensive networks of plots.

The ground vegetation manual needs to be amended to include elements of quality control and quality assurance. Rather than rush this revision ahead of the Task Force 2009, it was agreed to revise the manual after the field exercise on quality control planned in NE Alps (Italy) for July 2009 and incorporate the findings. A questionnaire has been prepared by the Italian team (CONECOFOR), as implementation of FutMon Action C1-GV-15IT, to demonstrate sources of variability and potential errors and recommend best practice and inform changes to the manual. This questionnaire, adopted by the Panel, will be circulated between all FutMon partners and ICP Forests NFCs, to be filled by the end of May, 2009.

Details of the trans-national training and intercalibration course were presented by the Italian team (CONECOFOR), who outlined the importance of this as a first approach to achieve a level of harmonization across the countries. The approach of the training course was given in detail. One major aim will be to adopt a common assessment method to be used on at least a sub-set of plots in addition to the national assessments.

Bruno Petriccione outlined the response from the Joint Research Centre of the European Commission to the evaluation of the BioSoil project results. A meeting on this topic is foreseen for the Autumn of 2009. Bruno then informed the Panel of some other closely related initiatives such as the LIFE + Project FACTS, INFORDIV a new COST Action project and LifeWatch. The joint chairmen, Bruno and Pat then thanked the participants for a productive meeting and declared the meeting closed.

**The EP chairmen** Pat Neville Bruno Petriccione

Sprejeti protokoli projekta FutMon Life+ do vključno 27. maja 2009 so:

- a) FutMon QA/QC Guide for Laboratory Work (1st version) -<u>QualLabs v4.pdf</u>
- b) Field protocol on continuous measures of forest growth, Action Group D1: Tree vitality and adaptation - <u>field prot growth V1\_1270509.pdf</u> (UPDATE!)
- c) FUTMON FIELD PROTOCOL PHENOLOGY (D1), V1.1; last update 19th May 2009 - field\_prot\_pheno\_V1\_1\_19May09.pdf (UPDATE!)
- d) Field protocol on permanent and continuous measures of forest growth Expert Panel Crown Condition and Assessment Damage Causes, Tree Vitality (D1), FutMon Field Protocol - field\_prot\_Vitality\_V1\_150509.pdf
- e) Expert Panel Crown Condition and Assessment Damage Causes Tree Vitality (D1) FutMon Field Protocol - **field\_prot\_Vitality\_V1\_150509.pdf**
- f) FutMon (Life+) field protocol: Sampling procedure for evaluation of nutrient budgets in vegetation in FutMon intensive monitoring plots and more intensive foliage surveys (D2), V 1.0; last update 15th May 2009 field\_prot\_biomass\_foliage\_V1\_150509.pdf
- g) Litterfall sampling and analysis, FutMon (Life+) Field Protocol 2009V1; last update 14th May 2009, IM1 recommended, mandatory on D1 and D2 Demonstration Project plots -field\_prot\_litter\_V1\_150509.pdf
- h) Field\_prot\_pheno\_V1\_15May09.doc page 1/6 1 FUTMON FIELD PROTOCOL PHENOLOGY (D1), V1.0; last update 15th May 2009 field\_prot\_pheno\_V1\_15May09.pdf
- i) field\_prot\_SoilWater\_v1\_150509.doc page 1 / 12FutMon Field Protocol, Determination of the soil water retention characteristic field\_prot\_SoilWater\_v1\_150509.pdf, V 1.0; last update 15th May 2009

Life+ / Further Development and Implementation of an EU-level Forest Monitoring System (FutMon) + European Union/United Nations Economic Commission for Europe

European Union/United Nations Economic Commission for Europe International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects on Forests

Working Group on QA/QC in Laboratories

# FutMon QA/QC Guide for Laboratory Work (1st version)

**ICP Forests Working Group on QA/QC in Laboratories** 

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(Version 1, February 2009)

Chapter		page
0.	Introduction	5
1.	Use of reference materials	5
1.1	Reference material for water analysis (deposition and soil	6
	solution)	
1.2	Reference material for foliar analysis	6
1.3	Reference material for soil analysis	7
2.	Use of control charts	8
2.1	Use of control charts for local reference material or	8
	laboratory control standards	
2.2	Use of control charts for blanks	10
2.3	Detection and quantification limit	11
3.	Check of analytical results	13
3.1	Check of analytical results for water samples	13
3.1.1	lon balance	14
3.1.1.1	Ion balance without DOC	14
3.1.1.2	Ion balance with DOC	19
3.1.1.3	Ion balance with DOC and metals	21
3.1.2	Conductivity check	22
3.1.3	Na/CI ratio check	24
3.1.4	N balance check	24
3.1.5	Phosphorus concentration as a contamination check	25
3.2	Check of analytical results for organic and mineral soil	25
0.2	samples	20
3.2.1	Plausible range checks for organic and mineral soil samples	25
3.2.2	Cross-checks between soil variables	28
3.2.2.1	pH check	28
3.2.2.2	Carbon check	28
3.2.2.3	pH-carbonate check	28
3.2.2.4	C/N ratio check	29
3.2.2.5	C/P ratio check	29
3.2.2.6	C/S ratio check	29
3.2.2.7	Extracted/total element check	29
5.2.2.1		30
		50
3.2.2.8	Reactive Fe and AI check	
3.2.2.9	Exchangeable element/total element check	30
3.2.2.10	Free $H^+$ and exchangeable acidity check	30
3.2.2.11	Particle size fraction sumcheck	31
3.3	Check of analytical results for foliar and litterfall samples	31
3.3.1	Plausible range check for foliage	31
3.3.2	Plausible range checks for litterfall	33
3.4	Analyses in duplicate	34
3.5	Avoidance of contamination problems	35
3.5.1	Water analyses	35
3.5.2	Organic and mineral soil analyses	36
3.5.3	Foliar and litterfall analyses	36
<b>4.</b>	Inter-laboratory quality assurance	30 37
<b>4</b> .1	Ring tests and ring test limits	37
4.1.1	Ring tests	37
7.1.1		57

4.1.2	Tolerable limits for ring tests	39
4.1.2.1	Tolerable limits for water ring tests	39
4.1.2.2	Tolerable limits for soil ring tests	42
4.1.2.3	Tolerable limits for plant (foliar and litterfall) ring tests	46
4.2	Exchange of knowledge and experiences with other laboratories	47
4.2.1	Exchange of know-how	48
4.2.2	Exchange of samples	48
5.	Quality Indicators	48
5.1	Percentage of the results of a ring test within tolerable limits for each ring test	
5.2	Percentage of the results of a ring test of repeatability below 10% Mean percentage of parameters for which laboratories use	
5.3	control charts	
6. 7.	Quality reports References	49 50
8.	Annexes	55
8.1	Excel worksheets for ion balance (with and without DOC correction), conductivity, N balance and Na/CI ratio checks	55
8.2	Excel worksheet for control charts	56
8.3	List of commercially available certified reference materials (CRM)	56

## 0. Introduction

Over the past years considerable efforts have been made to improve the quality of laboratory analyses in the various monitoring programmes within the framework of the ICP Forests programme. The Soil and Soil Solution, Deposition and Foliage and Litterfall expert panels have carried out a number of ring tests and held discussions on quality control. The expert panels' sub-group, 'Working Group on QA/QC in Laboratories', has extended its activities from the quality control of water analyses to encompass all forms of laboratory analysis, and now also includes experts in the fields of soil, foliage and litterfall.

This paper presents all the quality control methods that have been devised for the relevant fields of analytical chemistry. The aim is to provide those laboratories carrying out analyses within the ICP Forests programme with a complete overview of the possibilities of applying quality control in their laboratories.

## 1. Use of reference materials

Within the FutMon project the usage of control charts for each parameter and matrix is mandatory. For the producing of these control charts a reference material is necessary.

There are two types of reference material:

- 1. Reference Materials (RM): a material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (ISO Guide 30, 1992)
- 2. Certified Reference Materials (CRM): Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure, which establishes its traceability to an accurate realisation of the units in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence (ISO Guide 30, 1992). The CRM can be of national or international origin. A list of commercially available CRMs is given in Annex 6.4.

Reference materials are available in a range of types and price. CRMs are expensive and should be used only when really needed: **calibration, method validation, measurement verification, evaluating measurement uncertainty** (Nordtest Report 537, 2003), **and for training purposes**. In many cases, however, the concentrations are not within the ranges encountered in daily practice. National Reference Materials are, in many cases, easier to acquire and are often not as expensive as CRMs. They are usually issued by national laboratories, and are extremely useful for ensuring quality over the laboratories within a country.

In addition, laboratories must use matrix-matched control samples of demonstrated stability to demonstrate internal consistency over time, e.g. through control charts. The analyte concentrations of these samples do not

need to be accurately known or traceable. However, traceability would be a bonus. Here, again, CRMs or ring test samples can be used.

The Local Reference Materials (LRMs) are prepared by the laboratory itself for routine use and can be easily and cheaply prepared in large quantities. They can often also be prepared within the concentration ranges for the more important parameters. These LRMs are extremely important for QA/QC activities, mainly for use in **control charts** (see next chapter), if there is a need to maintain a constant (stable) quality over a longer time scale.

The following reference materials can be used in each field of interest:

#### 1.1 Reference material for water analysis (deposition and soil solution)

One alternative approach is to use natural samples that are preserved with stabilising agents (e.g. low chloroform concentrations), after first ensuring that their use does not cause interferences in the analytical methods or has an adverse effect on other activities performed in the laboratory. The use of natural samples makes it possible to have concentrations close to those normally measured. It is advisable to use two standards for each type of analysis, one of medium-low and one of medium-high concentrations, in relation to the range normally analysed. The stability of LRMs should be tested; their stability for individual ion species may vary.

One very cheap method for preparing an LRM is to buy mineral water that has chemical characteristics close to the range normally measured. Before you can use an LRM, however, you first have to validate your method (CRM). You should run your LRM together with the CRM or a ring test sample so as to determine the **conventional true value**.

For deposition samples, mineral water derived from volcanic bedrock has very similar concentrations. For soil solution samples, a specific type of mineral water has to be selected in accordance with the prevailing soil types in the monitoring network. The advantage of using mineral water is that they are relatively stable over time as long as the bottles of the same batch are stored in a dark place. However, mineral water does not contain dissolved organic carbon (DOC) in a form similar to that occurring in either deposition or soil solution samples.

#### 1.2 Reference material for foliar analysis

The matrix properties and the analyte concentrations of the reference material should be similar to those of the samples from the regional/national network. As there is only a limited number of forest-tree foliage reference material available worldwide, agricultural plant material with similar matrix and analyte concentrations, e.g. flour, hay, cabbage, olive leaves, apple leaves, sometimes has to be used. However, check the sales conditions before ordering –they are given on the webpage.

"Old" ring test samples are also stable enough and extensively analysed for use as reference material in method validation.

(A list of commercially available CRM`s is given in Annex 6.4)

One good cheap method for producing a high quality LRM is to prepare foliage material for use as a ring test sample. In the ring tests the Forest Foliar Co-ordinating Centre (FFCC) always utilizes dried, powdered foliage samples from one type of tree and leaf or a homogenized litterfall sample. Removal of the foliage, drying, milling and the first homogenization should be performed in the laboratory. One part (dry weight min. 4-5 kg) should be send to the FFCC (Contact: alfred.fuerst@bfw.gv.at). The FFCC homogenizes the sample again, divides it up and uses it in one of the subsequent ring tests. The advantage for the laboratory is in having a large amount of reference material with a similar element concentration as their normal samples and known accuracy of the mean concentration. The analytical results for this material should be used in the control charts (see next chapter) if it is necessary to have constant (stable) quality over a longer time scale or for calibration, method validation, measurement verification, evaluating measurement uncertainty and for training purposes.

#### 1.3 Reference material for soil analysis

International certified reference material is expensive and should be used only when really needed. In many cases, however, the concentrations are not within the ranges encountered in a specific country/region. (A list of commercially available CRMs is given in Annex 6.4)

National reference material is easier to obtain, is often not as expensive as international ones, and is produced by national laboratories in order to assure quality over the laboratories within a country. The advantage of local reference material is that it can be relatively cheaply prepared by the laboratory in question and is available in sufficient quantity to cover those concentration ranges encountered in normal laboratory work.

#### a. Preparation of local reference material for soils

Due to the type of soil samples and the nature of the two-step analysis, LRM samples are needed for both the solid phase (to control the quality of digestion) and the liquid phase (to control the quality of the chemical analyses).

#### - solid phase:

Take several large (10 to 50 kg) samples from one site (e.g. OL/OH horizons, mineral soil: preferably by horizon). Dry all the sampled material and homogenise the samples several times to ensure a uniform homogeneous sample. Split or riffle each sample into several parts and store in a cool, dry place. It may be worthwhile preparing several sets of the individual soil types and concentration ranges occurring in the country (e.g. one for clay soils in the coastal area with high sea salt concentrations, and one for sandy soil from an inland site).

#### - liquid phase:

After digestion of larger amounts of the solid phase LRM, store the solution (liquid phase) in a cool, dark place.

In general, no control of high concentrations is necessary because the errors are the higher the lower the concentration. Solutions with excessively high concentrations often have to be diluted in order to fit within the ranges for which the analysers have been calibrated. The amount of LRM has to be large enough to be used for an extended period of time (preferably up to one year). The amount needed annually will depend on the type of analytical equipment and methods used by the laboratory. The sample should be stored in such a way that no or minimal changes occur over time.

Note: a small standard deviation is good and an indicator of very accurate and precise work, but it is not the primary objective of this QA/QC document.

#### b. Calibration of local reference material for soils

After the preparation of the LRM, a test run has to be performed with perfectly calibrated equipment. A number of replicates (e.g. 5 for the solid and 30 for the liquid phase) have to be analysed for all relevant parameters, <u>and at least one</u> (but preferably more) national or international reference samples. The absolute accuracy is determined for each parameter on the latter samples. The standard deviation (SD) calculated from the results of analysis of the LRM should be as small as possible. The results of the first test run should be treated according to the ISO standard 8258 (1993, Shewhart control charts). The mean value of the parameters for the LRM is of less importance, but it should be within the same range as the values of the real samples that will be subsequently analysed.

Each parameter now has its own SD, which allows evaluation of the parameters and the relevance of the analysis by the method in question. If the SD is significantly larger than the expected values, then the relevance of analysing the parameter by the selected method is low. Other methods/equipment may have to be used to analyse the parameter within an acceptable range.

This procedure should be repeated whenever equipment is changed, important components are replaced, or when temporal trends appear in the results. The absolute values obtained from the national and international reference material are extremely importance in the last case.

## c. Use of local reference material for soils

After successful calibration, a systematic re-sampling of the LRM (liquid phase) is included in every batch or series of samples. Depending on the number of samples to be analysed and the methods and equipment used, this could be in the range of one LRM per 10 to 30 analysed real samples. For the solid phase (digestion and analysis) this could be reduced to one LRM per 30 to 50 analysed real samples.

The results of the repeated analysis of the LRM permit evaluation of the stability of the method/equipment over time. It is therefore important that no changes take place in the LRM over time. It is thus strongly recommended that the result of every analysis of the LRM is plotted on a graph over time (see ISO 8258, 1993; see next chapter on Shewhart control charts).

## 2. Use of control charts

Within the FutMon project the usage of control charts for each parameter and matrix is mandatory.

Control charts form an important practical aspect of internal QC in the laboratory. Using reference materials (see Chapter 1) the quality of the method can be checked immediately, while control charts are a useful tool for checking the quality and the variation in quality over a longer time scale. The

laboratory runs control samples together with the real samples in an analytical batch and, immediately after the run is completed, the control values are plotted on a control chart. There are various types of control chart available (for details see the ISO 8258, 1993). The most commonly used control charts are the **mean chart** and **range chart** for laboratory control standards, and the **blank chart** for background or reagent blank results.

In addition the control charts can be used for calibration, method validation and comparison, estimation of measurement uncertainty and limit of detection, checking the drift of equipment, comparison or qualification of laboratory personnel, and evaluation of proficiency tests.

For more information about the use of control charts see ref. Nordtest report TR 569, 2007.

# 2.1 Use of control charts for local reference material or laboratory control standards

**Means chart (X-chart).** The main aim of the means chart is to check the repeatability of the measurements in every batch of analyses It is constructed from the average and standard deviations of a standard, determined from a solution of one or more analyte(s), or a natural sample, that is sufficiently stabilised to keep the concentrations constant over time for at least 2-4 months. In the case of deposition samples, the choice of preservative (e.g. inorganic acids or chloroform) is determined by the analyte of interest and the conditions under which the analyses are carried out. It is advisable to use more than one control chart, at different concentration levels for each analyte.

The means chart is prepared on the basis of the first 20 to 25 measurements used to calculate the mean concentration  $(X_m)$  and the standard deviation(s). These variables are used to evaluate the upper and lower warning levels (UWL, LWL) and the upper and lower control levels (UCL, LCL). It is a common practice to use  $\pm$  2s and  $\pm$  3s limits for the warning limit (WL) and control limit (CL), respectively (Figure 2.1a). For variables with a non-normal distribution, transformation to a normal distribution may be necessary.

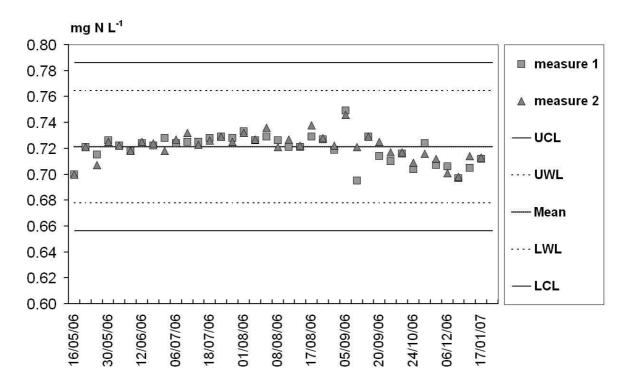
Assuming that s is correctly estimated, 95% of the measurements should fall within the range of  $X_m \pm 2s$  (WL) and 99% in the range of  $X_m \pm 3s$  (CL). In long-term routine analyses, on the other hand, UWL and LWL may be chosen by the analyst on the basis of experience with previous control charts or according to specific goals that are to be reached in the analyses.

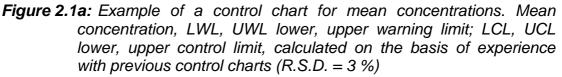
The means chart can also incorporate a target or nominal value of the analyte in the case of reference material with the reported concentration. The target control limits may also be used, and the laboratory results then be compared with these values.

If measurement uncertainty is determined for an analyte as a part of method validation, this value can be added to a means chart. Measurement uncertainty limits in the chart should lie between the warning and control limits (2s and 3s), in most case nearer the warning limit. The results of a control sample should not exceed the measurement uncertainty limits and, in the case of a synthetic control sample, they should remain between these limits. A target or nominal value can also be used with the measurement uncertainty limits. Because measurement uncertainty is propositional to the concentration of the analyte, different measurement uncertainty limits should be used for

different control charts of the same analyte. With this type of x-chart it is possible to check that the set measurement uncertainty is achievable in the course of time.

Every batch of analyses should include one or more measurements of the standard for the control chart. This measurement is plotted on the control chart: if a measurement exceeds the CL, the analysis must be repeated immediately. If the repeat is within the CL, then the analysis can be continued; if it exceeds the CL, the analysis should be stopped and the problem corrected. As regards the WL: if two out of three successive points exceed the WL, then an additional sample should be analysed. If the concentration is less than the WL, the analysis can be continued; if it exceeds the WL, then analysis can be continued; if it exceeds the WL, then analysis can be continued; if it exceeds the WL, then the analysis can be continued; if it exceeds the WL, then the analysis should be stopped and the problem corrected.





**Range chart (R chart).** The difference between two (or more) determinations on the same sample can also be described on a graph. This R chart is used for checking the repeatability of the analysis, usually of duplicate determinations. As the range is normally proportional to the sample concentration, it will therefore be more appropriate to use a control chart where the control value is the relative range r %.

#### 2.2 Use of control charts for blanks

Blank chart. A blank is defined as a solution of the purest available water that contains all the reagents used for the analysis, but not the analyte. The solution should be subjected to all the steps of the analysis (filtration, digestion, addition of reagents) up until the final measurement. The blank signal then indicates the sum of the analyte released in the different phases of the process, and a check must be made in order to exclude the possibility of occasional contamination. An example of a blank chart is shown in Figure 2.2a. The chart makes it possible to compare the blank values obtained in different batches of analyses at different times; an abnormally high blank value indicates the presence of contaminants at some stage of the process. The upper limit of acceptance is chosen by the analyst, either based on a previous set of analyses (e.g. two times the mean values of the blank dispersion absorbance) or on the of values around the mean.

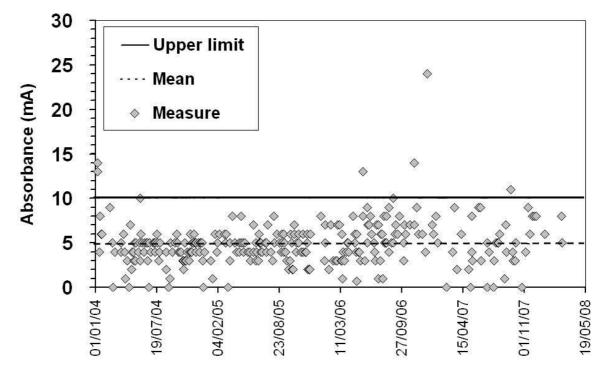


Figure 2.2a: Example of a blank chart

The standard deviation  $(s_b)$  of the blanks makes it possible to determine the detection limit (LOD) and the quantification limit (LOQ) of the analytical method. The LOD in most instrumental methods is based on the relationship between the gross analyte signal  $S_t$ , the field blank  $S_b$ , and the variability in the field blank  $(s_b)$ . The limit of detection and quantification may be defined by the extent to which the gross signal exceeds Sb:

$$\begin{split} LOD &= S_t - S_b \geq K_d \, s_b \\ LOQ &= S_t - S_b \geq K_q \, s_b \end{split}$$

Recommended values for  $K_d$  and  $K_q$  are 3 and 10, respectively (Analytical Methods Committee, 1987, Currie, L.A. 1999).

#### 2.3 Detection and quantification limits

Detection and quantification capabilities are fundamental performance characteristics of any chemical measurement process (Currie, 1999). For each matrix (soil, water, foliage) and each analytical method, the limit of detection (LOD) and quantification (LOQ) should be determined by each laboratory.

The limit of detection (LOD) is the smallest measure,  $x_L$ , that can be detected with reasonable certainty for a given analytical procedure.

The value of  $x_L$  is given by the equation:

 $x_L = x_{bi} + Ks_{bi}$ 

where  $x_{bi}$  is the mean of *n* blank measurements,  $s_{bi}$  is the standard deviation of *n* blank measurements, and *K* is a numerical factor chosen according to the confidence level desired (IUPAC, 1997). For LOD, this *K* factor is commonly set at 3 (see also  $K_d$  in Chapter 2.2). The LOD is the concentration at which we can decide whether an element is present or not. It is the point where we can just distinguish a signal from the background (Thomson et al., 2003).

It is recommended that the number of blank measurements (n) is higher than 30, preferably determined under within-lab reproducibility conditions (e.g. different operators, different runs on different days).

The limit of quantification (LOQ), also referred to as the quantitation limit, is generally agreed to begin at a concentration equal to 10 standard deviations of the blank ( $K_q = 10$ ). Therefore, LOQ is 3.3 times LOD. Quantitatively, the relative standard deviation (RSD) of repeated measures is 10% at the LOQ, and 33% at the LOD (Thomsen et al., 2003). This is in fact a statistical simplification of the uncertainty problem near the lower measurements limits, as explained by Currie (1999), but in practice it is a useful approximation.

			Absolute SD	Relative SD
Limit of detection		LOD	3σ	33 %
Limit	of	LOQ	10 σ	10 %
quantification				

Table 2.3.1. IUPAC recommendations for uncertainty associated with limits of detection and quantification (after Thomson et al., 2003).

A distinction should be made between instrument detection/quantification limits and method (or matrix) detection limits. Generally, instrument detection limits (IDLs) are based on a clean matrix. Method/matrix detection limits (MDL) consider real-life matrices such as soil, organic matter and rainwater. Spectroscopists commonly accept that the MDL can be anywhere from about two to five times worse than the IDL.

Therefore, labs should clearly mention whether the reported limits are instrument or matrix detection limits. In the case of environmental research, MDLs provide more relevant information than IDLs.

Measurement precision and concentration (or content) are often clearly related, as shown in Figure X. Generally, as the concentration or content of

the analyte decreases, the precision for determination, as expressed in the relative standard deviation, gets worse. When empirically precision data are gathered for each concentration or content level, a graph may be constructed as in Figure 2.3.1. Each data point represents the RSD of 8 to 20 replicate measurements per level.

When a curve is fitted with a suitable equation (e.g.  $y = a x^{-b}$ ) the limits of detection and quantification may be estimated from this equation by solving the RSD values of 30% and 10%, respectively. These limits are indicated on the graph and illustrate clearly that reliable determination of total N in this example is guaranteed for concentrations above the LOQ, whereas determination becomes highly uncertain between the LOD and LOQ.

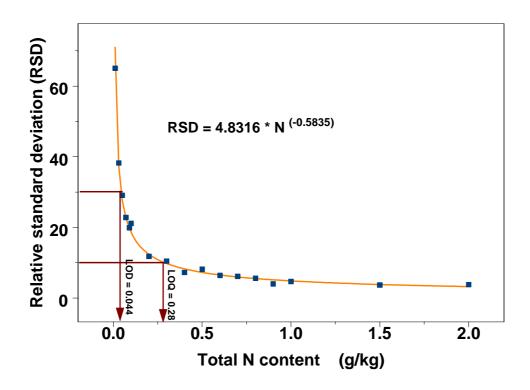


Figure 2.3.1. Relationship between measurement precision (RSD) and N concentration in a test mineral soil sample.

An example of application of the LOD and LOQ estimation method for the determination of carbon by the Walkley-Black method in forest soils can be found in De Vos et al. (2007).

This empirical method is time-consuming and laborious. However, it immediately shows the matrix detection and quantification limits for real-life samples under specific laboratory conditions.

# 3. Check of analytical results

#### 3.1 Check of analytical results for water samples

The solutes present in deposition and soil water samples and in soil extracts are mainly in ionic form. This enables the use of two checks on the consistency of the results of the analyses performed on individual samples: calculation of the ion balance, and comparison of the measured conductivity and the conductivity calculated from the sum of the contribution of the conductivity of each ion. A third consistency test, which is only valid for deposition samples, employs the ratio between the Na<sup>+</sup> and Cl<sup>-</sup> concentrations, which should normally be relatively close to the value in seawater. A fourth check, aimed at identifying analytical errors, is based on the relationship between the different forms of nitrogen analysed. Other statistical procedures that employ the relationship between the equivalent sum of ions (cations, anions) and conductivity, can be applied to the datasets. These are based on the relative similarity of the ratio between certain ions in deposition samples, due to their common origin (e.g. Na<sup>+</sup> and Cl<sup>-</sup> from sea spray,  $SO_4^{2-}$  and  $NO_3^{-}$  from combustion processes,  $Ca^{++}$  and alkalinity from soil dust). However, these methods require a relatively large set of data for the same type of precipitation before they can be applied to the results of single analyses in order to identify outlier values.

A more detailed explanation of the use of these tests and their incorporation in the analytical QC procedures is given in the ICP Forests manual (UN ECE, 2004, Ulrich et al., 2006). Examples of the application of these checks on sets of data from different sites in Europe have been reported by Mosello et al., 2005.

Most of the calculations needed to use the validation check, starting from concentration values, can be simplified by using a worksheet file similar to the one given in Annex 6.2.

## 3.1.1 Ion balance

## 3.1.1.1 Ion balance without DOC

As prescribed in the ICP Forests manual (UN ECE, 2004, Ulrich et al., 2006), each laboratory performs checks the chemical analyses by calculating the ion balance (for bulk open field and wet only deposition) and comparing the measured and calculated conductivity (for bulk open field and wet only deposition, throughfall and stemflow) values in order to validate the results. However, these checks are not always applicable to soil water (SW) samples. If the threshold values of these checks are exceeded, then the analyses must be repeated. If the result is confirmed but the threshold values are still exceeded, then the results must be accepted.

The ion balance is based on the equivalent concentration of anions vs. the concentration of cations ( $\Sigma$  Cat *vs.*  $\Sigma$  An):

$$\Sigma \text{ Cat} = [\text{Ca}^{++}] + [\text{Mg}^{++}] + [\text{Na}^{+}] + [\text{K}^{+}] + [\text{NH}_{4}^{+}] + [\text{H}^{+}]$$
  
$$\Sigma \text{ An} = [\text{HCO}_{3}^{-}] + [\text{SO}_{4}^{--}] + [\text{NO}_{3}^{-}] + [\text{CI}_{4}^{--}] + [\text{Org}_{4}^{--}]$$

The limit of acceptable errors varies according to the total ionic concentration and the type of solution. The percentage difference (PD) is defined as:

 $PD = 100 * (\Sigma Cat - \Sigma An)/(0.5*(\Sigma Cat + \Sigma An))$ 

The limits adopted in the ICP Forests/EU Forest Focus programmes are given in Table 3.1.1.1a

Table 3.1.1.1a: Acceptance threshold values in data validation based on ion balance and conductivity (see definition of PD and CD in the text).

Conductivity (25 ℃)	PD	CD
<10 µS cm⁻¹	±20%	±30%
<20 µS cm <sup>-1</sup>	±20%	±20%
>20 µS cm <sup>-1</sup>	±10%	±10%

The conversion factors required to transform the units used in the ICP Forests Deposition manual (into  $\mu$ eq L<sup>-1</sup> are given in Table 3.1.1.1b.

Table 3.1.1.1: The conversion factors used in converting the concentrations used in the ICP Forests Deposition Monitoring Programme to  $\mu eq L^{-1}$ , and the values of equivalent ionic conductivity at infinite dilution.

	Unit (ICPF standard)	Conversion factor to µeq L <sup>-1</sup>	Equivalent conductance at 20℃	Equivalent conductance at 25°C
			kS cm <sup>2</sup> eq <sup>-1</sup>	kS cm <sup>2</sup> eq <sup>-1</sup>
рН	unit	10 <sup>(6-pH)</sup>	0.3151	0.3500
Ammonium	mg N L <sup>-1</sup>	71.39	0.0670	0.0735
Calcium	mg L <sup>-1</sup>	49.9	0.0543	0.0595
Magnesium	mg L <sup>-1</sup>	82.24	0.0486	0.0531
Sodium	mg L <sup>-1</sup>	43.48	0.0459	0.0501
Potassium	mg L <sup>-1</sup>	25.28	0.0670	0.0735
Alkalinity	µeq L⁻¹	1	0.0394	0.0445
Sulphate	mg S L <sup>-1</sup>	62.37	0.0712	0.0800
Nitrate	mg N L <sup>-1</sup>	71.39	0.0636	0.0714
Chloride	mg L <sup>-1</sup>	28.2	0.0680	0.0764

Bicarbonate is calculated from total alkalinity (Gran's alkalinity) in relation to pH, assuming that total alkalinity is determined only by inorganic carbon species, protons and hydroxide:

 $TAIk = -[H^+] + [OH^-] + [HCO_3^-] + [CO_3^{2-}]$ 

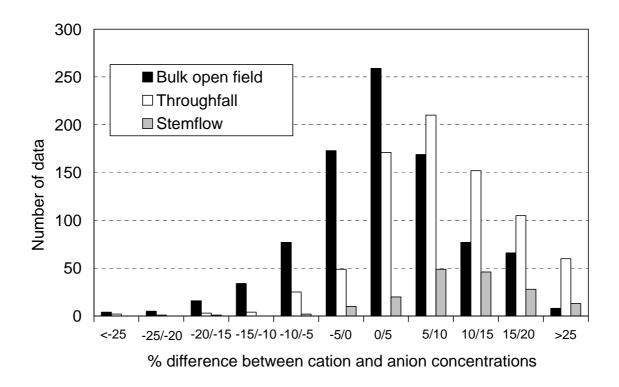
This definition is not completely correct in the case of high organic carbon concentrations (DOC > 5 mg C  $L^{-1}$ ), and in the presence of metals (AI, Fe, Mn etc) that may contribute to alkalinity or to the cation concentrations (see

Chapters 3.1.1.2 and 3.1.1.3) This sets limits on the use of the ion balance check in validating the analyses for certain types of solution, as summarised in Table 3.1.1.1c.

Table 3.1.1.1c: Applicability of the validation tests for different types of solution.

	lon balance	lon balance DOC corrected	Conductivity	Na/CI ratio	N test
Bulk open field	Y	Y	Y	Y	Y
Wet only	Y	Y	Y	Y	Y
Throughfall	N	Y	Y	Y	Y
Stemflow	N	Y	Y	Y	Y
Soil water	N	N	Y <sub>(2)</sub>	Ν	Y
Surface water	Y <sub>(1)</sub>	Y	Y	Ν	Y
	(1) If DOC <	centrations			
	(2) If metal c				

Examples of comparisons between  $\Sigma$  Cat and  $\Sigma$  An are given in Figure 3.1.1.1a for different types of solution. The departure from zero of the ion balance for different types of deposition sample is shown in Figure 3.1.1.1b, illustrating the failure of the check in the case of THR and STF samples.



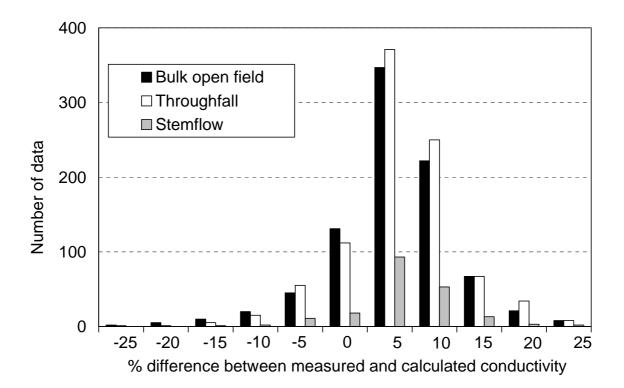
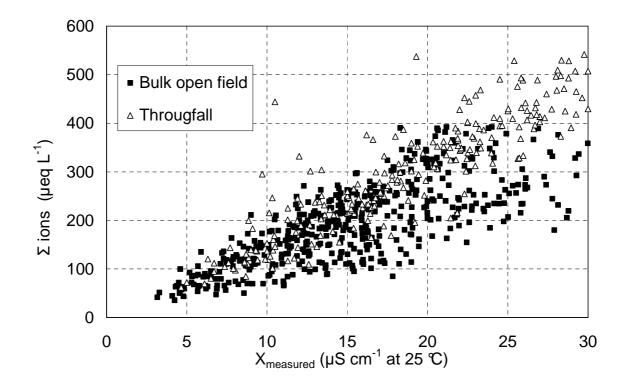


Figure 3.1.1.1a: Departure from zero of the percentage difference between  $\Sigma$  Cat and  $\Sigma$  An (PD), and (below) of the percentage difference between measured and calculated conductivity (CD) for different types of deposition sample.



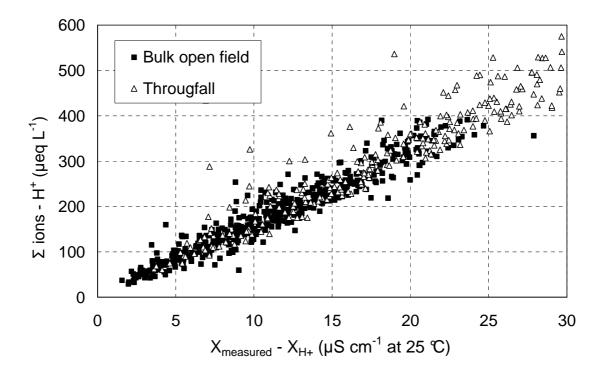


Figure 3.1.1.1b: Examples of the relationships between conductivity and  $\Sigma$  Cat or  $\Sigma$  An, above without the correction for H+ contribution to conductivity, and below with the correction.

## 3.1.1.2 Ion balance with DOC

Figure 3.1.1.1b clearly illustrates the failure of the ion balance check in the case of THR and STF samples. This is also the case for soil water samples (not shown in figure) in which, in addition to high DOC concentrations, elevated concentrations of metals may also be present (see Chapters 3.1.1.2 and 3.1.1.3).

The ion balance test can be used to evaluate the ionic contribution of DOC (all solutions are filtered through 0.45 um membrane filters before analysis) (Mosello et al., 2008). This study was carried out as part of the activities of the WG on QA/QC in laboratories regularly performing the chemical analysis of deposition and soil water samples within the framework of the ICP Forests and the EU/Forest Focus Programmes. About 6000 chemical analyses of bulk open field, throughfall and stemflow samples, which contained complete sets of all ion concentrations, alkalinity, conductivity and DOC, carried out in 8 different laboratories, were used to calculate empirical relationships between DOC and the difference between the sum of cations and the sum of anions. The aim was to determine the formal charge per mg of organic C. The samples covered a wide range of geographical and climatic conditions, as well as variables such as the proximity of the sea (chloride concentration) and the type of vegetation for THR and STF.

Regression coefficients were obtained for the data sets from each laboratory, as well as for all the data combined, as follows:

$$\Sigma \text{ Cat} - \Sigma \text{ An} = \delta_1 \text{ DOC} + \delta_0$$

where the units are  $\mu$ eq L<sup>-1</sup> for the sum of ions and  $\delta_0$ , mg C L<sup>-1</sup> for DOC, and  $\mu$ eq (mg C)<sup>-1</sup> for  $\delta_1$ . The regressions were not significant for BOF, because of the relatively high error associated with the low DOC concentrations. In contrast, the regressions were statistically highly significant for THR and STF in all the 8 laboratories.

In the next step, the charge contribution of DOC was determined as:

$$[Org^{-}] = \beta_1^* DOC + \beta_0$$

where  $[Org^-]$  (µeq L<sup>-1</sup>) is the ionic contribution of DOC. The value of PD was calculated again using the  $\Sigma$  An value including  $[Org^-]$ , and evaluated using the threshold values given in Table 3.1.1.1c.

An example of the regression coefficients,  $\beta_1$  and  $\beta_0$ , as well as the appropriate statistical parameters, are given in Table 3.1.1.2a. The coefficients were further tested using an independent set of data from each laboratory. Comparison of the differences between the individual laboratories and the overall regression coefficients showed that the coefficients were generally applicable for deposition samples, and also suitable for estimating the contribution of organic acids in the ion balance test. This means a considerable improvement in the applicability of the ion balance as a validation criterion for samples with high DOC concentrations. The improvement in the ion balance check in an example data set is shown in Figure 3.1.1.2a. This evaluation can also be found in the annexed Excel file, which contains examples of analysis validation.

Table 3.1.1.2a: Statistical parameters of the regression equations for determining the DOC contribution to the ion balance. THR = throughfall, STF = stemflow, N = number of samples,  $\sigma =$  standard deviation.

		Broadl	eaves	Conifers
	Units	THR	STF	THR
N	-	1454	597	1657
pH range	u	4.0 - 7.9	3.8 - 8.1	4.1 – 7.0
pH mean± $\sigma$	u	5.8±0.6	5.6±0.6	5.3±0.5
DOC range	mg C L⁻¹	0-37	1-39	0-40
DOC mean± $\sigma$	mg C L⁻¹	8±6	11±7	10±7
∑ Cat range	µeq L <sup>-1</sup>	37-2736	30-5287	13-2601
∑ Cat mean± σ	µeq L <sup>-1</sup>	418±321	593±539	316±278
∑ An range	µeq L⁻¹	29-2606	22-5303	10-2584
∑ An mean±σ	µeq L⁻¹	377±304	545±523	279±265
∑ Cat - ∑ An range	µeq L⁻¹	258	263	225
$\sum Cat - \sum An mean \pm \sigma$	µeq L⁻¹	41±59	48±58	37±41
Slope β <sub>1</sub>	µeq (mg C) <sup>-1</sup>	6,8±0,16	5.04±0.25	4.17±0.11
Intercept β <sub>0</sub>	µeq L⁻¹	-12,32±1,63	-6.67±3.29	-5.01±1.32
P-value		<0,0001	<0.0001	<0,0001
R <sup>2</sup>		0.56	0.4	0.47

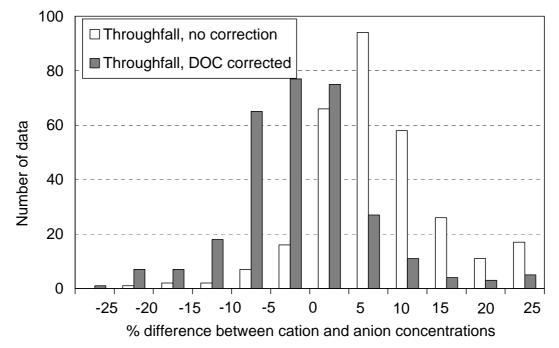


Figure 3.1.1.2a: Departure from zero of the percentage difference between  $\Sigma$  Cat and  $\Sigma$  An (PD, see text) without and with DOC correction.

## 3.1.1.3 Ion balance with DOC and metals

The ion balance for soil water samples is more complicated owing to the presence of metals (e.g. Al, Fe, Mn), their species (e.g.  $Al^{3+}$ ,  $Al(OH)^{2+}$ ,  $Al(OH)_{2^+}$ ,  $Fe^{3+}$ ,  $Fe(OH)^{2+}$ ,  $Fe(OH)_{2^+}$ ), their oxidation state (e.g.  $Fe^{3+}/Fe^{2+}$ ; iron complexed with organic matter can occur in both oxidised and reduced forms and the reduced forms can exist under oxidising conditions when complexed with organic matter; see e.g. Clarke and Danielsson, 1995) and metal complexes with DOC (e.g. DOC-Fe, DOC-Al, DOC-Mn) in the solution.

The calculation of bicarbonate from total alkalinity (see Chapter 3.1.1.1) is not completely correct because it is influenced by the different species of DOC in the solution.

Therefore calculation of the formal charge per mg of organic C from the difference between the sum of cations and the sum of anions, as described in Chapter 3.1.1.2 for throughfall samples, also has to take into account the metals, their species and their complexes with DOC:

Σ Cat + Σ Met (all inorg. species) + Σ Met (from DOC complexes) = Σ An + Σ Org<sup>-</sup> (from DOC complexes)

where:

 $\Sigma \text{ Met} = \text{Al}^{3+} + \text{Al}(\text{OH})^{2+} + \text{Al}(\text{OH})^{2+} + \text{Fe}^{3+} + \text{Fe}(\text{OH})^{2+} + \text{Fe}(\text{OH})^{2+} + \text{Mn}^{2+} + \text{Mn}(\text{OH})^{2+} + \text{Mn}^{2+} + \text{Mn}^{2+} + \text{Mn}(\text{OH})^{2+} + \text{Mn}^{2+} + \text{Mn}^{2+} + \text{Mn}(\text{OH})^{2+} + \text{Mn}^{2+} + \text{Mn}$ 

 $\Sigma \text{ Org}^{-}$  (from DOC complexes) = DOC-Fe + DOC-AI + DOC-Mn

Normally only the total concentrations of the metals and the total concentration of DOC are measured in soil solution samples. Therefore calculation of the formal charge per mg of organic C using the following formula overestimates the formal charge of DOC when the highest possible charge for the metals (Al<sup>3+</sup>, Fe<sup>3+</sup>,Mn<sup>2+</sup>) is used and there is no correction for bicarbonate:

$$\Sigma$$
 cat +  $\Sigma$  met<sub>total</sub> -  $\Sigma$  an =  $\delta_1$  DOC total

In an ongoing study being carried out by the WG on QA/QC in Laboratories, about 6200 chemical analyses on soil solution samples (complete sets of all ion and total metal concentrations, alkalinity, conductivity and DOC, carried out in the laboratories of 6 countries, were used to calculate empirical relationships between DOC and the difference between the sum of cations and metals and the sum of anions. The aim was to determine the formal charge per mg of organic C. The samples cover a wide range of geographical and climatic conditions. The results are shown in Figure 3.1.1.3a:

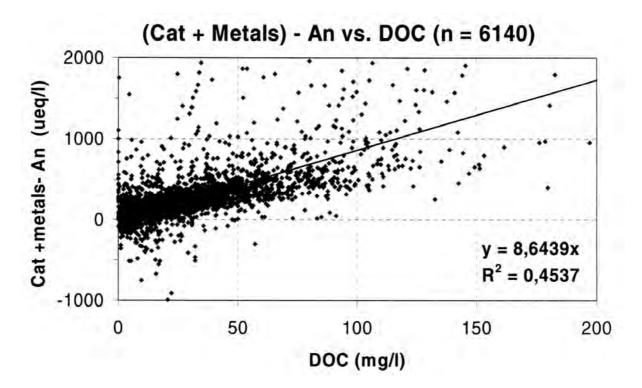


Figure 3.1.1.3a: Calculation of the formal charge of DOC in 6140 soil solution samples from 5 countries (Germany, Finland, France, Norway and the United Kingdom)

When the calculated charge factor for DOC was included in the ion balances of these soil solution samples, 64 % of the samples had equal ion balances (within +/- 10 %) while only 30 % of the samples had equal ion balances without using the DOC correction.

The results are different in the individual countries and at different pH values. Therefore the charge factor value obtained here can only be used as a first step in the procedure. It would be better to calculate the charge factor for specific countries or for similar types of plot. The chemical composition of DOC varies with depth down the soil profile (e.g. it is more polar at greater depth, Clarke et al., 2007), so the charge factor is also likely to vary with depth.

#### 3.1.2 Conductivity check

Conductivity is a measure of the ability of an aqueous solution to carry an electric current. This property depends on the type and concentration of the individual ions and on the temperature at which conductivity is measured. It is defined as:

$$\mathsf{K} = \mathsf{G}^*(\mathsf{L}/\mathsf{A})$$

where G = is the conductance (unit:  $ohm^{-1}$  or siemens;  $ohm^{-1}$  is sometime written as mho), defined as the reciprocal of resistance, A (cm<sup>2</sup>) is the electrode surface area, and L (cm) is the distance between the two electrodes. The units of K are  $ohm^{-1}$  cm<sup>-1</sup>. In the International System of Units

(SI), conductivity is expressed as millisiemens per meter (mS m<sup>-1</sup>); this unit is also used by the IUPAC and accepted as the Nordic standard. The unit  $\mu$ S cm<sup>-1</sup>, where 1 mS m<sup>-1</sup> = 10  $\mu$ S cm<sup>-1</sup> = 10  $\mu$ mho cm<sup>-1</sup>, is also widely used in practice. The unit adopted in the ICP Forests programme is  $\mu$ S cm<sup>-1</sup>, and the reference temperature 25 °C.

Conductivity depends on the type and concentration (activity) of the ions in solution; the capacity of a single ion to transport an electric current is given, in standard conditions and in ideal conditions of infinite dilution, by the equivalent ionic conductance ( $\lambda_i$ , unit: S cm<sup>2</sup> equivalent<sup>-1</sup>).

Careful, precise conductivity measurement is an additional way of checking the results of chemical analyses. It is based on comparison between measured conductivity (CM) and the conductivity calculated (CE) from the individual ion concentrations ( $c_i$ ), multiplied by the respective equivalent ionic conductance ( $\lambda_i$ )

$$\mathsf{CE} = \Sigma \ \lambda_i \ \mathbf{c}_i$$

The ions used in the conductivity calculations are the same as those used in calculating the ion balance; the values of  $\lambda_i$  for the different ions at temperatures of 20 and 25°C are given in Table 3.1.1.1b. As the concentrations are expressed in µeq L<sup>-1</sup>,  $\lambda_i$  is given as kS cm<sup>2</sup> eq<sup>-1</sup> in order to obtain the conductivity in µS cm<sup>-1</sup>. The percentage difference, CD, is given by the ratio:

$$CD = 100 * |(CE-CM)|/CM$$

At low ionic strength (below 100  $\mu$ eq L<sup>-1</sup>) in deposition samples, the discrepancy between measured and calculated conductivity should be no more than 2% (Miles & Yost 1982).

At an ionic strength higher than 100  $\mu$ eq L<sup>-1</sup> (approximately at conductivity higher than 100  $\mu$ S cm-1) it is necessary to use activity instead of concentration. This can be done by first calculating the ionic strength (*Is*, meq L<sup>-1</sup>) from the individual ion concentrations as follows:

 $Is = 0.5 \Sigma c_i z_i^2 / w_i$ 

where:

 $c_i$  = concentration of the *i*-th ion in mg L<sup>-1</sup>;  $z_i$  = absolute value of the charge for the *i*-th ion;  $w_i$  = gram molecular weight of the *i*-th ion.

For an ionic strength higher than 100  $\mu$ eq L<sup>-1</sup>, activities must be used instead of concentrations; in the range 100-500  $\mu$ eq L<sup>-1</sup> the Davies correction of the activity of each ion can be used, as proposed e.g. by Stumm and Morgan (1981) and A.P.H.A., A.W.W.A., W.E.F. (2005):

$$y = 10^{-0.5 \left(\frac{\sqrt{Is}}{1 + \sqrt{Is}} - 0.3Is\right)}$$

Finally, the corrected conductivity is calculated as:

$$\mathsf{CE}_{\mathrm{corr}} = y^2 \quad \mathsf{CE} = y^2 \quad \Sigma \ \lambda_i \ c_i$$

Immediate comparison of the measured and calculated conductivity makes it possible to identify single outlier values (see example in the annexed Excel file).

Figure 3.1.1.1a shows the departure from zero of the CD values for different types of deposition sample. The pattern is different from that for the ion balance: the CD values do not show any great asymmetry for BOF, THR, STF. The reason for this is that the DOC (organic matter), which causes an imbalance between the cation and anion concentrations ,does not contribute significantly to conductivity.

In conclusion, a plot of measured and calculated conductivity is useful in the routine checking of a set of analyses. Departure of the results from linearity suggests the presence of analytical or some other kind of error.

## 3.1.3 Na/CI ratio check

In many parts of Europe sea salt is a major contributor of sodium and chloride ions in deposition and, as a result, the ratio between the two ions is similar to that of sea salt. This is true even in parts of Europe situated far from the sea, as has been shown from a statistical study conducted on more than 6000 samples covering the area from Scandinavia to South Europe (Mosello et al., 2005). In the validation file (annexed Excel file), samples with a ratio outside the range given below are marked as possible failures, and checks and/or reanalyses should be carried out. The ratio is calculated by expressing the concentrations on a molar (or equivalent) basis.

0.5 < (Na/Cl) < 1.5

If the Na/Cl ratio results systematically fall outside this range, this may be due to to poor analytical quality in the measurement of low concentrations of sodium and chloride.

#### 3.1.4 N balance check

The test is based on the fact that total dissolved nitrogen (DTN) concentration must be higher than the sum of nitrate  $(N-NO_3)$ , ammonium  $(N-NH_4)$  and nitrite  $(N-NO_2)$  concentrations. Although the measurement of nitrite is not mandatory in the ICP Forests programme, the following relationship must be verified, within the limits of analytical errors and whatever unit is used:

$$[N-NO_3] + [N-NH_4] < [DTN]$$

If the relationship does not hold true, then the determination of one of the forms of nitrogen must be erroneous. However, if DON is very low, DTN may be approximately equal to  $NO_3-N + NH_4-N$ . In this case, normal (random) analytical errors may result in a slightly negative value of ([DTN] – ([NO<sub>3</sub>-N] + [NH<sub>4</sub>-N])), without there being any major problem with the analyses.

# 3.1.5 Phosphorus concentration as a contamination check

If bird droppings pass into the precipitation/throughfall/stemflow sample, this will considerably alter the chemical composition of the sample. The concentrations of  $PO_4^{3-}$ ,  $K^+$ ,  $NH_4^+$  and  $H^+$ , for instance, will be affected. A phosphate concentration of 0.25 mg l<sup>-1</sup> has been suggested as the threshold value for sample contamination by bird droppings (Erisman et al., 2003). Contamination by bird droppings is not always easily visible, so it may sometimes be detected only after the chemical analyses have been performed.

# 3.2 Check of analytical results for organic and mineral soil samples

An important step in laboratory QA/QC is to check whether the result of an analysis is within the expected range and that the general relationships between soil variables are valid. Therefore two checking procedures are recommended: plausible range checks and cross-checks.

# 3.2.1 Plausible range checks for organic and mineral soil samples

For each variable, there is a 95 % probability that the analytical result will fall within the plausible min-max range given in Table 3.2.1a. Values outside this range may occur, but they need to be validated (e.g. checking of equipment and method, dilution factor, reported unit, sample characteristics, signs of contamination). Re-analysis may be necessary when no obvious deviations are found in order to ensure that the results are correct.

Specific plausible ranges have been developed for organic material (forest floor, peat) and mineral soil samples. The number of significant decimal places for each variable is in accordance with the reporting format given in ICP Forests manual IIIa, Sampling and Analysis of Soil.

Generally, the lower limit of the min-max range depends on the limit of quantification (LOQ) which is, in turn, determined by the instrument, method and dilution factor used. Instead of merely mentioning 'LOQ', we have listed the average LOQ values reported by the soil laboratories that participated in the 4<sup>th</sup> FSCC Ring test (Cools et al., 2006). This is more informative. Laboratories with lower LOQ values than the average will be able to quantify lower concentrations reliably. However, each laboratory should always report concentrations lower than its LOQ as "-1" and reporting the LOQ concentration to the required number of decimal places in the data quality report.

The maximum value of the plausible range is determined by the maxima (mainly 97.5 percentile values) in the European forest soil condition database (first ICP Forests Level I Soil Survey). Information on the methods and data evaluation can be found in the Forest Soil Condition Report (EC, UN/ECE, 1997).

As it encompasses all the European soil types, this range is relatively broad. For some parameters, national plausible ranges will be narrower due to the restricted set of soil and humus types and their local characteristics. It would be worthwhile developing regional plausible ranges specifically for soil samples originating from the region. When the analytical data from the soils part of the BioSoil Project become available for elaboration, it will be possible to further develop the plausible ranges on both a European and regional scale.

If the values obtained in the analyses are outside the plausible range, the values should be marked with a flag for further investigation by the head of the laboratory and/or the responsible scientist. The head of the laboratory should be able to make comments in their report on possible reasons for the deviating value(s).

table 3.2.1a: Plausible ranges for organic and mineral soil samples at the European level. The number of decimal places indicates the required precision for reporting.

		Organic Plausibl		Plausibl	soil sample e range
Parameter	Unit	Min <sup>#</sup>	Max	Min <sup>#</sup>	Max
Moisture content (air-dry	%wt				
sample)		< 0.1	10.0	< 0.1	10.0
pH(H <sub>2</sub> O)	-	2.0	8.0	2.5	10.0
pH(CaCl <sub>2</sub> )	-	2.0	8.0	2.0	10.0
Organic carbon	g/kg	120.0	580.0	< 1.2	200.0
Total N	g/kg	< 0.5	25.0	< 0.1	20.0
CaCO <sub>3</sub>	g/kg	< 3	850	< 3	850
Particle size: clay	%wt			< 0.6	80.0
Particle size: silt	%wt			< 0.4	100.0
Particle size: sand	%wt			< 0.6	100.0
Aqua regia extractable P	mg/kg	< 32.8	3000.0	< 35.2	10000.0
Aqua regia extractable K	mg/kg	< 74.2	10000.0	< 81.4	40000.0
Aqua regia extractable Ca	mg/kg	< 45.9	100000.0	< 50.0	250000.0
Aqua regia extractable Mg	mg/kg	< 33.3	80000.0	< 38.5	200000.0
Aqua regia extractable S	mg/kg	< 128.6	7500.0	< 134.6	3000.0
Aqua regia extractable Na	mg/kg	< 20.6	3000.0	< 21.1	1000.0
Aqua regia extractable Al	mg/kg	< 76.1	40000.0	< 77.1	50000.0
Aqua regia extractable Fe	mg/kg	< 75.5	50000.0	< 82.6	250000.0
Aqua regia extractable Mn	mg/kg	< 7.2	35000.0	< 7.8	10000.0
Aqua regia extractable Cu	mg/kg	< 1.9	300.0	< 2.0	100.0
Aqua regia extractable Pb	mg/kg	< 2.4	1000.0	< 2.4	500.0
Aqua regia extractable Ni	mg/kg	< 1.5	300.0	< 1.6	150.0
Aqua regia extractable Cr	mg/kg	< 3.3	600.0	< 3.3	150.0
Aqua regia extractable Zn	mg/kg	< 2.0	1000.0	< 2.1	500.0
Aqua regia extractable Cd	mg/kg	< 0.5	18.0	< 0.5	6.0
Aqua regia extractable Hg	mg/kg	< 0.3	4.0	< 0.3	2.0
Exchangeable acidity	cmol+/kg	< 0.23	10.00	< 0.21	8.00
Exchangeable K	cmol+/kg	< 0.23	5.00	< 0.23	2.00
Exchangeable Ca	cmol+/kg	< 0.25	60.00	< 0.22	40.00
Exchangeable Mg	cmol+/kg	< 0.19	15.00	< 0.18	5.00
Exchangeable Na	cmol+/kg	< 0.18	1.50	< 0.17	1.00
Exchangeable Al	cmol+/kg	< 0.22	9.00	< 0.20	8.00
Exchangeable Fe	cmol+/kg	< 0.05	0.70	< 0.04	2.00
Exchangeable Mn	cmol+/kg	< 0.03	6.00	< 0.03	1.50
Free H+	cmol+/kg	< 0.25	10.00	< 0.21	3.00
Total K	mg/kg	< 50.0	10000.0	< 50.0	50000.0
Total Ca	mg/kg	< 20.0	100000.0	< 20.0	500000.0
Total Mg	mg/kg	< 5.0	80000.0	< 5.0	250000.0
Total Na	mg/kg	< 20.0 < 40.0	5000.0	< 20.0 < 40.0	12000.0
Total Al	mg/kg mg/kg	< 40.0 < 3.5	50000.0	< 40.0 < 3.5	100000.0
Total Fe	mg/kg		60000.0		250000.0
Total Mn	mg/kg	< 0.5	35000.0	< 0.5 < 44.6	15000.0
Reactive Al	mg/kg	< 44.6	5000.0		7500.0
Reactive Fe	mg/kg	< 48.4	5000.0	< 48.4	7500.0

<sup>#</sup> Values in bold are the average limit of quantification (LOQ) reported by the laboratories (Cools et al., 2006). The syntax is 'less than' LOQ (< LOQ).

# 3.2.2. Cross-checks between soil variables

Because different parameters are determined on the same soil sample and many soil variables are auto-correlated, cross-checking is a valuable tool for detecting erroneous analytical results. Obviously, soils high with a high organic matter content should have high carbon and (organically bound) nitrogen concentrations. Calcareous soils should have elevated pH values, high exchangeable and total Ca concentrations, but low exchangeable acidity. Simple cross-checks have been developed for easy verification and detection of erroneous results.

# 3.2.2.1. pH check

The soil reaction of organic and mineral soil material is measured potentiometrically in a suspension of a 1:5 soil:liquid (v/v) mixture of water (pH<sub>H2O</sub>) or 0.01 mol/l calcium chloride (pH<sub>CaCl2</sub>). The actual pH (pH<sub>H2O</sub>) and potential pH (pH<sub>CaCl2</sub>) are generally well correlated. Outliers may be detected using simple linear regression.

Theoretically, without taking measurement uncertainty into account, the difference between both pH measurements should be less than 1 pH-unit. In practice, the difference between both pH measurements is generally less than 1.2 pH-unit, with  $pH_{CaCl2}$  always less or equal to  $pH_{H2O}$ .

## Check algorithm: $0 < [pH_{H2O} - pH_{CaCl2}] \le 1.2$

Note that for peat soils, the difference between both pH measurements may be higher, up to 1.5 pH-units.

## 3.2.2.2. Carbon check

According to the manual, the recommended method for C determination is dry combustion using a total analyser (ISO 10694, 1995). In general, total organic carbon is obtained by subtracting inorganic carbon (TIC) from total carbon (TC), both of which are determined by the same analyser.

Inorganic carbon can be estimated from the carbonate measurement (ISO 10693, 1994) using a calcimeter (Scheibler unit).

Check algorithm:  $[C_{CaCO3}+TOC] \leq TC$  with  $C_{CaCO3} = CaCO_3 \times 0.12$ 

and

Check algorithm: C<sub>CaCO3</sub> ≈ TIC

The latter check cannot be performed if the carbonate concentration is below the LOQ (3 g kg<sup>-1</sup> carbonate or 0.36 g kg<sup>-1</sup> TIC).

## 3.2.2.3. pH-Carbonate check

Routinely determining carbonate in soil samples with low pH values is a waste of time and resources. Carrying out a fast, cheap pH measurement can be

used to decide whether carbonates are present and carbonate analysis is necessary.

For an organic sample (> 200 g kg<sup>-1</sup> TOC): Check algorithm: **if**  $pH_{CaCl2} < 6.0$  then  $CaCO_3 < 3$  g kg<sup>-1</sup> (= below LOQ)

For a mineral soil sample:

Check algorithm: if  $pH_{H2O} < 5$  then  $CaCO_3 < 3 \text{ g kg}^{-1}$  (= below LOQ) or: if  $pH_{CaCl2} < 5.5$  then  $CaCO_3 < 3 \text{ g kg}^{-1}$  (= below LOQ)

Conversely, if  $pH_{CaCl2} > 6$ , quantifiable amounts of carbonate are most likely present in the sample.

#### 3.2.2.4. C/N ratio check

Most of the nitrogen in a forest soil sample is organically bound. Carbon and nitrogen are linked through the C/N ratio of organic matter, which varies within a specific range.

For an organic sample (> 200 g kg<sup>-1</sup> TOC): Check algorithm: **5 < C/N ratio < 100** 

For a mineral soil sample: Check algorithm: **3 < C/N ratio < 75** 

## 3.2.2.5. C/P ratio check

Similarly to C/N, the C/P ratio varies within expected ranges for organic and mineral soil samples.

For an organic sample (> 200 g kg<sup>-1</sup> TOC): Check algorithm: **100 < C/P ratio < 2500** 

Note that for peat soils, the C/P ratio may be greater than 2500. In the  $5^{th}$  FSCC soil ring test, the C/P ratio of the peat sample was ca. 4500.

For a mineral soil sample: Check algorithm: **8 < C/P ratio < 750** 

#### 3.2.2.6. C/S ratio check

The C/S ratio varies within specific ranges for organic samples only.

For an organic sample (> 200 g kg<sup>-1</sup> TOC): Check algorithm: **20 < C/S ratio < 1000** 

#### 3.2.2.7. Extracted/total element check

In both organic and mineral soil samples the concentration of the aqua regia extractable elements K, Ca, Mg , Na, Al, Fe and Mn (pseudo-total extraction)

should be less than their total concentrations after complete dissolution (total analysis).

Therefore: Check algorithm: **Extracted element \leq Total element** for the elements K, Ca, Mg ,Na, Al, Fe and Mn.

## 3.2.2.8. Reactive Fe and Al check

Acid oxalate extractable Fe and Al indicate the active ( $\approx$  "amorphous") Fe and Al compounds in soils. Their concentration should be less than the total Fe and Al concentration.

#### Check algorithm: Reactive Fe ≤ Total Fe Reactive Al ≤ Total Al

For mineral soil samples, reactive Fe is usually less than 25 % of the total Fe, and reactive Al less than 10 % of the total Al.

#### 3.2.2.9. Exchangeable element/total element check

The elements bound to the cation exchange complex in the soil are also readily extracted using Aqua regia. Therefore, the concentration of exchangeable cations should always be lower than their Aqua regia extractable concentration.

A conversion factor is needed to convert from  $cmol_{(+)} kg^{-1}$  to mg kg<sup>-1</sup>.

(K <sub>exch</sub> x 391) ≤ Extracted K
(Ca <sub>exch</sub> x 200) ≤ Extracted Ca
(Mg <sub>exch</sub> x 122) ≤ Extracted Mg
(Na <sub>exch</sub> x 230) ≤ Extracted Na
$(AI_{exch}x 89) \leq Extracted AI$
(Fe <sub>exch</sub> x 186) ≤ Extracted Fe
(Mn <sub>exch</sub> x 274) ≤ Extracted Mn

In general, the ratio between an exchangeable element and the same extracted element is higher in organic matrices than in mineral soil.

#### 3.2.2.10. Free H<sup>+</sup> and Exchangeable acidity check

Two checks can be applied to Free  $H^+$  and Exchangeable acidity (EA).

## Check algorithm: Free $H^+ < EA$ Check algorithm: $EA \approx AI_{exch} + Fe_{exch} + Mn_{exch} + Free H^+$

For mineral soil samples, Free  $H^+$  is usually < 60 % of the Exchangeable acidity.

# 3.2.2.11. Particle size fraction sumcheck

According to the ICP Forests Manual IIIa, laboratories have to report the proportion of sand, silt and clay fractions in mineral soil samples. However, different methods are used for determining each fraction. After shaking with a dispersing agent, sand (63  $\mu$ m-2 mm) is separated from clay and silt with a 63  $\mu$ m sieve (wet sieving). The clay (< 2  $\mu$ m) and silt (2-63  $\mu$ m) fractions are determined using the standard pipette method (sedimentation).

When correctly applying the Soil manual procedure (SA03), which is based on ISO 11277 (1998) and includes the correction for the dispersing agent, the sum of the three fractions should be 100 %. The mass of the three fractions should equal the weight of the fine earth (0- 2mm fraction), minus the weight of carbonate and organic matter which have been removed.

# Check algorithm: Σ [ clay (%), silt (%), sand (%) ] = 100 %

Please check that the clay, silt and sand fractions are reported in the right format because mistakes occur regularly, even in ring tests.

## 3.3 Check of analytical results for foliar and litterfall samples

Compared to the checks for the analytical results on soil, deposition and soil solution samples, devising checks for foliage and litterfall samples is relatively difficult. In unpolluted "background" areas, the concentration range in foliage is usually small compared with that in other matrices and so most of the results are plausible.

Correlations between elements in foliage could be one possible tool for checking analytical results, but this is only suitable in cases where the sample plots are located very close to each other and have similar soil characteristics and the same tree species. As a result, this is probably not a useful procedure for checking the results in a European-wide survey.

# 3.3.1 Plausible range check for foliage

In order to provide the laboratories carrying out foliage analyses with support on QA/QC issues, a preliminary list of plausible ranges for the element concentrations in foliage was agreed on at the 4th Expert Panel Meeting in Vienna 1997. However, these limits were very broad (see: http://bfw.ac.at/600/pdf/ Minutes\_4.pdf).

In order to improve the list and put it on a more sound statistical basis, the Forest Foliar Coordinating Centre removed 5% of the lowest and 5% of the highest results from the European Level I database. 90% of all the submitted Level I results fell within these limits. As the manual covers a large number of different tree species it was necessary, in order to obtain sufficient data for meaningful statistical analysis, to group them into the main tree genera

Table 3.3.1a: Plausible range of element concentrations in the foliage of different tree species calculated from the Level II data sets (indicative values in grey).

Tree species	n	Limit		S	Ρ	Ca	Mg	K	С	Zn	Mn	Fe	Cu	Pb	Cd	В
			g/kg	g/kg	g/kg	g/kg	g/kg	g/kg	g/kg	µg/g	µg/g	µg/g	µg/g	µg/g	ng/g	µg/g
Fagus sylvatica	611	low	20.41	1.26	0.89	3.44	0.65	4.81	450	17.0	127	62	5.67	-	50	9.1
		high	29.22	2.12	1.86	14.77	2.50	11.14	550	54.2	2902	178	12.18	6.8	462	40.0
Quercus cerris	37	low	12.86	0.91	0.63	4.81	0.98	1.19	450	13.0	509	83	6.89	-	63	15.9
<b>•</b> "		high	30.79	3.24	2.29	16.49	3.24	15.64	550	-	-	-	-	-	-	-
Quercus ilex	141	low	11.95	0.81	0.69	4.00	0.76	3.42	450	12.7	278	73	4.00	-	-	21.7
<u> </u>	000	high	17,24	1,41	1,22	10,32	2,62	8,46	550	41,0	5385	717	7,00	-	-	-
Quercus petraea	268	low	19.75	1.24	0.90	4.12	1.06	5.86	450 550	11.0	905	60 1 4 0	5.39	-	24	5.5
Quercus		high	29.84	2.01	1.85	10.46	2.26	11.16	550	25.0	4209	149	11.64	-	-	-
pyrenaica																
(Q. toza)	27	low	17.85	1.18	1.48	4.60	1.40	3.52	450	18.0	434	81	8.07	-	-	-
		high	25,50	2,33	3,12	12,03	3,00	11,81	550	-	-	-	-	-	-	-
Quercus robur	242	law	20.24	4.00	0.07	0.00	4 00	F 00	450	110	040	~ 1	F F0	0.4	40	00.4
(Q. pedunculata)	313	low	20.31	1.36	0.97	3.33	1.09	5.80	450	14.0	219	64 222	5.50	0.1	40	23.4
0	00	high	30.69	2.21	2.55	12.26	2.85	12.64	550	50.0	2820	233	14.10	18.0	183	54.8
Quercus suber	39	low	11.39	0.85	0.47	4.29	1.22	4.37	450	17.0	291	62	6.11	-	-	17.5
Abies alks	000	high	23.09	1.61	1.53	11.02	2.55	9.85	550	47.0	2887	621	20.00	-	-	-
Abies alba	230	low	11.55	0.79	0.95	3.50	0.68	4.29	470	22.0	185	21	2.31	-	48	15.5
		high	16.16	1.69	2.23	11.71	1.90	8.48	570	45.0	2510	85	5.89	-	-	-
		low	11.67	0.95	0.86	4.19	0.37	3.97	470	20.0	250	32	2.00	-	56	14.4
Picea abies (P.		high	16.46	1.79	2.21	16.39	1.70	7.57	570	47.5	5241	121	6.45	-	-	-
excelsa)	1763	low	10.39	0.70	1.01	1.83	0.66	3.65	470	16.0	165	22	1.41	-	-	7.2
encereary		high	16.68	1.31	2.10	7.01	1.56	8.36	570	47.0	1739	91	5.94	2.9	226	29.4
		low	9.47	0.69	0.81	2.26	0.44	3.41	470	12.0	198	27	0.94	-		6.2
		high	15.97	1.34	1.82	9.77	1.51	7.05	570	51.8	2376	118	7.07	5.2	169	32.9
Diago aitak anaia	100	<u> </u>														
Picea sitchensis	108	low	12.67	0.98	1.04	1.21	0.78	5.56	470 570	8.4	147	31	0.70	-	-	6.0
		high	17.61	1.75	2.56	8.02	1.41	10.89	570	33.8	1489	232	5.91	-	-	42.0
		low	11.87	0.92	0.84	1.41	0.50	4.62	470	9.5	160	33	0.70	-	-	5.0
<b>D</b>	4.0	high	18.19	1.94	2.43	8.23	1.18	10.05	570	29.3	1734	133	4.67	-	-	52.0
Pinus contorta	40	low	11.31	0.75	0.98	1.02	0.79	3.56	470	-	-	-	-	-	-	-
		high	21.51	1.66	1.73	2.70	1.31	6.06	570	-	-	-	-	-	-	-
		low	13.12		0.88	1.96	0.75	1.21	470	-	-	-	-	-	-	-
Dinus holononsis	30	high	20.22	1.70	1.55	4.41	1.50 1.84	6.02 3.20	570 470	-	- 32	- 230	-	-	-	-
Pinus halepensis	30	low	9.22	0.92	0.80	2.12				23.0			-	-	-	-
Diassa aisara	04	high	14.28	1.68	1.79	8.04	2.89	8.67	570	-	-	-	-	-	-	-
Pinus nigra	81	low	8.42	0.51	0.81	0.97	0.56	3.88	470 570	18.8	60	29	1.81	0.6	399	8.9
		high	21.18	1.44	1.57	4.42	2.08		570	67.7	1072	131	18.08	-	-	-
		low	7.97	0.44	0.75	1.17	0.35		470	19.0	109	69	1.80	0.9	380	8.7
<b>D</b> :		high	23.49	1.93	1.71	6.90		7.34	570	70.0	1000	-	-	-	-	-
Pinus pinaster	116	low	6.85	0.61	0.55	0.80	1.01	3.26	470	15.6	41	23	1.70	-	-	15.0
		high	13.71	1.29		3.80	2.47		570	39.0	825	579	5.03	-	-	-
		low	6.25	0.55	0.40	1.09	0.94	2.40	470	12.3	35	23	1.13	-	-	20.0
		high	13.27	1.44	1.38	6.02	2.88	6.86	570	36.8	794	111	4.68	-	-	-
Pinus pinea	24	low	7.51	0.65	0.58	1.53	1.80	3.25	470	6.0	89	44	4.30	-	-	28.5
		high	11.30	1.65	1.20	4.40	3.00	6.70	570	-	-	-	-	-	-	-
Pinus sylvestris	1859	low	11.40	0.75	1.11	1.61	0.64	3.77	470	32.0	172	18	2.28	-	50	9.2
		high	20.41	1.56	2.06	4.61	1.31	7.27	570	77.6	912	139	7,70	3.9	447	30.5
		low	10.94	0.77	1.00	2.57	0.50	3.51	470	31.5	222	28	1.96	0.1	60	7.4
		high	19.38	1.61	1.88	6.71	1.18	6.52	570	96.0	1332	171	6.88	5.6	507	33.9
Pseudotsuga	107	low	10 F 4	1 00	1.00	1 00	1 00	E 17	170	15 0	150	10	0 70		1 1 1	20.0
menziesii	137	low bigb	13.54			1.98 5.01	1.02		470 570	15.0 45.2	159 1661	43 120	2.72	-	141	30.9
		high	22.71	1.80	1.70	5.91	2.10		570	45.3	1661	129	5.95	-	-	-
		low	13.55		0.71	3.09		2.97	470 570	14.0	444 155	58 270	2.91	-	-	-
	1	high	29.23	2.18	1.45	9.64	2.73	7.30	570	-	155	279	-	-	-	-

(Stefan et al., 1997). The new limits were adopted at the Expert Panel Foliage and Litterfall meeting in Madrid/Spain (2007).

The Joint Research Centre was asked to carry out a statistical evaluation on the submitted Level II results in order to obtain statistical information about the concentration range for different tree species. The 5% and the 95% percentile limits for each tree species were calculated. 90% of the submitted results fell within these limits (see Table 3.3.1a). Results falling outside these limits should be checked and, if necessary, be reanalyzed.

The report of the Level I foliage survey (Stefan et al., 1997) clearly shows that element concentrations in foliage vary considerably in different parts of Europe. There is a thus a need to calculate these limits for each country/laboratory using their own results. This would result in narrower limits that would provide a more reliable tool for detecting non plausible results.

#### 3.3.2 Plausible range check for litterfall

To develop tolerable limits for litterfall is much more difficult than for foliage. Litterfall is sorted in different fractions – in minimum in two, foliar and non-foliar litter. Many countries sort it in three fractions – foliage, wood and fruit coins & seeds. Litterfall is analyzed then as a pooled sample or each fraction is analysed separately.

The plausible range of the results of the chemical analysis of litter must be much bigger than for foliage. An important fraction in the litter is the foliar fraction, and for this fraction plausible ranges for selected tree species, based on the expert experience, are given in table 2. Plausible ranges for the nonfoliar fraction in litterfall is a project for the future.

Table 3.3.2a: Plausible range of element concentrations in the foliar-litter of different tree species (indicative values in grey).

Tree Species (Foliar litter)	Limit	с	s	N	Р	к	Ca	Mg	Zn	Mn	Fe	Cu	в
, , , , , , , , , , , , , , , , , , ,		mg/g	mg/g	mg/g	mg/g	mg/g	mg/g	mg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Betula pendula	low	290		7.30	0.20	0.30	5.,00	1.00	105.00	600	45.0	6	
	high	330		21.00	1.20	1.40	12.50	2.00	170.00	3000	300.0	19	38
Castanea sativa	low	390		9.00	0.20	0.20	4.50	1.40	35.00	700		5	
	high	420		13.00	0.70	0.55	10.50	2.00	45.00	2500	90.0	13	100
Fagus sylvatica	low	460	1	9.00	0.50	2.00	4.00	0.80	25.00	650	70.0	4	2
	high	510	2.2	19.00	1.90	8.00	17.00	2.00	35.00	1600	140.0	7	40
Fraxinus excelsior	low	470		12.00	0.75	0.40	20.00	2.00	15.00	110	120.0	7	
	high	470		18.00	1.50	1.40	25.00	3.50	20.00	200	200.0	9	50
Quercus frainetto	low		1.1	8.00	1.10	4.50	14.00	1.20					
(Q. conferta)	high		1.1	11.70	1.30	5.20	18.30	1.40					
Quercus petraea	low	460		8.00	0.30	2.00	7.00	1.30	14.00	700	50.0	5	
	high	510		12.00	0.60	4.00	10.00	2.00	25.00	1700	200.0	8	35
Quercus robur	low	460	0.85	10.00	0.82	4.00	5.00	1.00	15.00	1000	90.0	6	7
(Q. pedunculata)	high	510	1.7	19.00	2.00	8,00	13.00	2.00	25.00	1200	150.0	7	35
Abies cephalonica	low high			8.00 13.00		2.70 8.30	11.00 24.00	1.00 1.50					
Picea abies	low		1	6.50	0.60	1.00	2.50	0.70					
(P. excelsa)	high	520	1.5	12.60	1.20	4.20	2.30	2.20					
Picea sitchensis	low	440	1.0	6.00	0.60	1.50	4.00	0.60	15.00	250	40.0	2	
	high	530	1.1	13.00	1.10	3.00	11.00	1.00	35.00	1400	120.0	4	35
Pinus sylvestris	low	490	0.62	5.00	0.40	1.00	2.00	0.50	20.00	180	35.0	2	
	high	530	0.62	10.00	0.80	3.00	11.00	0.80	45.00	800	150.0	5	45

#### 3.4 Analyses in duplicate

Performing duplicate analyses represents a very worthwhile quality check. The samples or digestion solutions/extracts are measured twice independently for the individual parameters, the results are compared, and their repeatability determined.

$$S = \sqrt{\frac{\sum (x_i - \overline{x})^2}{n-1}} \bullet \frac{1}{\sqrt{n}}$$

s = Standard deviation  $\overline{x}$  = Mean value x = Measured value

n = Replicates

As this is a very time-consuming and expensive procedure when the number of samples is large, it may be sufficient to analyse only part (e.g. 5%) of the samples in duplicate. If this is adopted, 5% of the samples should be randomly selected and analysed again at the end of the batch. Thus one can check repeatability on the one hand and make sure that samples weren't mistakenly exchanged (for example during bottling on a sampler) in the course of a series on the other. If a mistake was found all samples of this batch must be repeated twice.

## 3.5 Avoidance of contamination

The contamination of samples can occur in the field during sampling, during the transportation of the samples to the laboratory, and during the pretreatment and analysis of the samples in the laboratory.

#### 3.5.1 Water analyses

Deposition samples can become contaminated already during the sampling period, e.g. as a result of bird droppings, and the laboratory should be informed about signs of any such contamination. The transfer of deposition and soil water samples in the field from the sampling devices to the bottles used for transportation to the laboratory is one stage that can result in contamination of the samples. The best way to avoid this problem is to transport the collection devices (bottles, bags etc.) directly to the laboratory, if possible. The most important point during this step, as well as throughout the whole sample preparation procedure in the laboratory, is to avoid skin contact by using disposable gloves (non talc), and the use of clean equipment (e.g. glass- and plasticware).

Special care must be taken when filtering the samples, and at least separate plastic tubing (if used) or other filtering devices for different types of sample (bulk, throughfall, stem flow, soil solution) should be used. Rinsing the filter capsule or funnel between the samples with the next sample, and not only with purified water, is recommended. If this is not possible, then an adequate amount of the next sample should be discarded after filtering before taking the sample for the analyses. Contamination control samples (ultra pure water) should be used after every 20 to 30 samples depending on the type of filtering system. It is always recommendable to start working with cleaner samples (e.g. bulk first) and continue with the other types of sample. Attention should also be paid to the different characteristics of the individual sample plots and their specific concentrations.

The material of the filters should be suitable for the analyses to be carried out, e.g. paper filters can affect ammonium and DOC determinations through contamination and the release of paper fibres that of course contain C. In some cases, the opposite may occur: sample loss through adsorption on filters. For the filtration of samples on which DOC is to be determined, glass fibre filters are recommended.

The filters and the amount of ultra pure water needed to rinse off possible contaminants should be tested and checked by using blank charts. The filters should be handled with clean forceps.

One highly recommendable procedure is to use a separate set of bottles for preparing the standard solutions for every single type of analysis. If the pH or conductivity value for a sample is exceptionally high, then it is recommendable to inform the persons carrying out the other analyses (which are usually performed later) about the "abnormal" sample.

# 3.5.2 Organic and mineral soil analyses

Samples of organic and mineral soil material need several preparatory steps prior to analysis. Contamination can occur In each of these steps.

Cleanliness of equipment, glass- and plastic-ware, is a prerequisite for avoiding contamination and conforming with good laboratory practice.

Milling and/or sieving is the first step in the pre-treatment of organic and mineral soil samples.

The milling equipment is one possible source of contamination. Metals, especially, may be released through abrasion of the inner compartments or sieves. In the laboratory responsible for preparing the FSCC ring test samples, the use of a hammer-mill system with a titanium rotor and a stainless steel sieve was tested for milling organic samples. Milling resulted in elevated Ni and Cr concentrations of up to 3.6 and 2.2 mg kg<sup>-1</sup>, respectively, whereas for manual pulverization the increase was below 0.6 mg kg<sup>-1</sup> for both metals. Although no systematic contamination was observed, the degree of contamination appeared to be a function of the hardness of the sample material (wood, bark) and the age of the sieve. The use of titanium rotors and sieves is therefore recommended, as well as periodical replacement of the sieves.

According to the manual, mineral soil samples should not be milled, but sieved over a 2 mm sieve. These sieves should be clean, with no traces of oxidation on their metallic parts. Attention should be paid to ensure that no residues from tools (crusher, pestle, brush, cleaning equipment) end up in the samples as a result of thorough cleaning by brushing or wiping. This also holds true for other equipment (sample divider, mixer, splitter, riffler). When pre-treating silty or clayey soil samples, appropriate methods (air extraction equipment) should be used to avoid contamination of other samples or equipment via the air.

If a separate container is used to weigh and transfer sub-samples to extraction vessels, then it should be carefully brushed clean between samples to avoid cross-contamination. All glass- and plastic-ware should be cleaned by rinsing with a dilute acid solution or appropriate cleaning agent. Rinsing twice with distilled or deionized water and drying before reuse is a common practice.

lons adsorbed on the inner surfaces of extraction flasks or sample bottles coming into contact with extracts may be a source of contamination for subsequent analyses using the same containers.

Finally, some types of filter paper used for filtration may contain contaminants. Many laboratories encounter problems with  $Na^+$  or other cations. Careful analysis of blanks and the filter material may indicate problematic elements that enhance the background noise.

## 3.5.3 Foliar and litterfall analyses

There are many possible contamination sources in foliage and litterfall analyses. A short overview is given in Table 3.5.3a.

Table 3.5.3a: Possible contamination sources in foliage and litterfall analyses for some elements

Element	Possible contamination source
N	NH <sub>3</sub> from the laboratory air (only if the Kjeldahl method is used),
	reagents
S	Water (distilled or deionised), reagents
Р	Dishwasher (detergent), water (distilled or deionised), reagents
Ca	Soil contamination from sampling, water (distilled or deionised),
	glassware, reagents
Mg	Soil contamination during sampling, water (distilled or deionised),
	glassware, reagents
K	Dishwasher (detergent), water (distilled or deionised), glassware,
	reagents
Zn	Soil contamination during sampling, Dishwasher (detergent),
	water (distilled or deionised), glassware, dust, reagents
Mn	Reagents
Fe	Soil contamination during sampling, water (distilled or deionised),
	glassware, dust, reagents
Cu	Water (distilled or deionised), glassware, reagents
Pb	Soil contamination during sampling, glassware, dust, reagents
Cd	Soil contamination during sampling, glassware, dust, reagents
В	Water (distilled or deionised), glassware, reagents
Cr, Ni	Instruments made of stainless steel used in sampling, pre-
	treatment etc.
С	Reagents

## 4. Interlaboratory quality assurance

In addition to the quality assurance carried out within each laboratory, there are also quality checks and procedures that can be used between different laboratories. These include ring tests, as well as the exchange of experiences and methods employed between laboratories. In the case of international programmes, especially, the use of identical analytical methods and regular ring tests are of particular importance in ensuring comparability and joint evaluation of the data.

## 4.1 Ring tests and ring test limits

## 4.1.1 Ring tests

Within the FutMon project the participation in different ring tests is mandatory for all laboratories which are analysing samples from the FutMon project. Water and plant ring tests will be organized yearly and soil and soil physics ring tests once in 2 years. For the participation in the ring tests each laboratory has to registrate on-line. Information about the registration procedure will be sent to the laboratories by the responsible person for the ring tests. A series of inter-laboratory comparison tests is an excellent tool for improving the quality of the data produced by the participating laboratories over time. This is because of the training effect in the use of a method, and because the remaining ring test sample material can be used as reference material in the laboratory up until the next ring tests. If the data (e.g. analytical results) generated in environmental monitoring or long-term ecological research programmes are of poor quality, then this may prevent the detection of trends, resulting in delays of up to three decades before they can be identified (Sulkava et al., 2007). Tolerable limits for the deviation of the individual test result from the comparison mean value were selected for each variable measured. Results falling outside the tolerable limits indicate problems in the analytical procedure, or more general quality problems in the laboratory. The tolerable limits were set in order to act as a driving force to reduce measurement uncertainty and increase the comparability of results among the participating laboratories. As a result, the tolerable limits have, in some cases, been adjusted downwards in order to maintain their role as a driver for quality improvement as an increasing number of the laboratories meet this quality requirement.

Ring tests should be carried out between the involved laboratories at regular intervals in order to ensure comparability of analytical data. This involves the dispatch of 3 to 10 samples or solutions to the participating laboratories, where they are analysed using previously agreed on methods. The results are then returned to the organizers of the ring test.

The ring test samples must be checked for homogeneity and, in the case of water samples, have been stabilized by means of filtration through a 0.45  $\mu$ m membrane filter, addition of acid or similar procedure. When mailed to the laboratories, the samples have to be packed in non-breakable flasks, and water samples should be kept cool during transportation.

The analysis of 4 to 6 samples, representing different concentrations of the individual parameters, is the optimum, because only then can clear analytical trends be identified for each participating laboratory. This simplifies the detection of possible analytical mistakes and differences in the methods used. Particularly in the case of water samples, it is necessary to set a time period during which the analysis must be carried out. This avoids chemical/biological changes in the samples which, in turn, would lead to differences in the results. Care should be taken to agree on standard treatment of the samples and analytical methods. This includes their preparation such as sieving or grinding, digestion or extraction and determination of element concentrations.

The effects of differing methods on the results of the ring test can only be investigated if the methods used are properly documented or a method-code used.

The participating laboratories should carry out the ring tests as a part of their normal laboratory analysis runs so that the functioning of their normal routine activities can be checked.

The organizers of the ring tests have to develop standard forms or internetbased files so that all the analysis data can be recorded in a standard fashion and used in standardized evaluation programmes for ring tests. It is particularly important to define the units to be used and the required number of decimal places for reporting. There are a number of computer programmes on the market that comply with standards such as DIN 38402/42 (1984), and these can be used for evaluating the analysis data. Custom-made programmes can also be developed. The deviation from the mean value and the coefficient of variation, as well as outliers, must be recorded for each parameter and for each sample.

## 4.1.2 Tolerable limits for ring tests

In order to evaluate the results of ring tests and of the participating laboratories, tolerable deviations from the mean value, expressed as a percentage for each parameter and method, have to be determined. As a rule, the permitted deviations for double-stepped analytical methods (e.g. digestion/extraction and subsequent determination of the element concentration in the solution) are significantly larger than for direct element determination.

Within the FutMon project all laboratories will get a qualification report after the participation in a ring test. In this report information about the analysed and not analysed parameters and the passing of the qualification criteria for each parameter is listed.

The actual qualification criterion for a ring test parameter is that 50 % or more of the results of all samples for this parameter must be within the tolerable limits.

The WG on QC/QA in Laboratories and the various expert panels of the ICP Forests programme have proposed tolerable limits for samples and parameters. They are described in the following.

## 4.1.2.1 Tolerable limits for water ring tests

Discussions on the results of the two deposition/soil water ring tests highlighted the need for quantification of the acceptable limits of errors among analyses performed in different laboratories. These Data Quality Objectives (DQO) are essential in ensuring the comparability of the results, and to avoid "border effects" in the evaluation of results from different countries. The DQOs need to be higher than the precision in the individual laboratories (when working in accordance with QA/QC criteria) because they include part of the systematic errors that are not included in the precision of the individual laboratories. As is the case for the acceptance values for the validation check of single analyses (Chapter 3.1.6), selection of the DQO should take into account the fact that excessively large acceptance thresholds are of little use for ensuring good data quality, while too strict threshold values that are frequently exceeded are soon forgotten. The proposed set of values is only a preliminary step and it needs to be verified in practice and, if needed, changed. It also may be necessary to use different DQOs for "low" or "high" concentrations. However, the results of the next inter-laboratory exercises will show whether this is necessary.

Examples of similar DQOs used in other networks, such as the Global Atmospheric Watch (Allan, 2004) and the EMEP (Uggered et al., 2005) are given in Table 4.1.2.1.

The proposed DQO values for deposition/soil water inter-comparison are listed in Table 4.1.2.2, and are compared with the average of all the samples of the 95% confidence limit of the results obtained in the second ring test exercise (Marchetto et al., 2006), after the exclusion of outliers. These DQOs are intended for general use with samples of average or high concentration.

A second set of DQOs, shown in Table 4.1.2.3, is provided for use with dilute samples, when one or more concentrations are very low, close to the detection limits of the analytical methods, and the expected errors became higher.

It is evident that a significant proportion of the results are still higher than the DQO values, indicating the need for improvements in the performance of the laboratory. On the other hand, many laboratories had values lower than the DQO, clearly indicating that it is possible to remain within these thresholds. The table also highlights a number of analyses that still require a considerable amount of work, such as alkalinity (low values in deposition samples), total nitrogen and DOC. The analytical problems associated with these determinations were discussion in connection with the two ring tests (Mosello et al., 2002, Marchetto et al., 2006).

Parameter	Unit	GAW Laboratory Inter-Network Bias	EMEP radii for Youden plot
рН		± 0.07 u. pH	± 0.1 u. pH
Conductivity	µS cm⁻¹	±7%	± 10 %
Calcium	mg L <sup>-1</sup>	± 15 %	± 15 %
Magnesium	mg L <sup>-1</sup>	± 10 %	± 15 %
Sodium	mg L <sup>-1</sup>	± 10 %	± 15 %
Potassium	mg L <sup>-1</sup>	± 20 %	± 15 %
Ammonium	mg N L <sup>-1</sup>	±7%	± 15 %
Sulphate	mg S L⁻¹	±7%	± 10 %
Nitrate	mg N L <sup>-1</sup>	±7%	± 15 %
Chloride	mg L <sup>-1</sup>	± 10 %	± 15 %
Alkalinity	µeq L⁻¹	± 25 %	± 25 %
Total dissolved nitrogen	mg L <sup>-1</sup>	-	± 20 %
Dissolved organic carbon	mg L <sup>-1</sup>	-	± 20 %
Other (metals)		-	± 20 %

Tab. 4.1.2.1: Data Quality Objectives for precipitation and soil water concentrations adopted in other atmospheric deposition networks.

Tab. 4.1.2.2: Data Quality Objectives proposed for the ICP Forests				
programme compared with the results of the second ICP Forests/Forest				
Focus ring test (Marchetto et al., 2006). DQOs valid for relatively high				
concentrations.				

Parameter	for values	DQO	2 s.d.	mean no. of outliers
рН	< 5.0 units	± 0.1 u. pH	0.17 u.	1.6
Conductivity	> 10 µS cm⁻¹	± 10 %	13%	0.7
Calcium	> 0.25 mg L <sup>-1</sup>	± 15 %	18%	1.7
Magnesium	> 0.25 mg L <sup>-1</sup>	± 15 %	14%	1.5
Sodium	> 0.5 mg L <sup>-1</sup>	± 15 %	12%	3.4
Potassium	> 0.5 mg L <sup>-1</sup>	± 15 %	11%	2.3
Ammonium	> 0.25 mg N L <sup>-1</sup>	± 15 %	16%	4.3
Sulphate	> 1 mg S L <sup>-1</sup>	± 10 %	7%	3.8
Nitrate	> 0.5 mg N L <sup>-1</sup>	± 15 %	10%	1.8
Chloride	> 1.5 mg L <sup>-1</sup>	± 15 %	11%	5.3
Alkalinity	> 100 µeq L⁻¹	± 25 %	66%	0.0
Total dissolved nitrogen	> 0.5 mg L <sup>-1</sup>	± 20 %	15%	3.3
Dissolved organic carbon	> 1 mg L <sup>-1</sup>	± 20 %	20%	1.8
Other (metals)		± 20 %		

Tab. 4.1.2.3: Data Quality Objectives proposed for the ICP Forests programme compared with the results of the second ICP Forests/Forest Focus ring test (Marchetto et al., 2006). DQOs valid for low concentrations.

Parameter	for values	DQO	2 s.d.	mean no. of outliers
рН	> 5.0 units	± 0.2 u. pH	0.27 u.	1.6
Conductivity	< 10 µS cm⁻¹	± 20 %	-	-
Calcium	< 0.25 mg L <sup>-1</sup>	± 20 %	31%	2.5
Magnesium	< 0.25 mg L <sup>-1</sup>	± 25 %	20%	3.5
Sodium	< 0.5 mg L <sup>-1</sup>	± 25 %	-	-
Potassium	< 0.5 mg L <sup>-1</sup>	± 25 %	30%	3.0
Ammonium	< 0.25 mg N L <sup>-1</sup>	± 25 %	42%	4.0
Sulphate	< 1 mg S L <sup>-1</sup>	± 20 %	11%	3.3
Nitrate	< 0.5 mg N L <sup>-1</sup>	± 25 %	38%	2.3
Chloride	< 1.5 mg L <sup>-1</sup>	± 25 %	22%	3.5
Alkalinity	< 100 µeq L <sup>-1</sup>	± 40 %	161%	1.3
Total dissolved nitrogen	< 0.5 mg L <sup>-1</sup>	± 40 %	51%	2.5
Dissolved organic carbon	< 1 mg L <sup>-1</sup>	± 30 %	98%	2.0

## 4.1.2.2 Tolerable limits for soil ring tests

For the inter-laboratory comparison of organic and mineral soil samples, tolerable limits were calculated on the basis of the Mandel's h (between laboratory variation) and Mandel's k (within-laboratory variation) statistics of the earlier FSCC soil ring tests (De Vos, 2008). An explanation of the evaluation methodology for the soil ring tests based on ISO 5725-2 (1994) is given in the FSCC ring test reports (Cools et al., 2003, 2006, 2007).

Tolerable limits for the soil ring tests are inferred from the coefficient of variation for laboratory reproducibility ( $CV_{repr}$ ). For many soil variables,  $CV_{repr}$  decreases with increasing concentrations, as shown for total nitrogen in Figure 4.1.2.2. In the lower range, the inter-laboratory variation relative to the mean may be as high as 100 %, or even more, whereas in the higher range this variation is much lower. Therefore, tolerable CV's are fixed for both a lower and a higher range for each soil variable. For the N concentration example, the  $CV_{repr}$  for the lower range ( $\leq 1.5$  g N kg<sup>-1</sup> DW) is set to the average of 30 % and for the higher range (> 1.5 g N kg<sup>-1</sup> DW) to 10% (Fig. 4.1.2.2). For some variables (e.g. pH), no split in a lower and higher range is justified due to the linear relationship of the reproducibility curve.

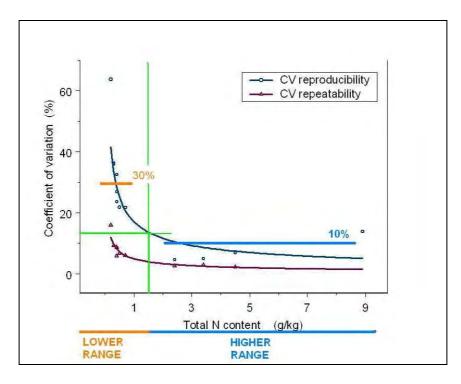


Figure 4.1.2.2: Power curves fitted to the results of total N analysis on the mineral soil samples of earlier FSCC ring tests, and estimation of the lower and higher ranges based on the turning point of the reproducibility curve. Average CV is 30 % and 10 % for the lower and higher range, respectively.

Tolerable limits are set using a z-score of 1: the deviation from the mean is equal to the standard deviation (SD). Consequently, tolerable limits equal the average  $CV_{repr}$  in the earlier FSCC ring tests, rounded off to the nearest 5 %.

Because the tolerable limits equal  $\pm$ SD, in theory 68% of the labs should meet this criterion. However, a simulation for the 5<sup>th</sup> ring test revealed that, on the average, 70-90 % of the laboratories reported results within the tolerable range and 10-30 % failed, depending on the variable in question.

In the future, as laboratory performance improves, these limits will be gradually narrowed using z-scores of less than 1.

Tolerable limits can also be inferred for intra-laboratory variation (repeatability). These limits can be used to evaluate within-laboratory repeatability on replicated analyses within the same run.

Table 4.1.2.2a: Tolerable limits for soil moisture content, pH, organic carbon (OC), total nitrogen (TN) and carbonate for inter-laboratory comparison and intra-laboratory performance.

Parameter	Observation Range	Level	Ring Test Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Moisture	lower	≤ 1.0	± 25	± 6
content (%)	higher	> 1.0	± 15	± 4
рН <sub>н20</sub> -	whole	2.0 - 8.0	± 5	± 1
pH <sub>CaCl2</sub> -	whole	2.0 - 8.0	± 5	± 1
OC	lower	≤ 25	± 20	± 5
g kg⁻¹	higher	> 25	± 15	± 3
TN	lower	≤ 1.5	± 30	± 9
g kg⁻¹	higher	> 1.5	± 10	± 3
Carbonate	lower	≤ 50	± 130	± 5
g kg⁻¹	higher	> 50	± 40	± 3

Table 4.1.2.2b. Tolerable limits for soil texture for inter-laboratory comparison and intra-laboratory performance.

Parameter	Observation Range	Level	Ring Test Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Clay content	lower	≤ 10.0	± 50	± 8
%	higher	> 10.0	± 35	± 4
Silt content	lower	≤ 20.0	± 45	± 8
%	higher	> 20.0	± 30	± 3
Sand content	lower	≤ 30.0	± 45	± 6
%	higher	> 30.0	± 25	± 2

Table 4.1.2.2c: Tolerable limits	fc	or total elements for inter-laboratory
comparison and intra-laboratory	/	performance.

Parameter	Observation Range	Level	Ring Test Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
TotAl	Lower range	≤ 20000	± 35	± 4
mg kg⁻¹	Higher range	> 20000	± 5	± 1
TotCa	Lower range	≤ 1500	± 20	± 7
mg kg⁻¹	Higher range	> 1500	± 15	± 2
TotFe	Lower range	≤ 7000	± 20	± 5
mg kg⁻¹	Higher range	> 7000	± 5	± 2
TotK	Lower range	≤ 7500	± 15	± 3
mg kg⁻¹	Higher range	> 7500	± 5	± 2
TotMg	Lower range	≤ 1000	± 60	± 7
mg kg⁻¹	Higher range	> 1000	± 5	± 2
TotMn	Lower range	≤ 200	± 25	± 6
mg kg⁻¹	Higher range	> 200	± 5	± 3
TotNa	Lower range	≤ 1500	± 20	± 4
mg kg⁻¹	Higher range	> 1500	± 5	± 2

Parameter	Observation Range	Level	Ring Test Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
ExtrP	lower	≤ 150	± 45	± 3
mg kg⁻¹	higher	> 150	± 20	± 3
ExtrK	lower	≤ 500	± 60	± 6
mg kg⁻¹	higher	> 500	± 40	± 4
ExctCa	lower	≤ 500	± 70	± 7
mg kg⁻¹	higher	> 500	± 30	± 3
ExctMg	lower	≤ 500	± 60	± 7
mg kg⁻¹	higher	> 500	± 15	± 3
ExctrS mg kg <sup>-1</sup>	whole	35 - 1300	± 35	± 4
ExtrNa	lower	≤ 75.0	± 65	± 8
mg kg <sup>-1</sup>	higher	> 75.0	± 50	± 6
ExtrAl	lower	≤ 2500	± 50	± 5
mg kg⁻¹	higher	> 2500	± 20	± 3
ExtrFe	lower	≤ 2500	± 40	± 4
mg kg⁻¹	higher	> 2500	± 15	± 3
ExtrMn	lower	≤ 150	± 30	± 4
mg kg⁻¹	higher	> 150	± 15	± 4
ExtrCu	lower	≤ 5	± 40	± 8
mg kg⁻¹	higher	> 5	± 15	± 4
ExtrPb mg kg <sup>-1</sup>	whole	3 - 70	± 30	± 4
ExtrNi	lower	≤ 10	± 40	± 6
mg kg⁻¹	higher	> 10	± 15	± 4
ExtrCr	lower	≤ 10	± 40	± 7
mg kg⁻¹	higher	> 10	± 25	± 4
ExtrZn	lower	≤ 20	± 40	± 7
mg kg⁻¹	higher	> 20	± 20	± 3
ExtrCd	lower	≤ 0.25	± 100	± 5
mg kg⁻¹	higher	> 0.25	± 55	± 6
ExctrHg mg kg⁻¹	whole	0 - 0.16	± 75	± 6

Table 4.1.2.2d. Tolerable limits for aqua regia extractable elements for interlaboratory comparison and intra-laboratory performance.

Table 4.1.2.2e. Tolerable limits for reactive iron and aluminium for interlaboratory comparison and intra-laboratory performance.

Parameter	Observation Range	Level	Ring Test Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Reactive Al	lower	≤ 750	± 30	± 3
mg kg⁻¹	higher	> 750	± 15	± 3
Reactive Fe	lower	≤ 1000	± 30	± 4
mg kg⁻¹	higher	> 1000	± 15	± 3

Parameter	Observation Range	Level	Ring Test Tolerable limit (% of mean)	Intra-Laboratory Tolerable limit (% of mean)
Exch Acidity	lower	≤ 1.00	± 90	± 9
cmol <sub>(+)</sub> kg⁻¹	higher	> 1.00	± 35	± 4
ExchK	lower	≤ 0.10	± 45	± 10
cmol <sub>(+)</sub> kg <sup>-1</sup>	higher	> 0.10	± 30	± 4
ExchCa	lower	≤ 1.50	± 65	± 12
cmol <sub>(+)</sub> kg <sup>-1</sup>	higher	> 1.50	± 20	± 3
ExchMg	lower	≤ 0.25	± 50	<b>± 10</b>
cmol <sub>(+)</sub> kg <sup>-1</sup>	higher	> 0.25	± 20	± 2
ExchNa cmol <sub>(+)</sub> kg <sup>-1</sup>	whole	0.01-0.14	± 80	± 14
ExchAl	lower	≤ 0.50	± 105	± 12
cmol <sub>(+)</sub> kg⁻¹	higher	> 0.50	± 30	± 4
ExchFe	lower	≤ 0.02	± 140	± 14
cmol <sub>(+)</sub> kg <sup>-1</sup>	higher	> 0.02	± 50	± 8
ExchMn	lower	≤ 0.03	± 45	± 7
cmol <sub>(+)</sub> kg <sup>-1</sup>	higher	> 0.03	± 25	± 6
Free H+ cmol <sub>(+)</sub> kg <sup>-1</sup>	whole	0.02-1.20	± 100	± 8

Table 4.1.2.2f. Tolerable limits for exchangeable elements and free acidity for inter-laboratory comparison and intra-laboratory performance.

## 4.1.2.3 Tolerable limits for plant (foliar and litterfall) ring tests

The first step in the evaluation procedure of foliage ring tests is the elimination of outliers in the results of the Needle/Leaf interlaboratory comparison test (DIN 38402/42, 1984). This method identifies three types of outlier. The Grubbs test can be used to check the four replicates from each laboratory for outliers (outlier type 1). The next step is to compare the recalculated mean values of each laboratory with the mean value from all the laboratories, as well as with the Grubb test for outliers (outlier type 2). Finally, the recalculated standard deviation from the laboratories must be compared with the total standard deviation (F-test) in order to eliminate laboratories with an excessive standard deviation (outlier type 3). The outlier-free, total mean value and the outlier-free maximum and minimum mean value of all the laboratories can then be calculated. Marked type 1 outliers between the outlier-free maximum and minimum mean value are no longer outliers, and they can be used in further evaluation of the interlaboratory comparison test. The last step is to calculate the outlier-free statistical values (Fürst, 2004, 2005, 2006, 2007, 2008).

In the next step an outlier-free mean value for each element/sample and the laboratory mean value and the recovery is calculated, and the results are compared with the tolerable limits given in Table 3. These tolerable limits for foliage samples were adopted by the Forest Foliar and Litterfall Expert Panel at the Meetings in Ås (1994), Vienna (1997), Bonn (1999), Prague (2003), Madrid (2007) and Hamburg 2009.

Table 4.1.2.3a: Tolerable limits for normal concentrations of mandatory and optional elements

Element	Tolerable deviation	Expert Panel-Foliar and Litterfall Meetings	
	from mean in % where the fixed limits were adopted		
Ν	90-110	6th Meeting - Bonn 1999	
S	85-115	10th Meeting – Madrid 2007	
Р	90-110	10th Meeting – Madrid 2007	
Ca	90-110	10th Meeting – Madrid 2007	
Mg	90-110	10th Meeting – Madrid 2007	
К	90-110	10th Meeting – Madrid 2007	
Zn	85-115	8th Meeting - Prague 2003	
Mn	85-115	8th Meeting - Prague 2003	
Fe	80-120	6th Meeting - Bonn 1999	
Cu	80-120	8th Meeting - Prague 2003	
Pb	70-130	6th Meeting - Bonn 1999	
Cd	70-130	6th Meeting - Bonn 1999	
В	80-120	6th Meeting - Bonn 1999	
С	95-105	6th Meeting - Bonn 1999	

As the concentration range in foliage and in litterfall is usually very small compared with that for soil and deposition matrices, it is not necessary to have different tolerable limits for normal and low concentrations of all the elements. Tolerable limits for some elements for low concentrations (eg. for non-foliage litterfall) is given in Table 4.1.2.3a.

Table 4.1.2.3a: Tolerable limits for low concentrations of mandatory and optional elements (adopted at the Combined FutMon/ICP-Forests meeting in Hamburg 2009)

Element	Tolerable deviation	For concentrations below
	from mean in %	
S	80-120	0.5 mg/g
Р	85-115	0.5 mg/g
Mg	85-115	0.5 mg/g
Zn	80-120	20 µg/g
Mn	80-120	20 µg/g
Fe	70-130	20 µg/g
Pb	60-140	0.5 μg/g
В	70-130	5 µg/g
Ν	85-115	5 mg/g
К	85-115	1 mg/g

Laboratory results falling inside of these limits can be accepted. Laboratories with values outside these limits need to improve their data quality.

## 4.2 Exchange of knowledge and experiences with other laboratories

The inter-laboratory comparisons conducted within the framework of the ICP Forests programme are aimed at testing the proficiency of the laboratories, i.e. evaluating the comparability of the results and, if possible, identifying the main causes of errors. The laboratories must be involved in discussions on the outcome of ring tests in order to obtain information useful in achieving, maintaining and optimizing their analytical quality.

Laboratories with unacceptable results in ring tests are invited to participate in **assistance program** organized by the WG on QA/QC in Laboratories. Close cooperation between these laboratories and laboratories with good laboratory practices is considered to be an effective way of improving laboratory proficiency.

When determining the scope of assistance it is necessary to take into account, in addition to the results of the ring test, the current state of the implementation of a quality programme, and the analytical methods used in the laboratory and described beforehand in a questionnaire filled in by the laboratory in question. The assistance consist of a few days' visit to the laboratory, as well as a return visit, in order to identify easily detectable problems in laboratory organization and/or specific analytical processes.

It is essential that the members of the staff actually involved in the analytical work in participate in the assistance programme.

A list of problems to be solved is drawn up, with the emphasis on problems linked to specific parameters analysed/determined in the ICP Forests programme. The main result of the two visits is a short report on the laboratory's activities, including problems to be solved and suggestions about how this can be achieved. The laboratory is thus provided with knowledge that enables them to make improvements in the quality of their results.

## 4.2.1 Exchange of know how

All laboratories are strongly invited to share their experience through internal **info-sheets**, developed as an easy tool for the exchange of information among laboratories about studies carried out in the laboratory which otherwise would not be published. The info-sheets are short Word files containing concise information about method comparison, development and implementation of new methods, material tests (e.g. on contamination or adsorption problems), sample pre-treatment, sample storage and technical information. Thus the work performed in one laboratory can help to avoid duplication in others.

The circulation of information within and between the WG on QA/QC in Laboratories and the all laboratories is ensured through the WG's own **website**. Information about past and ongoing ring tests, Excel files for QA/QC, scientific publications that can be downloaded, analytical info-sheets, contact addresses and useful links are to be found at http://www.icp-forests.org/WGqual\_lab.htm .

#### 4.2.2 Exchange of samples

The exchange of a limited number of routine samples between two laboratories is a simple and easy way to test the quality and comparability of the methods used. About 20 routine samples should be analysed in each

laboratory and the results compared. This ensures that differences in the methods used and analytical problems can be quickly and easily identified, and steps taken to rectify the situation.

## 5. Quality indicators

The development of the quality over time can be followed by using quality indicators.

Several quality indicators are possible; for the evaluation of the quality development of the European laboratories within ICP Forests and the FutMon project only 3 indicators have been chosen:

- 1. Percentage of the results of a ring test within tolerable limits for each ring test
- 2. Percentage of the results of a ring test of repeatability below 10% (not for water ring tests)
- 3. Mean percentage of parameters for which laboratories use control charts

The first two of them can be inferred from results of the ring tests. The third one must be obtained from laboratories (as e.g. an answer submitted with the ring test results or from the quality report forms, see chapter 6).

# 5.1 Percentage of the results of a ring test within tolerable limits for each ring test

For each ring test the number of results within the tolerable limits for all mandatory parameters of all participating laboratories is assessed and compared to the total number of results.

The percentage of results within the tolerable limits should increase over the years.

It has to be decided whether the number of results within the tolerable limits has to be compared to the total number of submitted results within a ring test or to the total number of possible results for all mandatory parameters. In the second case missing results for mandatory parameters are counted as results outside the tolerable limit.

## 5.2 Percentage of the results of a ring test of repeatability below 10%

Normally the repeatability within a laboratory should be below 10 % for all parameters. In ring tests each sample typically has to be analysed 3 or 4 times (except water samples). Therefore the repeatability for each parameter of each sample can be calculated from the ring test results. The number of calculated repeatabilities below 10 % has to be compared to the total number of calculated repeatabilities.

The percentage of repeatabilities below 10 % should be between 90 and 100 % and should stay there over time.

# 5.3 Mean percentage of parameters for which laboratories use control charts

Control charts are a useful tool for checking the quality and the variation in quality over a longer time scale for routine analyses (see chapter 2). For each parameter and each matrix a laboratory has to use control charts. To force the use of control charts for all parameters it was decided to take the percentage of parameters for which laboratories use control charts as a quality indicator for the next years. In the future all laboratories have to send a yearly quality report together with the data submission. In this report (see chapter 6) each laboratory has to submit for each parameter in each matrix the mean and the standard deviation of regularly measured reference materials (CRM or LRM). From this report the percentage of parameters for which control charts are used can be calculated for each laboratory. The mean percentage of all laboratories is an indicator of better within laboratory quality control and should develop up to 100 % during the next years.

#### 6. Quality reports

In the ICP Forests program and the FutMon project millions of analytical data are submitted to central European databases. But there is poor information about the quality of the data. With a defined quality report submitted together with the yearly data submission it is possible to link quality information to the data in a database. For the linkage the quality report must have the same base information as the data submission report (e.g. plot No., country code, year, lab code). The quality information parameters which have to be reported are:

- country code
- year
- plot No.
- lab code
- LOQ for each parameter (if needed)
- detection method (coded like in ring test reports) for each parameter,
- ring test No
- % of results within tolerable limits for each parameter
- requalification information (yes/no)

- mean and standard deviation (%) for each parameter from control charts (if a laboratory use more than one control chart for a parameter it has to submit only data from one control chart in a normal concentration range)

The quality report forms are designed in a similar way as the data submission forms (see submanual data submission).

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## 8. Annexes

## 8.1 Excel worksheet for ion balance (with and without DOC correction), conductivity, N balance and Na/CI ratio checks.

The Excel worksheet permits different quality checks to be performed, as described in the text (Chapter 3.1). It can be downloaded from the ICP Forests website (<u>www.icp-forests.org/WGqual\_lab.htm</u>): click on "excel file for analytical data validation". It can be used as a tool for validating the results and as a file for data storage, according to the requirements of the operator and the procedure for data handling in the laboratory. The sheet contains green cells in which new data are to be entered <u>using</u> the units given at the top of the column. The units are the same as those in the ICP Forests database, and the correct use of units is essential for all further checking (ion balance, measured/calculated conductivity check etc.) of the results. Information about the type of sample (BOF, THR, STF) and the type of forest cover on the plot (BL = broadleaves, CON = conifers) is required for DOC correction of the ion balance calculation. They are used as strings for the calculations, and therefore they must be entered correctly.

After entering the data in the green cells, the sheet calculates the **ion balance** (in accordance with the method described in Chapter 3.1.1.1) and the **calculated conductivity**, with and without correction for the ion strength (Chapter 3.1.2). The results of the tests are expressed in the worksheet as OK (test passed) or NO (test not passed) in the columns highlighted in yellow. The **DOC contribution to ion balance** is calculated using the empirical regressions described in Chapter 3.1.1.2. Selection of one the three alternative regression equationss is based on the codes depicting the type of sample and the type of forest cover, as given in Table 3.1.1.2a.

The principles and validation criteria for the **Na/Cl ratio** and **N forms balance** (i.e. N balance check) are described in Chapters 3.1.3 and 3.1.4. The **graphs** help in interpreting the results and identifying outliers. There are three graphs in the Excel worksheet: one for the ion balance, one for the comparison between measured and calculated conductivity, and one for the Na/Cl ratio. Other graphs can easily be added by the analysts themselves, e.g. for the comparison between measured conductivity and sum of anions or sum of cations, and the conductivity corrected for the contribution of H+ and the sum of cations, with H+ excluded (Figures 3.1.1.1a, b).

The Excel worksheet includes a sheet (**notes**) giving the meaning of the acronyms and a summary of the adopted validation criteria.

The theoretical and statistical bases applied in developing the validation criteria for deposition data in the worksheet are based on thousands of full analysis sets provided by different laboratories, and are representative of different forest types and climatic conditions in Europe, ranging from Northern Finland to Southern Italy. The results of this work have been published in two papers (Mosello et al., 2005, 2008).

## 8.2 Excel worksheet for control charts

The Excel worksheet described in Chapter 6.2 cab be used for creating control charts (paragraph 2.1). It can be downloaded from the ICP Forests website (<u>www.icp-forests.org/WGqual\_lab.htm</u>): click on "Excel file with instruction and example of control chart use". It also includes instructions on how to use the worksheet.

Reference	Matrix	Туре	Comments	Supplier
material				
BCR-408	water	simulated rain water	low concentrations	European Commission, Directorate-General Joint Research Centre Institute for Reference Materials and Measurements Reference Materials Unit Retieseweg 111 B-2440 Geel Belgium E-Mail: jrc-irmm-rm- sales@ec.europa.eu Webpage: www.irmm.jrc.be Order by Fax: +32 (0)14 590 406
BCR-409	water	simulated rain water	high concentrations	see above
BCR-100	plant	beech leaves		see above
BCR-062	plant	Olea europea (olive leaves)		see above
BCR-129	plant	powdered hay		see above
BCR-141R	soil	calcareous Ioam soil		see above
BCR-142R	soil	light sandy soil		see above

#### 8.3 List of commercially available reference materials

BCR-143R	soil	sewage sludge amended soil	heavy metal pollution	see above
BCR-146R	soil/organic material	sewage sludge of industrial origin	heavy metal pollution	see above
BCR-320	soil	river sediment		see above
FSCC RM1	soil	loamy forest soil	moderate concentrations	ICP - Forest Soil Coordinating Centre Gaverstraat 4 9550 Geraardsbergen Belgium
1575a	plant	pine needles		Standard Reference Materials Program, National Institute of Standards and Technology 100 Bureau Drive, Stop 2322 Gaithersburg, MD 20899-2322 USA E-Mail: srminfo@nist.gov Webpage: www.nist.gov/srm Order by Fax: (301) 948-3730
1515	plant	apple Leaves		see above
1547	plant	peach Leaves		see above
1570a	plant	spinach leaves		see above
1573a	plant	tomato leaves		see above
Sample 2 from the 8 <sup>th</sup> needle/leaf inter- laboratory test (ICP Forests)	plant	spruce needles		Federal Research and Training Centre for Forests, Natural Hazards and Landscape M. Alfred Fürst Seckendorff-Gudent Weg 8 A-1131 Vienna Austria E-Mail: alfred.fuerst@bfw.gv.at Web: www.ffcc.at Order per fax: +43-1- 87838-1250
Sample 4 from the 6 <sup>th</sup>	plant	maple leaves		see above

needle/leaf inter-		
laboratory test		
(ICP Forests)		

**AMENDMENTS compared to V1:** 

**Rephrasing mainly in 1<sup>st</sup> paragraph** 

#### Field protocol on continuous measures of forest growth

Action Group D1: Tree vitality and adaptation

In addition to the five-year periodic growth measurements - which will be continued - within demonstration action D1 a continuous measurement of diameter on a sub-sample of trees is expected. Continuous measurements may be realized by either of two methods: (1) by electronical dendrometers **and/or** (2) permanent girth bands, to be read manually. Both allow not just the recording of **annual** increment values but also the **distribution of increment** and **swelling** and **shrinking** of the bark and wood during the year with different resolution in time and different need for technical infrastructure. They serve, therefore, to identify stem **growth** and tree **physiological** reactions to seasonal climatic conditions, in particular **water availability**. Stem growth is one indicator of tree condition and tree vitality (Dobbertin 2005) and thus an essential measurement in D1. Where electronic girth bands are installed it is strongly recommended that they should be complemented with manually read girth bands.

While five-year tree growth should be measured on all trees in the (sub)plot (for details see the ICP Forests Manual on Forest Growth, which will be revised until 2010), trees for growth bands are selected according to different criteria. To be representative of the stand however, trees should be selected according to the observed diameter distribution across the plot, and should allow the estimates of both the relative growth of the stand and the uptake of carbon to be calculated. Selection of trees should allow comparison with periodic measures of increment (five year intervals measured on all trees in the plot).

#### Permanent measurements with manually read girth bands

#### Tree selection procedures

It is recommended to select at least **fifteen** trees of the main tree species of the plot for measurement. If it is a mixed stand then the species may be selected in proportion of their percentage in the canopy. Trees can be selected randomly or stratified randomly using the diameter distribution of all trees in the plot, or using the social class (i.e. selecting dominant or co-dominant) or both of the latter two. For example, trees could be ordered by stem basal area and total basal area cumulated. Trees will then be randomly selected from within certain even sizes proportion of this cumulative proportion (for example 3 trees from the lowest 20% of the basal area distribution, 3 trees from the next 20% etc.). Bear in mind that to end the sampling interval with data from for example ten trees it may be prudent to install more than ten bands as in a given year some may be lost or damaged due to e.g. animal or other disturbance.

The stems of the selected trees should not have any visible damage or injuries at the measuring height, should have no branches or knots at the measuring height and no visible resin flow from above. Their cross section should be as circular as possible with little irregularities. Avoid extreme thick bark if possible.

#### **Installation of the girth bands**

For trees with naturally thick bark or bark that peals of in chunks, the outer bark has to be carefully removed. This should be reported as comment in the submission forms. This can happen with a knife or sickle for the larger parts and with a steel brush to remove loose dead bark. Mosses and lichens should also be removed around the stem where the circumference band will be placed. Care should be taken not to remove or disturb the live bark.

The measurement instrument (girth band) consists of a band (metal or plastic), a spring and a scale (preferable a Nonius scale - also called vernier scale - which allows accuracies to 0.1 mm). Short springs are favourable and spring tension at installation is important. The band may need to be moved on the tree until it fits tightly and well. The girth band should be selected with an appropriate length and the spring positioned to allow measuring for several years without having to readjust the spring or replace the band. The spring should be placed in a way that only the end of the spring facing the Nonius scale need to be replaced and can be hooked into the next hole. By this way the band remains tight around the stem.

The position on the stem should be permanently marked on the stem and should be slightly above breast height to not infer with the periodic measurements. To see if the girth band had been moved along the stem, it is useful to mark the position of the band at two to three points along the stem with a color spray. When spraying, cover the girth band, otherwise you won't be able to read the scale anymore and don't spray to close to the Nonius scale. If necessary you can repeat the spraying during the annual reading. Control the tightness and the position of the spring each time you perform a reading. Make sure that the spring is not touching the Nonius scale. In stands with high UV radiation, plastic bands my most likely weaken sooner und should be checked more frequently (bleaching of the color is an obvious sign of such weakening of the material).

#### **Timing of Installation and Reading**

Installation should ideally take place in winter which would allow trees with shrinking and swelling during and following frost events to 'adjust' into the girth bands. When readings are made several times during winter a stable reading may be obtained. If the band is not tightly fixed to the stem, the first-year reading will underestimate growth. When bands are installed in late winter or early spring some tree species may show bark shrinkage and first-year growth may be overestimated. For annual readings it seems that in central and northern Europe mid to late fall is the best date to compare readings (less influence due to drought shrinkage and frost shrinkage). In southern Europe other dates may be better suited.

#### **Sampling Intervals**

Sampling intervals may vary from annual to weekly recordings. It may be useful to carry out readings at four-weekly (monthly or even two weekly) intervals when collections of deposition samples at the plot are being carried out. Sampling frequency may be increased during the growing season. It is strongly recommended to conduct measurements at least monthly during the growing season. For annual increment calculations the values of approximately the same date in the autumn of every year should be selected. Air temperature at the time of reading should be recorded.

#### Continuous recording of stem changes with electronic dendrometers

Dendrometer can be separated into point dendrometer (the radius change at one point of the stem is measured) and band dendrometers (a wire or metal tape is placed around the stem to measure its changes). For detailed review we refer to the attached publication by Drew and Downes in press). Automatic dendrometers are typically connected via cable to a data logger, which collects the reading. The number of trees that can be measured is restricted by the capacity of data logger. Readings may be collected, for example hourly (or even every 15 minutes!) and either stored directly or first averaged and stored. One at the moment frequently used dendrometer band (UMS München) uses a Teflon mesh, which is placed between the bark and the invar steel cable.

#### Sample tree selection

As dendrometers are expensive, require a central data logger and their distribution is therefore restricted by cable length, only few trees per stand and fewer sites will be selected. Therefore sampling can not be representative for the stand growth as in the case for periodic measurement or girth bands. Instead, trees should be selected to be dominant or codominant, because small or understorey trees grow slow and often have higher seasonal fluctuations of stem diameter due to swelling or shrinking of the bark than annual growth.

#### **Installation of band dendrometers**

Installation of band dendrometers follows essentially the same procedures than that for the manual girth bands (see above).

A Teflon mesh can also be used around the tree to reduce the friction of the cable and to protect it from icing, resin or callousing. Expansion and contractions of the tree stem is recorded here via a strain-gage clip-censor as the change of measured voltage (or amperage if a measuring amplifier is used) of the strain gage as a function of the change of the clip. This change is stored in the data logger and therefore the voltage must be known to calculate voltage change to increment values. The UMS D6 band has the advantage that it can be easily fixed to the trunk without any damage to the bark or disturbance of growth. Most other dendrometers have to be screwed to the tree stem. Disadvantages of dendrometer bands are that they are somehow more sensitive to temperature changes due to the long cable or bands and also to disturbance by animals, snow or ice.

Point dendrometers have the disadvantage that they require screws to fix them permanently to the stem. This can in the long-run alter the recorded growth due to increased callus cells. Therefore, point

#### field prot growth v2 270509.doc

dendrometers should not be installed closed to where the periodic measurements are conducted. The advantage of point dendrometers is that the bark needs to be removed at only one position around the stem. They may also be less sensitive to temperature changes and can be more easily protected against biotic or abiotic damages. Continuous electronic dendrometers may be used with measuring amplifiers or without. These amplifiers are recommended as they allow cables of more than 10m to be used.

#### Sources of error

It is important to remember that there are sources of error associated with continuous dendrometers, such as frost expansion, animal disturbance, electrical errors and general damage. Therefore, one band should be installed to a fixed material to test the effect of the instrument on temperature changes (i.e. a rock or a plastic tube of deposition sampling). All electronic dendrometers needs cables and these may be damaged by animals. Remotely, automatical uploading of data at certain intervals will help to detect malfunctioning of the dendrometers.

#### Note:

It is strongly recommended that manually read girth bands are also installed along with electronical dendrometers in the event of damage or failure of equipment or power supply in order to adjust the relative stem changes to actual stem changes. The dendrometer should be installed above the manually read girth band to avoid disturbance.

#### Accuracies

Manually read girth bands should allow an accuracy of reading of 0.1 mm

Electronic dendrometer should reach a recording accuracy of 0.02 mm (Accuracy of UMS Clip sensor is 5  $\mu$ m).

## FUTMON FIELD PROTOCOL PHENOLOGY (D1)

## V1.1; last update 19<sup>th</sup> May 2009

## Amendment index (compared to V1 from 15<sup>th</sup> May 2009):

- "form QQQ" at start of Annex 1 replaced by "form .PHE"
- codes for score of "Intensity of flowering and damage" were updated to 6, 7, 7.1, 7.2, and 7.3 in Annex 1
- "NFC" at end of Annex 2 replaced by "associated beneficiary"

Phenological observations under FutMon Action D1 can be made in two different ways, manually or using digital cameras.

The phenology observations are made according to the guidelines provided in chapter 9 "Phenological Observations" of the ICP Forests Manual. However, the following points/exceptions should be taken into account:

- Both plot level and single tree level observations are accepted, but wherever possible single tree level phenology should be preferred.
- For plot level observations the guidelines were revised and the data submission form (.PHE) was updated.
- For single tree observations the minimum required frequency is once a week during the critical phases, but daily observations is the optimum. For the selection, trees are preferred that are also selected for D1 girthband measurements.
- For the registration of the trees selected the form (.PLP) has been updated.
- Guidelines for the recording of the events at single tree level are revised. For the submission of the data the updated form .PHI has to be used.
- For the field observations **flushing** and **autumn colouring** are mandatory, the other events are optional. When using cameras all events are mandatory.
- For the use of digital cameras guidelines are given below. For the registration of the observed trees and the analyses of the pictures the same guidelines and forms as for the single tree observations should be used. Data should be submitted at a daily basis.
- The use of cameras is recommended for remote plots where the use of relatively expensive camera systems helps to save a lot of travel costs; in order to fulfil the FutMon contract each associated beneficiary is recommended to test at least one camera system.
- As far as biotic damage is observed during the phenology assessments its appearance is submitted with the phenology data as far as foreseen with the updated forms .PHE and .PHI. The more detailed examination, assessment and data submission on the observed damage has to be done according to the ICP Forests manual on damage assessment. Whenever needed, trained staff should be consulted for damage assessment within the next 4 weeks beginning from the phenology assessment.

#### Annexes:

Annex 1: revised guidelines for recordings Annex 2: guidelines for the use of cameras revised forms are presented on FutMon webpage in a separate excel file on forms and explanatory items

## Annex 1: revised guidelines for recordings

## 1 Recording of phenological events at the plot level

For the recording of the phenological phenomena at the plot level form .PHE is used.

The event codes for the monitored effects and phenological phenomena are:

- 1 = Flushing;
- 2 =Colour changes;
- 3 = Leaf/needle fall;
- 4 = Significant signs of leaf or crown damage (e.g., eaten leaves or bare crown parts);
- 5 = Other damage (breakage, uprooted trees);
- 6 = Lammas shoots / secondary flushing;
- 7 = Flowering.

#### Scoring system

Occurrence of the events and phenomena (proportion of the forest crown affected):

- 1 < 1% (not to be applied on flowering and damage, codes 7, 4, and 5)
- 2 = 1 33% (not to be applied on flowering and damage, codes 7, 4, and 5)

3 = >33 - 66% (not to be applied on flowering and damage, codes 7, 4, and 5)

4 = >66 - 99% (not to be applied on flowering and damage, codes 7, 4, and 5)

5 > 99% (not to be applied on flowering and damage, codes 7, 4, and 5)

- Intensity of flowering and damage (optional quantifiaction) (NEW!)
- 6 = Flowering / damage absent
- 7 = Flowering / damage present
  - 7.1 = flowering sparse (optional)
  - 7.2 = flowering moderate (optional)
  - 7.3 = flowering abundant or mast (optional)

#### 2 Recording of phenological events at the individual tree level

2.1 Registration of trees selected

For the registration of the trees selected for the recording of phenological events form .PLP is used.

The <u>part of the crown</u> observed (here: the visible part of the crown) should be reported at the time the trees are selected, or whenever it changes, using the following codes (same as described in ICP Forests Manual):

- 1 = top of the crown
- 2 = middle of the crown
- 3 = top and middle of the crown.

The codes for the direction <u>from</u> which observations are made are (same as described in ICP Forests Manual): 1 = North

- 1 NOLUL
- 2 =north-east
- 3 = East
- 4 =south-east
- 5 =South

- 6 =south-west
- 7 = West
- 8 = north-west

The codes for the vertical direction <u>from</u> which the observations were made are (NEW!):

- 1 =from below
- 2 = at crown level
- 3 =from above

## 2.2 Recording of phenological phenomena

For the recording of the phenological phenomena at the single tree level updated form .PHI is used.

The method used for making the observations

- 1= field observation
- 2= Digital camera
- 3= Both field observation and digital camera

The event codes for the monitored effects and phenological phenomena are:

- 1 = Flushing;
- 2 = Colour changes;
- 3 = Leaf/needle fall;
- 4 = Significant signs of leaf or crown damage (e.g., eaten leaves or bare crown parts);
- 5 = Other damage (breakage, uprooted trees);
- 6 = Lammas shoots / secondary flushing;
- 7 = Flowering.

## Scoring system

Occurrence of the events (proportion rate of tree compartments affected):

- 1 < 1% (not to be applied on flowering and damage, codes 7, 4, and 5)
- 2 = 1 33% (not to be applied on flowering and damage, codes 7, 4, and 5
- 3 = >33 66% (not to be applied on flowering and damage, codes 7, 4, and 5)
- 4 = >66 99% (to be applied on flowering and damage, codes 7, 4, and 5)
- 5 > 99% (to be applied on flowering and damage, codes 7, 4, and 5)

Flowering phases:

The number of male flowers that are in the described stage or have already passed this stage is to be recorded using the following classification:

Intensity of flowering and damage (optional quantifiaction) (NEW!)

- 6 = Flowering / damage absent
- 7 = Flowering / damage present
  - 7.1 = flowering sparse (optional)
  - 7.2 = flowering moderate (optional)
  - 7.3 = flowering abundant or mast (optional)

## Needle appearance, leaf unfolding, autumn colouring and leaf fall:

The proportion of needles or leaves of the visible part of the crown that are in the described stage or have already passed this stage is to be recorded using the following classification:

0 = 0% 1 = >0 - 33% 2 = >33 - 66% 3 = >66 - <100%4 = 100%

#### 3 Recording of biotic or abiotic damaging events

Reporting of observed biotic damage will be done using event code 4 or 5 respectively for form .PHE and .PHI, respectively. For damage description the form .TRD and the respective coding described in the ICP Forests manual on Crown condition and biotic damage assessment (Part II) are used.

#### Annex 2: guidelines for the use of cameras

#### Advantages of the use of cameras:

- Enables frequent (continuous) monitoring, also on remote sites
- Assessments can be made any time when staff is available
- Enables comparison between sites
- Improves comparison between years
- Enables comparison between countries/regions
- Enables better timing of appearance of damages

Points to be considered

- High investment costs
- Need for power supply
- Difficult in dense (conifer) stands
- Possible technical failures
- Possible vandalism

#### The use of digital cameras for monitoring phenology

When using digital cameras the first priority should be that the quality of the pictures (resolution) obtained allow the assessment of phenological phases at individual tree level according the guidelines in the manual on phenological observations with 33% classes. In addition also other aspects of the crown such as damages can be assessed. At each plot at least 10 trees per species should be assessable (tree selection as mentioned above in this protocol).

#### Technical requirements

The cameras should be weather-resistent, e.g outdoor surveillance cameras are suitable. Important is that the pictures are of high resolution (minimum requirements 6 Mpix with 300 pix/inch / 120 pix/cm), even with full zoom properties. The camera should have its own memory, or be connected to a datalogger. The datalogger and steering unit should be stored in a weather-proof place, and the whole system should be protected against lightning. Power supply can be obtained through batteries, solar panels or connection to the electricity network. The working of the camera should be checked every time the plot is visited.

#### Location of the camera

If possible the camera should be mounted to a mast that reached over the top of the crowns, e.g. the towers used for the meteorological assessments. In order to be able to observe a number of trees, the camera should be movable and programmable so it can take pictures of the same spot at regular intervals. The position of the camera is selected so that it can cover an optimal number of individual trees within the plot at a area as large as possible. The observations can also be made from below the crowns, but this way the area, and the number of visible trees per camera is more limited. Alternatively more than one camera could be used. The camera should take pictures of the whole upper part of the crown. Trees around the camera are selected and registered using form 12a. For each tree also the part of the crown observed, as well as the direction from which the camera takes the picture are marked. The codes used are the same as for the manual single tree observations.

#### Data handling

Pictures should be taken a number of times each day (at least 2) because the light conditions change during the day because of the position of the sun. At least every 2 months the data should be collected from the plots in order to secure the data. The camera can also be connected to a network, so the observations can be made at distance. In this case it is still advisable to have the pictures also stored at the plot for backup.

The pictures of the different plots should be analysed by one and the same person, or at least for the different plots of each tree species. This way the effect of the observer is eliminated. The assessments should be made using the same stages and codes as used for the field observations. Only one observation per day should be made.

The pictures should be stored by the associated beneficiaries so they can be used later for inter-calibration as well as for comparison between countries. During the phenological phases to be assessed for each tree at least one picture per day should be saved. For the rest of the growing season at least one picture per week is sufficient. If pictures are also taken during winter also one picture per week can be stored. At form xx metadata about the stored pictures should be submitted annually.

Pictures should be available to other partners of the project. In order to enable for an easy data access the photos and movies may be stored in addition at the FutMon data centre. In order to allow for a consistent and uniform identification and submission of the digital images the form .PHD and a respective Explanatory Item are to be applied.

## Field protocol on permanent and continuous measures of forest growth

Action Group D1: Tree vitality and adaptation

In contrast to the five-year periodic growth measurements, permanent and continuous measurements allow not just the recording of **annual** increment values but also the **distribution of increment** and **swelling** and **shrinking** of the bark and wood during the year. They serve, therefore, to identify stem **growth** and tree **physiological** reactions to seasonal climatic conditions, in particular **water availability**. Stem growth is one indicator of tree condition and tree vitality (Dobbertin 2005) and thus an essential measurements in D1.

While five-year tree growth should be measured on all trees in the (sub)plot (for details see the ICP Forests Manual on Forest Growth, which will be revised until 2010), trees for growth bands are selected according to different criteria. To be representative of the stand however, trees should be selected according to the observed diameter distribution across the plot, and should allow the estimates of both the relative growth of the stand and the uptake of carbon to be calculated. Selection of trees should allow comparison with periodic measures of increment (five year intervals measured on all trees in the plot).

#### Permanent measurements with girth bands

#### **Tree selection procedures**

It is recommended to select at least **fifteen** trees of the main tree species of the plot for measurement. If it is a mixed stand then the species may be selected in proportion of their percentage in the canopy. Trees can be selected randomly or stratified randomly using the diameter distribution of all trees in the plot, or using the social class (i.e. selecting dominant or co-dominant) or both of the latter two. For example, trees could be ordered by stem basal area and total basal area cumulated. Trees will then be randomly selected from within certain even sizes proportion of this cumulative proportion (for example 3 trees from the lowest 20% of the basal area distribution, 3 trees from the next 20% etc.). Bear in mind that to end the sampling interval with data from for example ten trees it may be prudent to install more than ten bands as in a given year some may be lost or damaged due to e.g. animal or other disturbance.

The stems of the selected trees should not have any visible damage or injuries at the measuring height, should have no branches or knots at the measuring height and no visible resin flow from above.. Their cross section should be as circular as possible with little irregularities. Avoid extreme thick bark if possible.

#### Installation of the girth bands

For trees with naturally thick bark or bark that peals of in chunks, the outer bark has to be carefully removed. This should be reported as comment in the submission forms. This can happen with a knife or sickle for the larger parts and with a steel brush to remove loose dead bark. Mosses and lichens should also be removed around the stem where the circumference band will be placed. Care should be taken not to remove or disturb the live bark.

The measurement instrument (girth band) consists of a band (metal or plastic), a spring and a scale (preferable a Nonius scale - also called vernier scale - which allows accuracies to 0.1 mm). Short springs are favourable and spring tension at installation is important. The band may need to be moved on the tree until it fits tightly and well. The girth band should be selected with an appropriate length and the spring positioned to allow measuring for several years without having to readjust the spring or replace the band. The spring should be placed in a way that only the end of the spring facing the Nonius scale need to be replaced and can be hooked into the next hole. By this way the band remains tight around the stem.

The position on the stem should be permanently marked on the stem and should be slightly above breast height to not infere with the periodic measurements. To see if the girth band had been moved along the stem, it is useful to mark the position of the band at two to three points along the stem with a color spray. When spraying, cover the girth band, otherwise you won't be able to read the scale anymore and don't spray to close to the Nonius scale. If necessary you can repeat the spraying during the annual reading. Control the tightness and the position of the spring each time you perform a reading. Make sure that the spring is not touching the Nonius scale. In stands with high UV radiation, plastic bands my most likely weaken sooner und should be checked more frequently (bleaching of the color is an obvious sign of such weakening of the material).

#### **Timing of Installation and Reading**

Installation should ideally take place in winter which would allow trees with shrinking and swelling during and following frost events to 'adjust' into the girth bands. When readings are made several times during winter a stable reading may be obtained. If the band is not tightly fixed to the stem, the first-year reading will underestimate growth. When bands are installed in late winter or early spring some tree species may show bark shrinkage and first-year growth may be overestimated. For annual readings it seems that in central and northern Europe mid to late fall is the best date to compare readings (less influence due to drought shrinkage and frost shrinkage). In southern Europe other dates may be better suited.

#### **Sampling Intervals**

Sampling intervals may vary from annual to weekly recordings. It may be useful to carry out readings at four-weekly (monthly or even two weekly) intervals when collections of deposition samples at the plot are being carried out. Sampling intervals may be increased during the growing season. It is strongly recommended to conduct measurements at least monthly during the growing season. For annual increment calculations the values of approximately the same date in the autumn of every year should be selected. Air temperature at the time of reading should be recorded.

#### Continuous recording of stem changes with electronic dendrometers

Dendrometer can be separated into point dendrometer (the radius change at one point of the stem is measured) and band dendrometers (a wire or metal tape is placed around the stem to measure its changes). For detailed review we refer to the attached publication by Drew and Downes in press). Automatic dendrometers are typically connected via cable to a data logger, which collects the reading. The number of trees that can be measured is restricted by the capacity of data logger. Readings may be collected, for example hourly (or even every 15 minutes!) and either stored directly or first averaged and stored. One at the moment frequently used dendrometer band (UMS München) uses a Teflon mesh, which is placed between the bark and the invar steel cable.

#### Sample tree selection

As dendrometers are expensive, require a central data logger and their distribution is therefore restricted by cable length, only few trees per stand and fewer sites will be selected. Therefore sampling can not be representative for the stand growth as in the case for periodic measurement or girth bands. Instead, trees should be selected to be dominant or codominant, because small or understorey trees grow slow and often have a higher seasonal fluctuations of stem diameter due to swelling or shrinking of the bark than annual growth.

#### Installation of band dendrometers

Installation of band dendrometers follows essentially the same procedures than that for the manual girth bands (see above).

A teflon mesh can also be used around the tree to reduce the friction of the cable and to protect it from icing, resin or callousing. Expansion and contractions of the tree stem is recorded here via a strain-gage clip-censor as the change of measured voltage (or amperage if a measuring amplifier is used) of the strain gage as a function of the change of the clip. This change is stored in the data logger and therefore the voltage must be known to calculate voltage change to increment values. The UMS D6 band has the advantage that it can be easily fixed to the trunk without any damage to the bark or disturbance of growth. Most other dendrometers have to be screwed to the tree stem. Disadvantages of dendrometer bands are that they are somehow more sensitive to temperature changes due to the long cable or bands and also to disturbance by animals, snow or ice.

Point dendrometers have the disadvantage that they require screws to fix them permanently to the stem. This can in the long-run alter the recorded growth due to increased callus cells. Therefore, point dendrometers should not be installed closed to where the periodic measurements are conducted. The advantage of point dendrometers is, that the bark needs to be removed at only one position around the stem. They may also be less sensitive to temperature changes and can be more easily protected against biotic or abiotic damages. Continuous electronic dendrometers may be used with measuring amplifiers or without. These amplifiers are recommended as they allow cables of more than 10m to be used.

#### Sources of error

It is important to remember that there are sources of error associated with continuous dendrometers, such as frost expansion, animal disturbance, electrical errors and general damage. Therefore, one band should be installed to a fixed material to test the effect of the instrument on temperature changes (i.e. a rock or a plastic tube of deposition sampling). All electronic dendrometers needs cables and these may be damaged by animals. Remotely, automatical uploading of data at certain intervals will help to detect malfunctioning of the dendrometers.

#### Note:

It is strongly recommended that permanent girth bands are also installed along with continuous dendrometers in the event of damage or failure of equipment or power supply in order to adjust the relative stem changes to actual stem changes. The continuous dendrometer should installed above the permanent girth band to avoid disturbance.

#### Accuracies

Manual girth bands should allow an accuracy of reading of 0.1 mm

Electronic dendrometer should reach a recording accuracy of 0.02 mm (Accuracy of UMS Clip sensor is 5  $\mu$ m).

#### **Expert Panel Crown Condition and Assessment Damage Causes**

#### **Tree Vitality (D1)**

#### FutMon Field Protocol

V 1.0; last update 15<sup>th</sup> May 2009

At the D1 section of the FutMon meeting in Hamburg January 2009, a mutual agreement was achieved, to define some parameters to be mandatory on D1-plots, which have proven to indicate tree vitality by literature. The field protocol and the decision of mandatory assessments of these parameters are mainly related to FutMon D1 plots in the years 2009 and 2010. In addition the parameters are optional for IM1 (ICP forests Level II) and ICP forests Level I-plots.

The additional definition of assessable crown will be used in 2009 and 2010 only at the ICCs.

A new form is prepared for special use at D1 plots (XX2009.D1T; see forms document). Some of the new variables use a new coding which has to be used instead of definitions in the existing Sub manual Crown (www.icp-forests.org). However, in addition the traditional forms have to be filled out with the existing ICP Forests coding and in addition the FutMon participants are asked to assess the new parameters and submit the data for the D1 plots. Thus, the reporting for the testphase2009 – 2010 will be done using both the existing methods and also the new/alternative methods for the D1 plots. Training and discussion of the new parameters will be part of the ICCs 2009 and 2010. It is suggested that experienced participants could give a short introduction of the use of these variables during the ICCs. This will be discussed and agreed with host countries before ICCs.

The tree species of Fagus sylvatica is brought into focus and used as an example for a new concept of tree vitality evaluations. Some indicators have also shown to be relevant for other tree species. Therefore other tree species are included as well.

#### Content

- 1. Assessable crown (only at ICCs 2009, 2010)
  - 1.1 Definitions
  - 1.2 Field position of assessment
- 2. Tree age (mandatory D1 plots)
- 3. Fruiting
  - 3.1 Fruiting of Fagus ssp., Picea abies and Pine ssp. (terrestrial inventory; mandatory D1) 3.2 Fruiting of Beech and Oak (mandatory D1 litter fall assessments)
- 4. Crown diameter related distance to neighbours (mandatory D1)
- 5. Apical shoot architecture (ROLOFF) (mandatory D1; Fagus sylvatica)
- 6. Species
- 7. New concept tree removals and mortality (mandatory D1 plots)
- 8. References

## 1. Assessable crown (only at ICCs 2009, 2010)

## 1.1Definitions

## **Motivation**

It has to be checked if one of the main causes for differing results in defoliation estimations in various European countries is the different understanding of what constitutes assessable crown.

## Note

The following definition is to be field-tested at the ICCs 2009 in Finland, the Czech Republic and Italy and used concurrently with the nationally differing definitions. A second test will be done at the photo ICC in 2010.

#### Method

Three definitions aiming at elimination of lower branches that have defoliation/ branch dieback due to light competition, e.g. by vicinal trees, are used in the European Crown assessment.

- a) Assessment of the tree crown ranges from the tip of the tree to the *widest span* of the crown or to where the distance between stem axis and living branches is greatest.
   Fig.1 shows good examples of this definition in the part "stand". For freely grown trees, a line indicates the lower limit of the assessable crown.
- b) Assessment of a defined lower limit; the *upper third* of a trees living crown will be assessed. The evaluations could be compared with the results "entire crown/upper third of the crown" from former ICCs which were made by participants from Estonia. From a practical view, sometimes it is very difficult to define the lowest living branches to be the lower limit of the total crown. This fact may bias the definition of upper third or half of a tree crown. In many cases countries use their own national definition of living crown before they define the assessable crown.
- c) Individual countries "*traditional*" procedure of the definition of assessable crown referring ICP Crown Manual (2006).

## 1.2) Field position of assessment

## Motivation

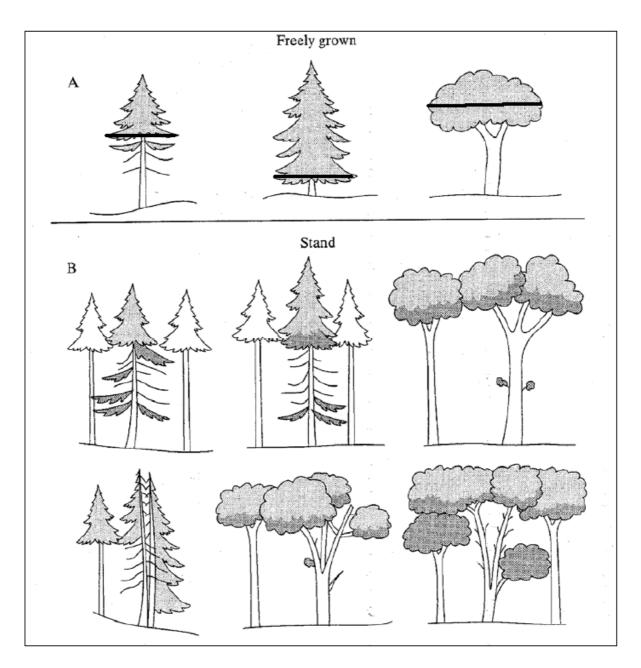
As described in the ICP Forests manual on Crown Condition Assessment, Annex on ICCs, it is necessary to evaluate the variation of defoliation scores which is due to different positions of the participants during the assessment.

## Method

The participants are asked to do the first assessments from a fixed position which has to be prepared and marked in the field by the host countries according to the same fixed positions which were used during former courses on the same plots and trees. A second group of assessments will be done following the position or assessment procedure which is used during the field assessments in the participating countries.

Thus, at ICCs on crown condition assessment, each tree will receive two X three = six defoliation scores. As most time during an ICC is needed to get to the different plots and to

find the correct position to the assessed tree the additional assessments on different parts of the crown should not lead to exhaustive time consumption.



<u>Fig. 1</u>: Illustration of definition a): Assessment of the tree crown ranges from the tip of the tree to the widest horizontal span of the crown (stand: the lighter colour indicates assessable crown; freely grown trees: black line)

# 2. Tree age (sample tree specific age for all trees used in e.g. crown condition assessment on D1 plots; mandatory)

# Motivation

Former evaluation of crown condition data have indicated that one of the main causes for differing results in defoliation estimations in various European countries is the different age of sample trees. Studies show, that even biased age estimations help to explain a substantial amount of defoliation variability.

At the D1 section of the FutMon meeting in Hamburg 2009, a subgroup recommended to include tree age in the list of parameters of D1 plots. In the plenum, a mutual agreement was achieved. It will be evaluated if the submission of tree specific age data will allow for a better interpretation of vitality data. Even if assessment accuracy is expected to be low in most cases the submission of tree specific age should help for a better understanding of stand structure during data evaluations.

# Method

For D1 plots, tree age of sample trees must be specified for all of the trees on a plot. The best exact method should be used and described, indicating also the uncertainties of this method. The method defines two new fields in a tree specific table (new "D1-tables"):

1. tree\_age

to be specified in age classes given A1.9 in the ICP Forests manual ("8" is deleted, since this parameter indicates tree age and not stand age; instead of the group >120, the new classes 121-140, 141-160 and >160 years will be added).

- 2. method\_of\_age\_determination (coding see below)
  - 1 = assured dates of stand establishment
  - 2 = tree stumps

3 = age determination of the lowermost twigs (add estimated time it has taken to grow to that height)

4 = increment borer, stem discs (from similar sized trees/median sized trees) outside the plot

- 5 = assessment (impossible in most cases)
- 6 = estimation without any exact information
- 3. free text option (comments on age assessment)

# 3. Fruiting

# **3.1 Fruiting of Fagus ssp., Picea abies and Pine ssp.** (terrestrial inventory; <u>mandatory D1</u>; in addition to Manual Crown A1.21 page 26)

# **Motivation**

Annual seed production of trees with heavy seeds such as beech and oak can cause considerable changes in internal cycles. Annual seed production may cause a significant change in allocation of carbon, nutrients and energy from leaves and stem growth to generative structures. This is an important criterion for tree vitality.

Fruiting always bears upon the assessment e.g. of defoliation as some countries reduce the expected 100% crown if fruiting occurs (e.g. on Scots pine), other countries decided that fruiting and the concurrent missing of needles must lead to a respective increase in defoliation scores.

Note

Only the fruit of the respective assessment year is to be considered.

Pine: only green cones.

Level 1 = 1000, will, in comparison to the sub-manual Crown, be distinguished into Level 1.1 (absent) and Level 1.2 (scarce), so that a sum of scarce (code 1.2) – medium (2) – high (3) of all trees fructifying in one year can be formed. The question if the scores 1.1 and 1.2 can be distinguished by the field observer for each tree will be discussed evaluating the comment on fruiting assessment which have to be submitted in free word text in case of any problems with the parameter assessment.

# Method

Hint: The sum of the new levels 1.1 and 1.2 indicate the group "without" and "low" fruiting (code 1) described in A1.21 of the ICP Forests Manual.

1.1 = absent

Fructification is absent or inconsiderable. Even reasonably lengthy observation of the crown with binoculars yielded no signs of fruiting.

1.2 = scarce

Sporadic occurrence of fruiting, not noticeable at first sight. It must be looked for on purpose with binoculars.

2 = medium

Fructification is such that it can be observed with the naked eye. The appearance of the tree is influenced but not dominated by fructification.

3 = high

Fructification is obvious and immediately meets the eye, determines the tree's appearance.

Annual assessments have to be done both 2009 and 2010.

# 3.2 Fruiting of Beech and Oak (mandatory D1 litter fall assessments)

# **Motivation**

In order to validify traditional terrestrial assessments and in order to quantify for element budget considerations the weight of fruits and capsules in the litter should be determined. Assessment of oak fructification is generally not possible in Central Europe during the period in which terrestrial assessment takes place.

# Method

Separate biomasses of "fruits" and "fruit capsules" of beech and oak are quantified in addition to the regular litter fall measurements.

The aim is to elaborate an annual sum of fruits and fruit capsules per hectare D1 plot. Chemical analyses should also represent an annual value per plot hectare.

During the FutMon programme this survey of litter fall biomass will be conducted on Beech and Oak D1 plots (main tree species) according to the ICP Forests Manual on Sampling and Analysis of Litter fall for the sampling of litter fall and the calculation of biomass (dry mass) and the Field Protocol on Litter fall. For the submission of data the litter fall forms will be used (XX2007.LFM; see forms document). The sample codes are according to the updated reference list. Here an excerpt with the components which are of special interest for Tree Vitality:

Deciduous trees (Beech, Oak)						
			larger litter			
Leaves		Fruiting components			components	Rest
main tree	other		Fruit		Twigs and	other plant
species	species	Fruiting	capsules	rest	branches	biomass
11.1	11.2	14.1	14.2	14.3	16	19



Fig. 2: Beech fruits

**4. Crown diameter related distance to neighbours** (CDRD\_N; <u>mandatory D1 all trees with</u> <u>defoliation assessments</u>; in addition to **Manual Crown A1.15 page 21 "Crown shading"**)

Hint: This is a new variable with a new coding which is used in addition to A1.15 on D1 plots.

# **Motivation**

The distance between trees explains to a high degree the scattering of characteristic defoliation data.

# Note

Crown diameter is a relative measure used to indicate from a given tree crown stand structure in four perpendicular directions. Score values are to be averaged.

# Method

# Scores for each perpendicular direction

- 1 = cramped. Canopies overlap.
- 2 = closed. Crowns touch one another.
- 3 = loose spread. Gap between crowns *up to* one third of average crown diameter
- 4 = spread. Gap between crowns *up to* two thirds of average crown diameter
- 5 = distant. Gap between crowns from two thirds *up to* one whole of average crown diameter
- 6 = very distant. Gap between crowns > than 1/1 of average crown diameter

#### **Calculation:**

 $[Score_1 + Score_2 + Score_3 + Score_4] / 4 = CDRD_N$ 

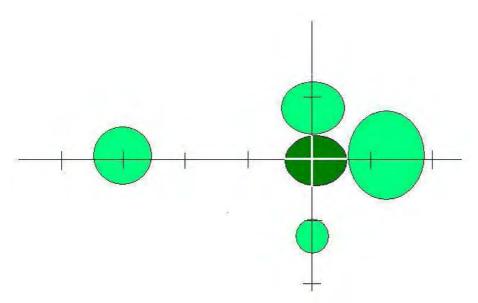


Fig. 3: Example: Crown diameter related distance to neighbours

*Example:* [2+2+6+5]/4= 3,75

#### **5.** Apical shoot architecture (<u>mandatory D1</u>; Fagus sylvatica) <u>in addition to [A1.23 Crown form/morphology</u> (optional Level II)]

# **Motivation**

The beech architecture model enables one to recognise vitality disorders in time series (Roloff (2001). Having a close look at beech branches, annual shoot length is clearly visible by signs of former buds up to 10 years. From a distance this apical shoot architecture indicates very typical growth patterns, which can be assessed using binoculars.

Note

Only the topmost twigs of a beech's crown are suitable for assessment of the apical shoot architecture. If there is a good visibility on top of the sample trees, it can be done during summer assessment. If there is only a limited view on the top of trees for example in crowded stands, it is recommended to assess apical shoot architecture and to carry out the assessment in the non vegetation period.

Annual assessments have to be done both 2009 and 2010.

# Method<sup>1</sup>

1 = Exploratory phase

Apical shoots and upper side buds form long shoots. Flat, longitudinal, expansive shoot development.

<sup>&</sup>lt;sup>1</sup> Photos: Roloff, Eichhorn; Drawings: Roloff



Fig. 4: Exploratory phase

- 2 = Intermediary form 1/3
- 3 = Degeneration phase

Only apical bud forms long shoot. Shoots of side buds are stunted. Spear-shaped development of main shoots with reduced side shoot formation "spear-shaped".



Fig. 4: Spear-shaped degeneration phase (right: from ROLOFF)

4 = Intermediary form 3/5



Fig. 5: Intermediary form 3/5

5 = Stagnation phase

Stunted long shoots, claw-like appearance because of pluriannual short shoot chains.

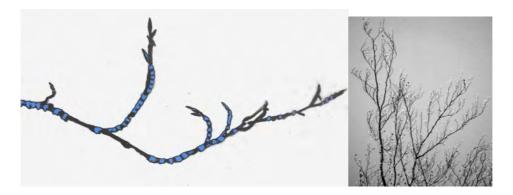


Fig. 6: Stagnation phase

6 = Intermediary form 5/7

7 = Resignation phase

Die-back of twigs of the topmost part of the crown or even the whole crown itself.



Fig. 7: Resignation phase

# 6. Species (mandatory D1)

In some genus it is difficult to assess tree species (e.g. Oak ssp.: Quercus x rosacea for the hybrid Q. robur x Q. petraea or alternatively Q. robur x petraea). In this case the respective NFC should submit one possible species and add the information on deviating phenotype in the "other observations" field and the accompanying report.

The list of broadleaved species has to be amended before new species are submitted to the data centre. The Expert Panel will do this – as it is described in the ICP Forests Manual – in co-operation with the FutMon data centre and PCC of ICP Forests and inform the respective NFC and the entire project partner community via the FutMon and ICP Forests web pages.

# 7. New concept tree removals and mortality (<u>mandatory D1</u> plots; instead of: Manual Crown A1.13, page 20)

# Motivation:

Data on removals and mortality provide essential information in the frame of forest condition monitoring. The present code list in the crown condition manual of ICP Forests (A1.13) is a combination of tree data regarding presence/absence, living/dead, symptoms and causes. Although many different situations are covered, the method has room for improvement. Regarding mortality for instance, reporting of the cause of death is only possible in broad categories, like 'biotic/abiotic reason', even when more detailed information is available (e.g. bark beetle attack, drought, ...). Therefore a different approach is proposed, based on the guidelines on the assessment of damage causes. 2009-2010 being a test phase for future forest monitoring, countries report information on 'removals and mortality' both using the existing code system and according to the alternative method for D1 plots. For other plots the additional submission of the new D1 parameter is recommended. For data submission the D1 form XX2009.D1T has to be used for submission of <removal/mortality>, <a href="#affected-part"><a href="#affected-part">affected part</a>, <symptom>, and <cause>. Evaluation of the test phase will result in one method to be continued.

# Method:

#### Definition

Removals are trees which for some reason are not included in the sample of assessment trees. Mortality refers to assessment trees which have died. A tree is defined as dead if all conductive tissues in the stem(s) have died. If a tree has died the cause must be determined (if possible). Standing dead trees (classes 30–32) of Kraft classes 1–3 should remain in the sample and should be assessed as dead trees as long as they are standing (until they are removed or have fallen down).

Reporting of removals/mortality is based on one single code, specifying if:

- the tree is present or removed;
- the tree is dead or alive;
- it is a new tree or an already existing tree in the inventory
- no assessment has been carried out for this particular tree (e.g. due to broken crown)

More information on the condition of the tree and the cause of removal/mortality is provided by using the guidelines of the submanual on the assessment of damage causes (Crown condition manual updated 06/2006 Annex 2) for the symptom description and the reporting of causes. For the symptom description and reporting of the cause the existing codes will be used. The reporting of removals/mortality is mandatory and should include: a/ code for removal/mortality, b/ symptom description, c/ cause of removal/mortality or reason why no assessment was carried out. No quantification of symptoms should be carried out for reporting removals/mortality (i.e. no information on extent).

Removals and mortality will be reported using the following codes:

- 01: tree alive in current and previous inventory
- 02: new alive tree (ingrowth)
- 03: alive tree (present but not assessed in previous inventory)
- 04: dead tree
- 05: tree is removed

- 06: tree is present and alive but no assessment could be carried out (e.g. due to crown breakage)
- 07: information on this tree is missing for this years inventory (e.g. tree was forgotten during field work)

Examples:

1/ Tree is dead due to bark beetle attack

Reporting:

a/ Removal/Mortality: tree is dead (code: 04)

b/ Symptom description:

- specification of affected part: e.g. stem (code: 32)
- symptom: signs of insects (code: 10)
- symptom specification: boring holes (code: 65)

c/ cause: stem, branch & twig borers (code: 220).

If determination of the causing organism is possible up to species level this can be reported using the existing codes of the Damage manual.

2/ Tree is dead but no symptoms can be seen providing information on the cause of death, which is unknown:

Reporting:

a/ Removal/Mortality: tree is dead (code: 04)

b/ Symptom description:

- specification of affected part: no symptoms on any part of tree (code: 00)
- symptom: blank
- symptom specification: blank

c/ cause: investigated but unidentified (code: 999).

3/ Tree is present and alive but considerable part of the crown is broken due to storm and as a result no crown condition assessment could be carried out:

Reporting:

a/ Removal/Mortality: tree is present and alive but no assessment could be carried out (code: 06)

b/ Symptom description:

- specification of affected part: branches  $\geq 10$  cm diameter (code: 24)
- symptom: broken (code: 13)
- c/ cause: e.g. wind (code: 431)

If the tree is no longer assessed e.g. because it is no longer in Kraft classes 1 - 3, the reported cause will be: competition (code: 850).

4/ Tree is removed due to planned utilisation (e.g. thinning):

Reporting: a/ Removal/Mortality: tree is removed (code: 05) b/ Symptom description:

- specification of affected part: no symptoms on any part of tree (code: 00)
- symptom: blank

c/ cause: sylvicultural operations or forest harvesting (code: 540)

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# FutMon (Life+) field protocol:

# Sampling procedure for evaluation of nutrient budgets in vegetation in FutMon intensive monitoring plots and more intensive foliage surveys (D2)

V 1.0; last update 15<sup>th</sup> May 2009

#### 1. Background

Even though understorey vegetation usually represents a relatively minor component of the whole biomass of high forests, it can play an important role in the annual biomass production and hence also in the nutrient cycling of the forest ecosystem. Furthermore, compared to the tree foliage (especially from evergreen trees), the litter of deciduous dwarf shrubs, herbs and grasses has a faster rate of decomposition. This means that the litter produced by the understorey vegetation is likely to have a major impact on soil microbiological processes, possibly with a different seasonal pattern to the canopy trees. In addition, vegetation cover protects the soil from erosion and alters its moisture content and temperature. This is particularly important in complex forests with tall shrub undergrowth (> 2m ht), such as may be found under stands of deciduous oak or ash. Consequently knowing the nutrient content and biomass of different components of standing ground vegetation. Together with information about deposition, soil water and soil nutrients nutrient budgets in forest vegetation.

The aim of this protocol is to present a methodology to yield comparable and reliable information about living biomass and nutrient concentrations of ground vegetation in FutMon intensive monitoring (IM) plots at Pan-European level. Together with ICP Forests manual part IV (Sampling and analysis of needles and leaves) this protocol will help to evaluate the nutrient budgets needed in FutMon Action D2. Estimates of biomass per species may be expanded to plot level through the results of the ground vegetation surveys (i.e. species list and cover/abundance values). Ideally different species present in each IM plot should be analysed separately but, especially in species rich plots, this would not be economically feasible. Therefore, in this protocol species have been amalgamated into seven groups on the basis of their structure and/or function so that species that have presumably similar life cycles, decomposition rate etc. belong to same functional group (see details in chapter 3.4). The underlying goal of the methods below is to have reliable estimates of living biomass and nutrient concentrations of these species groups expressed per known ground surface area.

# 2. Definitions

Here ground vegetation refers to:

- ground layer (terricolous bryophytes and lichens) and
- field layer (all non-ligneous and ligneous plants < 0.5m height)

Ligneous plants exceeding 0.5m height belong to:

- shrub layer (ligneous plants of > 0.5m height) or
- tree layer (ligneous plants of > 5m height)

Note that the "shrub layer" (i.e. ligneous plants exceeding the 0.5 meters) in the protocol means tree and bush species that exceed 0.5 m. Hence dwarf shrubs (e.g. Vaccinium, Calluna, Empetrum etc.) possibly exceeding 0.5 m belong to field layer and should thus be sampled and analysed.

# **3.** Destructive sampling, biomass weighing and pre-treatment of living ground vegetation before chemical analysis

This sampling design pertains only to understorey vegetation of less than 50 cm height as recorded under the Level II protocol for site ecology. The field sampling should be undertaken by or under the supervision of an experienced plant ecologist.

# 3.1 Time of year.

Sampling should be aimed at attaining maximum biomass values at the peak growing period. This might require more than one sampling, for example spring for communities dominated by vernal ground flora (largely herbs) but later in summer for grasses, sedges and rush. Also evergreen vegetation shedding leaves in mid-summer will need to be sampled later, when new canopy is fully developed. Suitable sampling dates to collect all functional groups (see later in chapter 3.4) present

in the plot need to be consulted with an experienced botanist. In the final analysis dried (or frozen) samples collected during different dates can be pooled to one composite sample per functional group. In plots where it is not possible to sample all species in one sampling occasion the sampling area is marked (with plastic or wooden markers) and the same sampling area is used in the following sampling time. If more than one sampling is used it is not necessary to collect regrowth of species collected earlier. Hence if grass species that have been already collected have regrown by the time other (e.g. evergreen) species are to be collected, these grass species can be ignored.

#### 3.2 Sampling design

Sampling of standing ground vegetation is performed in FutMon IM plots in places where this does not interfere with other activities in the plot, such as deposition and soil water collection, or assessment of ground vegetation. Suitable areas within the IM plot are e.g. in the surroundings of subplots used for vegetation assessment leaving, however, a large enough buffer zone around the area used for vegetation assessment, so that there is no disturbance by e.g. trampling or secondary succession. If possible, sampling should be established no less than 10m outside the sample plot used for vegetation assessment and its buffer zone and in areas similar to the conditions inside that area; so called site type representative sampling.

Ground vegetation is sampled using a frame of known area (see chapter 3.3). The frame is placed on the ground and all above ground parts of the vegetation passing through the frame are cut at ground level using scissors, loppers or a pair of shears. Hence a "projectional" approach should be adopted: parts of plants (of plants rooted inside the frame) which grow outside of the frame are omitted from the study; likewise parts of plants which grow into the frame (of plants rooted outside the frame area) are included. The number of the frames (and hence the total area sampled) need to be carefully recorded and reported. The samples of each frame are stored separately in plastic bags or durable paper bags to be transported to a laboratory for a further preparation. It is recommended to measure the shoot length of at least the 5 most abundant species within the frames. These results are submitted using the form XX2009.GBH in order to allow biomass modelling on the basis of vegetation height in the coming studies.

#### 3.3 Number of sampling units and quantity of sampled material

The number of sampling units (i.e. the number of the frames in which the vegetation is collected) within each IM plot depends on the area of frame used. A minimum total area requirement is  $2 \text{ m}^2$  that can be achieved e.g. by collecting ground vegetation from four sampling units (frames) of an area of  $0.5 \text{ m}^2$  or eight frames of an area of  $0.25 \text{ m}^2$ . It is recommended that the bigger frame is used for more robust vegetation. The area of  $2 \text{ m}^2$  is enough only in case the vegetation on the plot is homogenous. A larger area (more sampling units or larger frames) needs to be collected in case the vegetation is very heterogeneous (a large number of different species) or the biomass of the collected samples is small (e.g. the coverage is scarce and dominated by small lichens or bryophytes). Because the total area needed for a representative sample depends on the ground vegetation diversity, the sampling should be done in co-operation with the persons responsible for the assessment of ground vegetation. The exact number of sampling units (i.e. the number of the frames within which the vegetation is harvested), the area of the frame, and the total area sampled (sum of the area of individual samples) must be recorded and reported.

The aim of the sampling is to have a representative sample of the ground vegetation in each plot. Hence, sampling units (frames) are positioned on the plot the way that gives statistically reliable estimate of different species in different conditions. However, unusual micro-topographic features, such as drains, paths, rides or animal disturbance should be avoided, so as to best represent uniform under-canopy conditions. Because IM plot design is different in different countries a common sampling design (e.g. random sampling, systematic sampling, cluster sampling etc.) can not be stipulated. Hence each country has to apply a method that fits to its plot design and will yield a representative sample of the vegetation in that particular IM plot. Whatever sampling design is used care must be taken that the sampling does not interfere other activities in the plot.

Different species are grouped in larger functional groups (to be submitted in field "sample number" with code 1-8, cf. chapter 3.4) for the element analysis. The amount of collected sample should be large enough so that for each functional group present on the plot the chemical element analyses can be carried out. The total amount of sample needed for the element analyses depends on the method in use. As a rule of thumb: 10 grams of fresh mass of plant material will give around 2-5 grams of dry mass of which about half is left after grinding which is enough for microwave wet digestion (preceding e.g. ICP or AAS measurements). However, in order to conserve samples for future use, to be able to do quality checks if needed etc. it is recommended to sample at least twice as much as the minimum requirement for the chemical analyses. If for some functional group there is not enough biomass for chemical analyses, only the biomass should be recorded, but no chemical

analyses are performed. In case there are two or more functional groups in the plot where the biomass is too small for individual chemical analyses, these groups can be pooled. In this case the biomass of these functional groups is recorded separately but the chemical analyses are reported for a group that is labelled "rest" (group number 8, cf. chapter 3.4 for the functional groups). If, despite the pooling, chemical analyses are still not feasible, the chemical analyses for these groups can be dismissed.

#### 3.4 Assorting species into functional groups

If collected samples contain detritus this is carefully separated from the living biomass and removed. Samples of living ground vegetation of each sampling unit (frame) are separated into following seven different functional groups, dried and weighted (see below chapter 3.5).

1) Bryophytes (mosses, liverworts and hornworts)

2) Lichens

3) Ferns (all Pteridophytes)

4) Grasses (Poaceae), including sedges (Cyperaceae) and rushes (Juncaceae)

5) Herbs

6) Deciduous shrubs, including deciduous tree seedlings <50 cm height\*

7) Evergreen shrubs, including evergreen tree seedlings < 50 cm height\*

8) Rest\*\*

\* In cases of functional groups 6 and 7 (shrubs) the minimum requirement is to analyze foliage. If stems are chemically analysed they are separated from foliage. The biomass of foliage and stems are recorded separately. The results for stems are reported in the formats with sub code "b" (i.e. 6b for stems of deciduous shrubs and 7b for stems of evergreen shrubs). In case chemical analyses for the stems are not performed, only biomass results are reported.

\*\* Group "rest" (code 8 in the formats) is used in case two (or more) groups are pooled for the chemical analysis due to small amount of available biomass. The biomass results are, however, reported for the actual functional groups.

#### **3.5. Biomass weighing**

After individual species are pooled into the above functional groups each of these are oven dried (keeping samples from different frames separated) at a maximum of 80°C, for at least for 24 hours. The presence of woody material requires longer drying period. After the drying, the dry mass of

each functional group is measured. After recording the weight of 80°C dried samples (functional groups within each frame) the following steps are to be follow:

- In the case that there is not enough material per functional group within a IM sample plot (i.e. when the samples of individual frames are pooled) to do the chemical analyses (including grinding, microwave digestion etc. described in chapter 3.3) different functional groups can be pooled to form the group "rest" (cf. chapter 3.4). If even the pooling, to form the group "rest", will not yield enough material per plot to do the chemical analyses, the functional groups should be kept separate and dried at 105 °C.
- In the case that there is enough material for chemical analyses the samples (a functional group or group "rest") is ground to fine powder following the procedure described in the ICP Forests manual (Part IV).
- 3) Two subsamples of the ground material are then taken for:
  - a) drying a known amount of material in 105 °C (to determine the moisture content of the 80°C dried samples), and
  - b) to be used for chemical analyses
- 4) Element concentrations and biomass are then reported on dry weight of 105 °C

#### 3.6. Pretreatment before chemical analyses

In cases where the weight of the total pooled sample of a given functional group is sufficiently large, a subsample (e.g 10 to 20 g dry weight, depending of the analysis method in use) is ground to obtain homogenous powder that is treated and analysed as in case of chemical analyses of foliage samples (see Part IV in the manual of ICP Forests ). If there is not enough material for chemical analyses only the biomass results are reported. Element concentrations are reported by reference to 105°C-dried material (cf. Table 1 in Part IV in ICP Forests manual).

#### 3.7 Data submission

The data on Laboratory Quality Assurance ("XX2009GB.LQA") will be submitted together with the reduced plot file ("XX2009.PGB") and the data files ("XX2009.GBM" and "XX2009.GBO").

# 4. Sampling methods for more intensive foliar survey (Foliage D2) Mandatory on D2 plots

The methods described in the ICP Forests manual (Part IV) are valid here with one exception. In case of evergreen species, foliage age classes older than C+1 are also sampled and analysed. Hence if the species collected for analysis of element concentration has older foliage classes than C+1, these are collected and analysed the same way as C and C+1 foliage classes. Foliage age class should be considered to be present when there are more than 50% of the original leaves/needles present in the annual shoot (i.e. less than 50% have been shed). The number of the foliage age classes present are recorded and reported in the data forms. Foliage age classes older than C+1 can be chemically analysed separately or by using a combined sample (all foliage age classes older than C+1 together). When the data is submitted to the database only one value for the older than C+1 foliage is reported, i.e. if foliage age classes (older than C+1) are analysed separately a mean is computed and reported.

In order to allow for the submission of further tree numbers (only 5 tree number may be submitted with .FOM yet) it is foreseen that those further tree numbers will be submitted in a second data line (or record, respectively) with the same analysis results for nutrients, biomass, etc. as those in the first respective data line.

The data on Laboratory Quality Assurance will be submitted together with the reduced plot file ("XX2009.PLF") and the data files ("XX2009.FOM" and "XX2009.FOO") using the form "XX2009FO.LQA".

# Litterfall sampling and analysis

FutMon (Life+) Field Protocol 2009

# V1; last update 14<sup>th</sup> May 2009

# IM1 recommended, mandatory on D1 and D2 Demonstration Project plots

# Contents

1.	Introduction	2
2.	Objectives	2
3.	Sampling	2
3.	1 Siting and number of litterfall traps	3
3.	2 Material and dimensions	3
3.	3 Frequency of sampling	4
4.	Litter analysis	4
4.	1 Sampling, preparation and storage	4
4.	2 Drying, sorting and weighing	5
5.	Litter quality: chemical analysis	6
5.	1 Treatment before analysis	6
5.	2 Additional measurements:	6
5.	3 Direct assessment of LAI	б
5.	4 Indirect assessment of LAI	6
6.	Quality assessment and quality control	7
6.	1 Quality assurance programme	7
6.	2 Data validation	7
6.	3 Data submission and reporting	7
7.	References	7

# 1. Introduction

Litterfall is a key parameter in the biogeochemical cycle linking the tree part to the water and soil part. Both the biomass of the litter and its chemical content (including heavy metals) are needed to quantify the annual return of elements and organic matter to the soil. Litter decomposition is a major pathway of nutrient fluxes and determines the organic matter input to forest soils and has a strong influence on forest productivity.

Effects of anthropogenic and natural factors and climate change could influence both litterfall production and its seasonal progression. Processes like C-cycling and C-sequestration are closely related to stand leaf area index (LAI) and litterfall.

Changes in litterfall are responses to disturbances caused by pests or environmental factors like spring frost, drought, wind and pollution. Litterfall production is a quantitative parameter of stand vitality and gives additional information to visual assessment of tree vitality already observed in each plot. Direct observation of abnormalities of the leaves can be performed on the collected litter (leaf size, fungi, galls and necrosis) for symptomatology.

Litterfall also provides temporal and quantitative information about phenological development of the stand. The quantification of the foliage amount, flowering and fruiting patterns allows direct measurements of year-to-year variation in phenology as a reaction to climate, vitality, and global change.

Litterfall biomass of leaves is also one of the components of direct estimate of leaf area index (LAI), the stand leaf area per ground area. LAI describes a fundamental property of the plant canopy in its interaction with the atmosphere, especially concerning radiation, energy, momentum and gas exchange (Monteith and Unsworth, 1990). Leaf area index plays a key role in the interception of radiation, canopy interception (rainfall and deposition), in the carbon assimilation and water evapotranspiration during the diurnal and seasonal cycles, and in the pathways and rates of biogeochemical cycling within the canopy-soil system (Bonan, 1995; Van Cleve *et al.*, 1983). Finally, various soil-vegetation-atmosphere models models use LAI (Sellers *et al.*, 1986; and Bonan, 1993a). Litterfall collection and sorting of leaves/needles among species is the only way of getting accurate assessment of total leaf area index , particularly for broadleaves, and the contribution of each species to the total LAI. Indeed, the LAI for one species is not simply related to its density or basal area contribution to the stand and cannot be derived immediately from dendrometrical stand information.

# 2. Objectives

The main objectives of litterfall sampling and analysis are to quantify litterfall production and chemical composition over time. This record of litterfall variation will allow assessment of its role in nutrient cycling, across environmental gradients of climate (moisture and temperature) and soil conditions at both local and regional scales.

Also, there is a need to understand the relationship of climate and species on litterfall rates, the turnover of the biomass, the amount of litter produced, and its composition and chemical content. Furthermore, there is a need to improve our knowledge on the link between the C and the nutrient budgets/cycles.

# 3. Sampling

Litterfall sampling is time-consuming and hence expansive. The number of plots including litterfall monitoring depends on the aim of litterfall assessment and the Demonstration actions being undertaken by contributing countries within the FutMon 2009-2010 programme. It is strongly recommended that litterfall is assessed on the IM1 plots where intensive monitoring of

meteorology, deposition, soil solution, and phenology are also performed (to be future 'key plots'), and it is mandatory in plots selected for Actions D1 (tree vitality) and D2 (Nutrient cycling).

# 3.1 Siting and number of litterfall traps

It is recommended that the litterfall traps are set up in a design enabling comparisons with deposition and soil water results. The traps are fixed and may be placed randomly <u>or</u> systematically e.g. at regular intervals and in a sufficient number to represent the whole plot and not only the dominant tree species. As litterfall is a canopy parameter, and not a tree one, litterfall traps should be distributed all over the plot area. It is recommended to sample litterfall from at least 10 collectors per plot and even up to 20-30 collectors depending on plot size and tree species involved in the assessment. Leaves from deciduous trees are more susceptible to turbulent air movement than conifer needles. This effect may be mitigated by increasing the number of litterfall traps in deciduous species (i.e. 10 traps for coniferous species and 20 traps for deciduous species) or by increasing the collecting area of each trap (especially for species with large leaves like oak).

# 3.2 Material and dimensions

The countries are free to select the type of traps for the monitoring of litterfall. Figure 1 gives two examples of a litterfall trap. It is recommended that the litterfall traps are fixed not too close to the ground, in order to ensure water drainage. The opening area of the collectors must be horizontal. This means that specific trap fixation has to be prepared for plots on a slope. Canopy leaves and other litterfall inputs are sampled in litter bags. The bags are attached to a frame of e.g. wood of known area of minimum 0.18 m<sup>2</sup>, preferably 0.25 m<sup>2</sup>. The sampling area must be sufficiently large to be able to determine litter amount and quality. For tree species with large individual leaf area, the collecting area of traps must be increased (i.e. up to  $0.5 \text{ m}^2$ ). It is recommended that the litter bags are at least 0.5 m deep to prevent litter from blowing out of the bags. Deposition of litter into these traps due to lateral movements by wind is assumed to be minimal. The material of the mesh must not interact with the litterfall sample. Litter bags of inert materials like polyethylene or mosquito nylon or natural cotton fibres are a suitable material not interfering with the major ions present in litter. The mesh size of the bags must be large enough to allow for easy drainage of water. It is recommended to adapt mesh size to the dimension of smallest elements, i.e. for needles from coniferous species up to 0.5 mm. In snowy areas during the winter season, elevated traps may be exchanged with one placed directly on the ground to avoid breakages due to heavy snow load



Mesh trap



Solid Funnel with bag

# 3.3 Frequency of sampling

It is recommended to collect litterfall bi-weekly or at least monthly in periods of heavy litterfall, such as main autumn abscission. This is to avoid pre-collection decomposition of litterfall due to long stay in the traps during rainy autumns. The samples may be pooled to periodic samples once the monthly variations in amount and quality have been investigated.

In regions with snow and frost in wintertime and in remote areas it may be necessary to let the traps stay over winter in the forest. Litterfall may then be collected once before the winter period and once after snowmelt, as frost limits drainage and litter decomposition.

# 4. Litter analysis

# 4.1 Sampling, preparation and storage

The bags must be carefully labelled before sampling with information on study site, species, sample type, trap number, and date of collection. As a minimum the litterfall should be collected as a pooled sample per plot per year. It is up to each country to have a more detailed sampling (e.g. collection of litter from each trap for each sampling period). The litter from each trap can be collected into the labelled bag using a small brush and dustpan if the trap is fixed, or by replacing the sampling bag at each plot visit, when the bag is attached separately to the bottom of the trap. The litter should be transferred to large bags using nitrile gloves.

The samples should be transferred immediately to the laboratory. All contamination should be avoided in the laboratory.

# 4.2 Drying, sorting and weighing

Especially in mixed stands and if leaf area index is to be derived, it is recommended to sort the litter by species. Insects, insect debris, or other faecal droppings may be removed or stored (if desired) as a special type of litterfall. It is recommended as a minimum to measure the plot specific amount of litterfall of at least foliar and non-foliar fractions for chemical analysis on IM1 and D2 plots. For D1 beech, spruce and oak plots an annual value for the biomass of fruit fractions will be of main interest.

It is expected for **FutMon D1 action** that litterfall from beech and oak sites are routinely sorted into the following fractions for dry weighting and chemical analysis. Any green immature cones from fir, spruce or pine should also be recorded. The updated codes for Littefall fractions are:

Code	Fraction of Litterfall
10	Total
11	Foliar litter
11.1	Foliar litter of main tree species
11.2	Foliar litter of other tree species
12	Non foliar litter total
13	Flowering total
13.1	Flowering main tree species
13.2	Other Flowering
14	Fruiting/seeds total
14.1	Fruiting/seeds (main species + green cones)
14.2	Fruit Capsules (main species + empty cones)
14.3	Rest of fruiting
15	Budshells-Bud scales
16	Twigs/branches
17	Fines and Frass
19	Other biomass

For **Fut Mon D2 action** finer sorting than 'foliar vs non-foliar' is also recommended, placing old cones with capsules, as carbon levels will be different to immature fruit.

After this sorting, the total amount of litter is dried at air temperature for approximately a week. If air-drying is not immediately possible, it is recommended to cool the samples below +5 °C until drying can be performed. After this first drying the litterfall is sorted in at least two fractions: foliar

litter and non-foliar litter. Many countries sort in at least three fractions: foliage, wood (bark, branches, twigs, etc.; with area exceeding 5 mm x 5 mm or diameter more than 2 mm) and fruits cones and seeds. Each fraction is weighted. Then subsamples of each fraction (or the whole amount of each fraction, if the quantity is not large) are dried at maximum 80 °C to constant weight in grams with 2 decimal points (usually 48 h will be sufficient). After this drying, the mass of 100 leaves or 1000 needles is determined at 105 °C. Knowing the percentage of moisture in the sub samples, the whole amount of each fraction can be converted to dried mass at 80 °C.

# 5. Litter quality: chemical analysis

#### 5.1 Treatment before analysis

For chemical analysis the litterfall samples or sub-samples are dried to constant weight in an oven at maximum 105 °C, as for foliar sampling. The samples are then ground to a homogeneous powder. The chemical analysis of litter is similar to the foliar chemical analysis. For techniques and analytical methods see the chapter IV of the ICP Manual on Sampling and Analysis of Needles and Leaves, Annex 2.

Elements to be determined: Mandatory: Ca, K, Mg, C, N, P, S Optional: Zn, Mn, Fe, Cu, Pb, Cd, B

#### 5.2 Additional measurements:

Litterfall may be used to assess the leaf area index (LAI in the units  $m^2/m^2$ ), particularly for broadleaf deciduous species, as well as determining other foliar parameters like length, width, and thickness of needles/leaves. The most suitable definition of LAI is half the total green leaf area (one-sided area for broad leaves) in the plant canopy per unit ground area (Chen and Black, 1992). Globally, LAI in forest stands varies from less than 1 to above 10 but also exhibits significant variation within biomes at regional level, as a result of climate and management (stand structure, species composition, thinning). For a given plot, even without any thinning, one can observe year-to-year LAI fluctuations due to stand reaction to stresses like drought, frost, defoliation or complex forest decline. From that point of view, LAI is a stand vitality parameter.

#### 5.3 Direct assessment of LAI

Litterfall collection is the most precise method to assess LAI in broad-leaved stands; this is the reference direct measurement. Periodic litter collection allows for the assessment of both maximum stand LAI and for monitoring the pattern of LAI decrease during the autumn, or other periods of insect attack. LAI is computed for each collection date from leaf litter dry biomass multiplied by a ratio to convert dry weight to leaf area. This ratio leaf area/dry mass is named **S**pecific Leaf Area and is expressed as  $cm^2/g$ . It has to be determined for each species on a sub-sample of litter leaves (at least 200 leaves from different traps). When establishing this parameter, direct quantification of individual leaf dimension have to be computed and can be used by themselves as vitality indicator (for example, smaller leaves can be observed as a result of fruiting, defoliation, or severe drought e.g. Europe in 2003).

#### 5.4 Indirect assessment of LAI

Leaf area index may also be estimated by indirect methods in the field using radiation interception by the canopy. Several wand-type canopy analyzers (like e.g. Li-Cor LAI2000) are available.

Hemispherical photography may also be used to measure LAI separately from the herbaceous, shrub, and overstory tree layers, particularly under the more sparse canopy of pines. However, such equipment is not suitable to quantify the contribution of individual tree species to the total stand LAI. Finally, these indirect methods need to be calibrated against direct measurements, as they do not measure LAI but Surface Area Index, including not only leaves but also stems, branches and all intercepting elements. It is recommended to measure at least maximum LAI reached at the middle of the growing season for broadleaves and at the end of the full new needle extension period for conifers.

A respective field protocol on radiation measurement and indirect LAI assessment is prepared under FutMon.

# 6. Quality assessment and quality control

# 6.1 Quality assurance programme

The condition of all traps is controlled at each visit to the plot. Several points have to be checked: horizontality of traps, integrity of bags to avoid litter loss; eventual cleaning after being emptied to ensure water drainage. It is recommended to number each trap unless bulk sampling is always performed. The visibility of this information must be checked before the litterfall assessments start.

# 6.2 Data validation

The national laboratories are encouraged to participate in the foliar inter-laboratory tests of the ICP-Forest programme. The results will be compared to the chemical analysis of the foliage of the respective plots. The Laboratory Quality Assurance information will be submitted together with the data on litterfall analyses. It is also suggested that ring-tests on the dried and fractionated parts of the litter samples be initiated, using litter available in sufficient quantity from sites with uniform stands.

Data checks should be performed as soon as possible after the performance of the analysis. Guidelines for the treatment of missing values and data below the detection limit are similar to the guidelines under the foliar analysis.

# 6.3 Data submission and reporting

The results of litterfall chemical analysis are reported to 105 °C (litterfall mass will need adjustment - see Tables 14b and c). Elemental litterfall fluxes are found by multiplying litterfall masses (expressed at 105 °C) times elemental concentrations. Validated data are sent at the end of the year to the European database on Forms .PLF, .PLM and .PLO (see forms document) and the Laboratory QA file on Litterfall analyses (XX2009LF.LQA).

ICP submission should be accompanied by a "Data accompanying report – questionnaire (DAR-Q) for. This DAR-Q includes all details on the sampling and analytical procedure, missing data, and other irregularities.

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#### Collated for ICP forests by Anne Marie Bastrup-Birk and Nathalie Breda 2003/2006 Ammended for FutMon by Rona Pitman 2009

# FUTMON FIELD PROTOCOL PHENOLOGY (D1)

# V1.0; last update 15<sup>th</sup> May 2009

Phenological observations under FutMon Action D1 can be made in two different ways, manually or using digital cameras.

The phenology observations are made according to the guidelines provided in chapter 9 "Phenological Observations" of the ICP Forests Manual. However, the following points/exceptions should be taken into account:

- Both plot level and single tree level observations are accepted, but wherever possible single tree level phenology should be preferred.
- For plot level observations the guidelines were revised and the data submission form (.PHE) was updated.
- For single tree observations the minimum required frequency is once a week during the critical phases, but daily observations is the optimum. For the selection, trees are preferred that are also selected for D1 girthband measurements.
- For the registration of the trees selected the form (.PLP) has been updated.
- Guidelines for the recording of the events at single tree level are revised. For the submission of the data the updated form .PHI has to be used.
- For the field observations **flushing** and **autumn colouring** are mandatory, the other events are optional. When using cameras all events are mandatory.
- For the use of digital cameras guidelines are given below. For the registration of the observed trees and the analyses of the pictures the same guidelines and forms as for the single tree observations should be used. Data should be submitted at a daily basis.
- The use of cameras is recommended for remote plots where the use of relatively expensive camera systems helps to save a lot of travel costs; in order to fulfil the FutMon contract each associated beneficiary is recommended to test at least one camera system.
- As far as biotic damage is observed during the phenology assessments its appearance is submitted with the phenology data as far as foreseen with the updated forms .PHE and .PHI. The more detailed examination, assessment and data submission on the observed damage has to be done according to the ICP Forests manual on damage assessment. Whenever needed, trained staff should be consulted for damage assessment within the next 4 weeks beginning from the phenology assessment.

# Annexes:

Annex 1: revised guidelines for recordings Annex 2: guidelines for the use of cameras revised forms are presented on FutMon webpage in a separate excel file on forms and explanatory items

# Annex 1: revised guidelines for recordings

#### 1 Recording of phenological events at the plot level

For the recording of the phenological phenomena at the plot level form QQQ is used

The event codes for the monitored effects and phenological phenomena are:

- 1 = Flushing;
- 2 =Colour changes;
- 3 = Leaf/needle fall;
- 4 = Significant signs of leaf or crown damage (e.g., eaten leaves or bare crown parts);
- 5 = Other damage (breakage, uprooted trees);
- 6 = Lammas shoots / secondary flushing;
- 7 = Flowering.

Scoring system

Occurrence of the events and phenomena (proportion of the forest crown affected):

1 < 1% (in case of event "flowering": absent)

2 = 1 - 33% (see also below "flowering phases")

- 3 = >33 66% (see also below "flowering phases")
- 4 = >66 99% (see also below "flowering phases")
- 5 > 99% (in case of event "flowering": present)

Intensity of flowering (optional quantifiaction) (NEW!)

- 1 = Flowering absent
  - 2 =sparse (optional)
  - 3 = moderate (optional)
  - 4 = abundant (optional)
- 5 = Flowering present

2 Recording of phenological events at the individual tree level

2.1 Registration of trees selected

For the registration of the trees selected for the recording of phenological events form .PLP is used.

The <u>part of the crown</u> observed (here: the visible part of the crown) should be reported at the time the trees are selected, or whenever it changes, using the following codes (same as described in ICP Forests Manual):

- 1 = top of the crown
- 2 = middle of the crown
- 3 = top and middle of the crown.

The codes for the direction <u>from</u> which observations are made are (same as described in ICP Forests Manual): 1 = North

- 2 = north-east
- 3 = East
- 4 = south-east
- 5 =South
- 6 =south-west

7 = West

8 = north-west

The codes for the vertical direction **from** which the observations were made are (**NEW**!):

1 =from below

2 = at crown level

3 =from above

# 2.2 Recording of phenological phenomena

For the recording of the phenological phenomena at the single tree level updated form .PHI is used

The method used for making the observations

1= field observation

2= Digital camera

3= Both field observation and digital camera

The event codes for the monitored effects and phenological phenomena are:

1 = Flushing;

- 2 =Colour changes;
- 3 = Leaf/needle fall;
- 4 = Significant signs of leaf or crown damage (e.g., eaten leaves or bare crown parts);
- 5 = Other damage (breakage, uprooted trees);
- 6 = Lammas shoots / secondary flushing;

7 = Flowering.

Scoring system

Occurrence of the events (proportion rate of tree compartments affected):

1 < 1% (in case of event "flowering": absent)

- 2 = 1 33% (see also below "flowering phases")
- 3 = >33 66% (see also below "flowering phases")
- 4 = >66 99% (see also below "flowering phases")
- 5 > 99% (in case of event "flowering": present)

Flowering phases:

The number of male flowers that are in the described stage or have already passed this stage is to be recorded using the following classification:

Intensity of flowering (optional quantifiaction) (NEW!)

- 1 = Flowering absent
  - 2 = sparse (optional)
  - 3 = moderate (optional)
    - 4 = abundant (optional)
- 5 = Flowering present

# Needle appearance, leaf unfolding, autumn colouring and leaf fall:

The proportion of needles or leaves of the visible part of the crown that are in the described stage or have already passed this stage is to be recorded using the following classification:

0 = 0%

1 =>0 - 33%

2 = >33 - 66%

3 =>66 - <100% 4 = 100%

# 3 Recording of biotic or abiotic damaging events

Reporting of observed biotic damage will be done using event code 4 or 5 respectively for form .PHE and .PHI, respectively. For damage description the form .TRD and the respective coding described in the ICP Forests manual on Crown condition and biotic damage assessment (Part II) are used.

# Annex 2: guidelines for the use of cameras

Advantages of the use of cameras:

- Enables frequent (continuous) monitoring, also on remote sites
- Assessments can be made any time when staff is available
- Enables comparison between sites
- Improves comparison between years
- Enables comparison between countries/regions
- Enables better timing of appearance of damages

Points to be considered

- High investment costs
- Need for power supply
- Difficult in dense (conifer) stands
- Possible technical failures
- Possible vandalism

#### The use of digital cameras for monitoring phenology

When using digital cameras the first priority should be that the quality of the pictures (resolution) obtained allow the assessment of phenological phases at individual tree level according the guidelines in the manual on phenological observations with 33% classes. In addition also other aspects of the crown such as damages can be assessed. At each plot at least 10 trees per species should be assessable (tree selection as mentioned above in this protocol).

#### Technical requirements

The cameras should be weather-resistent, e.g outdoor surveillance cameras are suitable. Important is that the pictures are of high resolution (minimum requirements 6 Mpix with 300 pix/inch / 120 pix/cm), even with full zoom properties. The camera should have its own memory, or be connected to a datalogger. The datalogger and steering unit should be stored in a weather-proof place, and the whole system should be protected against lightning. Power supply can be obtained through batteries, solar panels or connection to the electricity network. The working of the camera should be checked every time the plot is visited.

#### Location of the camera

If possible the camera should be mounted to a mast that reached over the top of the crowns, e.g. the towers used for the meteorological assessments. In order to be able to observe a number of trees, the camera should be movable and programmable so it can take pictures of the same spot at regular intervals. The position of the camera is selected so that it can cover an optimal number of individual trees within the plot at a area as large as possible. The observations can also be made from below the crowns, but this way the area, and the number of visible trees per camera is more limited. Alternatively more than one camera could be used. The camera should take pictures of the whole upper part of the crown. Trees around the camera are selected and registered using form 12a. For each tree also the part of the crown observed, as well as the direction from which the camera takes the picture are marked. The codes used are the same as for the manual single tree observations.

#### Data handling

Pictures should be taken a number of times each day (at least 2) because the light conditions change during the day because of the position of the sun. At least every 2 months the data should be collected from the plots in order to secure the data. The camera can also be connected to a network, so the observations can be made at distance. In this case it is still advisable to have the pictures also stored at the plot for backup. The pictures of the different plots should be analysed by one and the same person, or at least for the different plots of each tree species. This way the effect of the observer is eliminated. The assessments should be made

using the same stages and codes as used for the field observations. Only one observation per day should be made.

The pictures should be stored by the NFC so they can be used later for inter-calibration as well as for comparison between countries. During the phenological phases to be assessed for each tree at least one picture per day should be saved. For the rest of the growing season at least one picture per week is sufficient. If pictures are also taken during winter also one picture per week can be stored. At form xx metadata about the stored pictures should be submitted annually.

Pictures should be available to other partners of the project. In order to enable for an easy data access the photos and movies may be stored in addition at the FutMon data centre. In order to allow for a consistent and uniform identification and submission of the digital images the form .PHD and a respective Explanatory Item are to be applied.

#### **FutMon Field Protocol Determination of the soil water retention characteristic** V 1.0; last update 15<sup>th</sup> May 2009

Soil water retention characteristic (pF analysis)				
Method sheet SA14				
Reference method ISO 11274				
Method suitable for Mineral and organic soil horizons, undisturbed samples				

#### 1. Introduction

During the FutMon LIFE+ project 2009-2010, the demonstration action D3 aims at the assessment of forest water budgets. Data is collected on more than 100 D3 plots being a subset of the IM1 plots. For the parameterisation of various water balance models meteo data, stand characteristics and soil physical data are essential. For the validation of the models soil temperature, soil moisture and stand precipitation measurements are needed.

The soil water retention characteristic is a physical soil property depending mainly on soil texture, organic material and bulk density. Therefore it will vary both vertically (horizons/layers in the profile) and horizontally in each plot. Stratified sampling according to horizons or specific layers is a prerequisite to determine the overall hydrological behaviour of a soil profile.

Specific points on the soil water retention curve (SWRC), which is the relationship between volumetric soil water content and matric pressure, are required to (1) determine indices of the volume of plant-available water, (2) estimate the soils' pore size distribution and (3) predict other soil physical properties (e.g. hydraulic conductivity). The SWRC is an essential part in most water budget models.

This protocol describes the determination of the soil water retention characteristic in the laboratory, extending from saturated soil (no pressure or suction; 0 kPa) to oven-dry soil (about  $-10^{6}$  kPa).

The format of this protocol is in line with the new standard structure for sub-manuals proposed by the QA committee (Quality objectives in FutMon).

#### 2. Scope and application

This FutMon protocol conforms the ISO 11274 international standard for determination of the soil water retention characteristic based on measurements of the drying or desorption curve. All methods described by ISO 11274 are allowed, except method B, using a porous plate and burette apparatus for matric pressures from 0 to -20 kPa.

The volumetric soil water content at matric pressure 0 kPa is approximated by the total porosity of the soil.

In addition this protocol describes the mandatory and optional matric pressures to assess in the lab as adopted for FutMon action D3. This further standardisation is a prerequisite for facilitation and harmonisation of database handling.

The protocol outlines the general description of basic sampling and laboratory operation for soil water retention analysis at plot level. Definitions of variables and guidelines for method selection and sampling are applied as described in the ISO 11274:1998 method (ISO, 1998).

#### 3. Operational objectives

The general operational objectives are:

- To determine on each plot the SWRC for specific soil layers of at least 3 profiles. The field matric potential for each layer will be inferred from the measured water content (WC) and its layer specific SWRC;
- To assess the SWRC at plot level only once. Just like texture, the SWRC is considered a constant soil property showing little change over time;
- To harmonise and standardize the field methods for sampling undisturbed soil samples and the determination of the SWRC in the lab;
- To quantify the accuracy (trueness and precision) of the results of SWRC determination, based on within lab analysis of replicate samples (e.g. twin field samples) and participation in interlaboratory physical soil comparisons using reference material;
- To assess the spatial variation of the SWRC within the plot;
- To use the SWRC for the parameterization of various water budget models (e.g. WATBAL, BROOK90, SIMPLE, COUP, THESEUS, WASIM-ETH, ...). The prediction capacity of these models will be partly determined by the uncertainty of parameters derived from the SWRC.

The specific operational objectives described in this protocol are:

- adequate sampling of undisturbed soil in situ;
- correct handling of undisturbed soil cores prior to analysis;
- analysis of the soil water characteristic in the laboratory;
- standardised reporting of the SWR results.

#### 4. Location of measurements and sampling

#### 4.1 Sampling design at plot level

#### 4.1.1. Sampling locations (profiles) within the plot

On each plot at least 3 profiles are sampled separately. The location of these profiles within the plot may be chosen freely, as long as their spatial design meets following requirements:

- the individual profiles are representative for the soil condition within the plot;
- the profiles are not located in one single profile pit (i.e. profiles are at least some meters apart);
- the profiles should be situated as close as possible to the location of the soil moisture measurement sensors;

The exact coordinates of each profile location should be determined.

#### 4.1.2. Sampling within the soil profile

At each location, adequate undisturbed soil sampling within the soil profile is done according to the sampling scheme in Table 1. At least one undisturbed core is taken within the fixed depth intervals 0 - 20, 20 - 40 and 40 - 80 cm, preferentially at the same depth as the soil moisture measurements (depth of TDR sensors). The exact depth range of the soil core (top to bottom of core) is reported, along with the ring ID information.

When forest floor thickness (OF + OH layer) is > 5 cm, the holorganic layer should be sampled also with a suitable cylinder. Optionally, extra soil layers or horizons could be sampled that are considered relevant for the hydrological regime of the soil profile.

Table 1.	Soil	profile	sampling	scheme
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Matrix	Depth interval (cm)	Minimum number of replicates		Requirements for D3
		per profile	per plot	
Organic Layer	Forest floor > 5 cm thick	1	3	Mandatory
	Forest floor <= 5 cm thick	-	-	not required
Mineral layer	0 - 20 cm	1*	3	Mandatory
	20 - 40 cm	1*	3	Mandatory
	40 - 80 cm	1*	3	Mandatory
	> 80 cm	-	-	Optional
	Extra (specific) layer	-	-	Optional

(\*) if the mineral layer is difficult to sample (e.g. caused by higher gravel content) a higher number of samples are strongly recommended).

Concluding from Table 1, on each plot at least 9 undisturbed and representative samples should be taken if the forest floor is less than 5 cm thick and 12 samples if the forest floor is more than 5 cm thick.

For each undisturbed sample, the pedogenetic horizon according to FAO (2006) designations, should be reported that contains the centre of the sampling cylinder. The pedogenetic horizon may be deduced from the soil profile description of the sampled plot.

Hence for each undisturbed core sample following information is reported:

- exact depth range of core cylinder in cm (e.g. 10 -15 cm for a cylinder of 5 cm in height);
- pedogenetic horizon containing centre of undisturbed sample (e.g. 12.5 cm is located in E horizon)

#### 4.2. Sampling equipments

#### 4.2.1. Sampling cylinders

Undisturbed soil cores are taken in dedicated metal cylinders (sleeves) with a volume between 100 and 400 cm<sup>3</sup>. Plastic cylinders are dissuaded. The same steel cylinders can be used as for determination of bulk density (method SA04). The sample ring dimensions should be representative of the natural soil variability and structure.

Recommended dimensions (height x diameter in mm) of cylinders for forest soil sampling are:  $50 \times 53$ ,  $40 \times 76$  and  $50 \times 100$ . It is important to verify that the laboratory that will process the undisturbed samples is equipped for the type of sample rings used.

The bottom of the sample ring should have a cutting edge. Plastic lids should perfectly fit to both ends of the steel cylinder.

#### 4.2.2. Sampling material

In a soil profile pit, undisturbed samples can be taken directly using the sample ring, without extra material like an open or closed ring holder. In that case, after introduction, the soil sample ring should be dug out carefully.

Alternatively, an open ring holder may be used. In such a holder, the ring is locked by means of a rubber or lever. Over the ring some space headroom is left allowing for taking an oversize sample. This prevents the sample for compaction during sampling.

In hard soil layers, an impact absorbing hammer may be used for hammering the ring holder into the soil.

When sampling is done in a bore-hole, a closed ring holder is recommended. This type of ring holder holds the cylinder in a cutting shoe. The ring is clamped inside the cutting shoe and no water or soil can come into the ring from the top. Moreover, the sample ring is protected, the sample is oversized on both sides and there is no risk of losing or damaging the sample ring. In hard layers, an impact absorbing hammer may be used with care.

Trimming both ends of the cylinder is preferably done using a small frame saw. A spatula or knife may be used but care has to taken avoid smearing the surface (closing macro- and mesopores).

After closing the cylinders with plastic lids, the sample should be labeled and wrapped in plastic bags or plastic or aluminium foil to prevent drying.

In conclusion, the sample material is:

- steel cylinders (sample rings) with lids
- open ring holder (optional)
- closed ring holder (needed when sampling in boreholes)
- spade and/or trowel for digging out the cylinder
- impact absorbing hammer (for hard soil layers only)
- small frame saw
- spatula or knife

- waterproof marker for labeling
- plastic bags or foil for wrapping the ring

#### 4.3. Sample collection

Undisturbed samples should be collected during a wet period, preferentially when soil matric pressures are at or near field capacity. Do not sample the soils when it is freezing.

The sampling procedure for undisturbed soil sampling (core sampling in steel rings) is as follows:

- Take soil cores carefully to ensure minimal compaction and disturbance to the soil structure, either by hand pressure in suitable material or by using a suitable soil corer and/or core holder. Take one sample (preferentially 3) for each freshly exposed soil horizon or layer; more replicates are required in stoney soils;
- The ring sample is taken vertically with its cutting edge downwards;
- Dig out the cylinder carefully with a trowel, if necessary adjust the sample within the cylinder before trimming flush, trim roughly the two faces of the cylinder with a small frame saw or a knife and fit lids to each end;
- Record the sampling date, sample grid reference, horizon encompassing the centre of the core, and the exact sampling depths (depth of top and bottom of the cylinder with respect to the top of the mineral horizon).
- Label the cylinder on the lid clearly with the sample plot reference, the sampling date, the horizon code and the sample depth;
- Wrap the ring samples in plastic bags or a plastic or aluminium foil to prevent drying;

#### 4.4. Sample storage and transport

The undisturbed samples are transported in plastic boxes or aluminium cases. They protect the samples from heat, humidity or dust. If transported in vehicles over long distances, shocking of samples should be avoided by using shockproof materials.

Prevent undisturbed soil samples from freezing. Store the samples at 1 to 2  $\degree$  to reduce water loss and to suppress biological activity until analysis. Samples with obvious macrofaunal activity should be treated with a suitable biocide, e.g. 0,05 % copper sulfate solution.

It is recommended to avoid weeks of storage of undisturbed soil samples. Ideally, undisturbed soil samples are analyzed in the lab immediately after sampling.

#### 5. Measurements

#### 5.1 Measurements to be done and reporting units.

In order to determine the SWRC, the volumetric water content ( $\theta$  in volume fraction, m<sup>3</sup> m<sup>-3</sup>) is determined at predefined matric potentials ( $\psi$ , in kPa). As indicated in Table 2, six of these matric heads are mandatory to determine. Extra observations of the SWRC at pressures -10, - 100 and -250 kPa are optional but they greatly improve fitting the SWRC.

Some matric heads immediately provide information on SWRC parameters: at 0 kPa the maximum water holding capacity (WHC) of the saturated soil sample is determined; depending on definitions and soil texture field capacity (FC) may be inferred from -5 till -100 kPa; permanent wilting point (PWP) is attained at a matric pressure of -1500 kPa and dry bulk density (lowest pressure at about  $10^{-6}$  kPa) derived in the oven at  $105^{\circ}$ C. The standard instruments required for each determination are listed in Table 2.

Matric potential ψ		Recommended Instrument	Estimator	
pF	kPa			
0.0	0	Pycnometer	≈θsat=WHC= Total porosity	
1.0	-1	Sand suction table		
1.7	-5	Sand suction table	FC	
2.0	-10	Sand suction table	FC sand	
2.5	-33	Kaolin suction table	FC siltloam	
3.0	-100	Kaolin suction table	FC clay	
3.4	-250	Ceramic plates		
4.2	-1500	Ceramic plates	PWP	
7.0	-10 <sup>6</sup>	Oven	Dry BD	

Table 2. Overview of matric heads to assess for the determination of the SWRC. Mandatory pressures to determine are in bold, optional in italic.

Where:

1) the pF is the logarithm of the absolute value of the matric potential expressed by the graduation of the water column (cm).

2) 1 kPa = 10.22 cm H<sub>2</sub>O or 1 cm H<sub>2</sub>O column = 0.097885 kPa

3) 100 kPa = 1 bar

#### 5.1.1. Determination of the soil water characteristic

The ISO 11274:1998 allows 4 methods to determine matric pressures within specific ranges:

- A) method using sand, kaolin or ceramic suction tables for determination of matric pressures from 0 kPa to 50 kPa;
- B) method using a porous plate and burette apparatus for determination of matric pressures from 0 kPa to 20 kPa; (single sample)
- C) method using a pressurized gas and a pressure plate extractor for determination of matric pressures from - 5 kPa to - 1500 kPa;
- D) method using a pressurized gas and pressure membrane cells for determination of matric pressures from 33 kPa to 1500 kPa.

Since method B allows only processing a single sample at the time, use of this method is not recommended. Laboratories are free to apply methods A, C and D according to the ISO 11274 standard.

Guidelines for choosing the most appropriate method for specific soil types are given in ISO 11274, chapter 3.

Before applying methods A, C or D, general recommendations for sample preparation are:

• For measurements at pressures from 0 to -50 kPa, use a nylon mesh to retain the soil sample in the sleeve and secure it with an elastic band or tape;

- Ensure maximum contact between the soil core, mesh and the porous contact medium of the suction tables, plates or membranes; remove any small projecting stones if necessary;
- Avoid smearing the surface of (clayey) soils, especially when water saturated;
- Inspect the sample for bioturbation (worms, isopods) or germination of seeds during analysis; the use of a biocide is discouraged;
- Report the temperature at which the water-retention measurements are made;
- Ideally, measurements start with field-moist samples [i.e. do not dry the undisturbed samples first (hysteresis effect)]. Then, samples are saturated with water.
- Respect wetting times before starting measurements to obtain a saturated sample. General guidelines for wetting times according to ISO 11274 are:
  - sand 1 to 5 days
  - loam 5 to 10 days
  - clay 5 to 14 days or longer
  - peat 5 to 20 days.

#### 5.1.2. Method A (recommended method for matric pressures 0, -1, -5, -10 and -33 kPa)

# Determination of the soil water characteristic using sand, kaolin and ceramic suction tables

#### Apparatus

- Suction table (watertight, rigid container with outlet in base and close fitting cover)
- Drainage system for suction table, enabling to maintain suction at specific matric pressures

• Sand, silt or kaolin packing material, appropriate for use in suction tables (homogenous, sieved, graded and washed, free of organic material or salts). Material should achieve the required air entry values (see ISO 11274 for details)

- Drying oven capable of maintaining temperature of 105 ± 2 ℃
- Balance (accuracy 0.1% of measured value)

#### Procedure

- Weigh the cores and then place them on a suction table at the desired matric pressure with table cover closed. The reference 0 cm height for setting the suction level is the middle of the core;
- Leave the cores for 7 days (minimum equilibration time). Equilibrium is reached if daily change in mass of the core is less than 0,02 %;
- If equilibrium is reached, weigh the cores, if not, replace cores firmly onto the suction table and wait until equilibrium is reached.

#### Calculation

ISO 11274 describes two procedures:

- 1. Procedure for soils containing less than 20 % coarse material (diameter greater than 2 mm)
- 2. Procedure for stony soils; conversion of results to a fine earth basis

#### For soils with less than 20% coarse material:

• Calculate the water content mass ratio at matric pressure  $\psi_i$  using the formula:

```
\begin{split} WC\psi_i &= \left(M\psi_i - M_{dry}\right) / M_{dry} \\ where \\ WC\psi_i & \text{is the water content mass ratio at a matric pressure } \psi_i, \text{ in grams}; \\ M\psi_i & \text{is the mass of the soil sample at matric pressure } \psi_i, \text{ in grams}; \\ M_{dry} & \text{is the mass of the oven-dried soil sample, in grams}. \end{split}
```

• Calculate the volumetric water content at matric pressure  $\psi_i$  using the formula:

 $\theta\psi_i = \left[\left(M\psi_i - M_{dry}\right) / \left(V \mathrel{x} \rho_w\right)\right] \mathrel{x} 10^{\text{-3}}$ 

alternatively

 $\theta \psi_i = W C \psi_i x (\rho_b / \rho_w)$ 

where

$\theta \psi_i$	is the water content volume fraction at matric pressure $\psi_i$ , expressed in m <sup>3</sup> m <sup>-3</sup>
	(volume of water per volume of soil);
Mψi	is the mass of the soil sample at matric pressure $\psi_i$ , in grams;
M <sub>drv</sub>	is the mass of oven dried soil sample, in grams;
V	is the volume of the soil sample in m <sup>3</sup>
ρ <sub>w</sub>	is the density of water, in kg m <sup>3</sup>
$\rho_{b}$	is the bulk density of oven dried soil at 105°C, in kg m <sup>-3</sup> .

# For soils with more than 20% coarse material, data needs conversion to a fine earth basis as follows:

The volumetric water content of the fine earth ( $\theta$ f) equals:

$$\theta f = \theta t / (1 - \theta s)$$

where:

- $\theta$ f water content of the fine earth, expressed as a volume fraction (m<sup>3</sup> m<sup>-3</sup>);
- $\theta$ s volume of non-porous stones, expressed as a fraction of total core volume (m<sup>3</sup> m<sup>3</sup>);
- $\theta$ t is the water content of the total earth, expressed as a fraction of total core volume (m<sup>3</sup> m<sup>-3</sup>);

For porous stones, a different correction should be applied as described in ISO 11274.

If volumetric water content is reported on fine earth basis, this should be clearly reported along with the volume of non-porous stones in the sample.

#### 5.1.3. Method C (recommended method for matric pressures -250 and -1500 kPa)

#### Determination of soil water characteristic by pressure plate extractor

#### Apparatus

- Pressure plate extractor with porous ceramic plate
- Sample retaining rings/soil cores with discs and/or lids

- Air compressor (1700-2000 kPa), nitrogen cylinder or other pressurized gas)
- Pressure regulator and test gauge
- Drying oven capable of maintaining temperature of 105  $\pm$  2  $^{\circ}$ C
- Balance (accuracy 0.1% of measured value)

Follow the manufacturer's instruction to assemble and operate the apparatus.

# Procedure

- Take small subsamples from the undisturbed sample: soil cores of approximately 5 cm diameter and between 5 mm and 10 mm in height; smaller samples for lower pressures are used in order to avoid long equilibration times;
- It is acceptable to use disturbed samples at pressures lower than 100 kPa, providing that the disturbance consists only in breaking off small pieces of soil and not in compressing or remoulding the soil.
- Use at least three replicate samples of each sample and place them on a presaturated plate;
- Wet the samples by immersing the plate and the samples until a thin film of water can be seen on the surface of the samples;
- Create a saturated atmosphere in the extractor;
- Apply the desired gas pressure and keep to a constant level, check for leaks;
- Record on a daily basis the evacuated water from the samples, when no change are observed (volume in a burette remains static) the samples have come to an equilibrium;
- At equilibrium status, soil samples are weighed, oven-dried and reweighed to determine the water content at the predetermined pressures

#### Calculation

The same calculation procedure as in 5.1.2 is applied, for samples without or with coarse fragments.

# 5.1.4. Method D (recommended method for matric pressures -250 and -1500 kPa)

# Determination of soil water characteristic using pressure membrane cells

# Apparatus

- Pressure cells with porous baseplates
- Cellulose acetate membrane
- Pressure regulator
- Air compressor (1700-2000 kPa, nitrogen cylinder or other pressurized gas)
- Drying oven capable of maintaining temperature of  $105 \pm 2 \ C$
- Balance (accuracy 0.1% of measured value)

Follow the manufacturer's instruction to assemble and operate the apparatus.

# Procedure

- Soil subsamples are placed on a porous cellulose acetate membrane
- Equilibrium status is attained when water outflow from the pressure cell ceases and soil water content is determined by weighing, oven-drying and reweighing the sample.
- Gas pressure methods are only suited to determine matric pressures below 33 kPa

#### Calculation

The same calculation procedure as in 5.1.2 is applied, for samples without or with coarse fragments

#### 5.1.5 Determination of the total porosity

A value for porosity can be calculated from the bulk density  $\rho_{\text{bulk}}$  and particle density  $\rho_{\text{particle}}$ :

$$\phi = 1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{particle}}}$$

Often the **particle density or true density** of soil is approximated by 2650 kg.m<sup>-3</sup> (mineral density of quartz). But the direct measurement of the particle density is strongly recommended to be done by the means of a pycnometer.

#### 5.1.6. Determination of dry bulk density

Determination of dry bulk density is also done according to method SA04 (submanual IIIa) The dry bulk density (BD) is recorded in kg  $m^{-3}$  with no decimal places.

In the case of stony or gravely soils the bulk density of the fine earth fraction (< 2 mm) should be reported. Furthermore, the bulk density of the coarse fragments should be known, but this may be approximated as  $2650 \text{ kg.m}^{-3}$ .

#### 5.1.7. Reported data, their units and numerical precision

Based on the SWRC measurement in the lab, data reported for each undisturbed soil sample are listed in Table 3.

Table 3. Raw SWRC data: measurement, unit and numerical precision to be reported for
each sample. Data in bold are mandatory to report.

Matric pressure (kPa) Ψ	Volumetric water content (VWC) = θ	unit	Numerical Precision
0	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-1	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-5	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-10	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-33	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-100	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-250	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
-1500	0.xxxx	m <sup>3</sup> m <sup>-3</sup>	0.0001
Matric pressure (kPa) Ψ	Dry bulk density (BD)	unit	Numerical Precision
-10 <sup>6</sup>	XXXX	kg m⁻³	0

#### 5.2. Data Quality Requirements

Plausibility limits for SWRC of mineral forest soils and organic layers will be developed in the future; partly based on the results of Action D3 in FutMon.

Tolerable limits for laboratory performance will be derived from the reproducibility data gained by performing the interlaboratory physical soil ringtest during FutMon.

Soil water retention data are considered complete if volumetric water content for all six mandatory matric heads (bold in Table 3) is determined. For scientific reasons analysing the optional matric heads also is strongly recommended.

Interpolation of volumetric water content between matric pressures is not allowed. All reported values should have been measured according to the methods described in this protocol.

#### 6. Data handling

#### 6.1. Data submission forms

Forms for storing the SWRC data in the FutMon databases will be developed under FutMon. Basically following data should be stored:

- the undisturbed sample metadata:
  - o sample ID
  - 0 plot ID

  - profile ID
    fixed depth layer
  - o horizon designation
  - o sample ring depth (top) in cm below top of mineral soil
  - sample ring depth (bottom) in cm 0
  - To be submitted with form .SWC
- the raw volumetric water content ( $\theta$  = VWC in m<sup>3</sup> m<sup>-3</sup>) data mentioned in Table 3:
  - VWC0 0
  - VWC-1 0
  - VWC-5 0
  - **VWC-10** 0
  - o VWC-33
  - o VWC-100
  - o VWC-250
  - VWC-1500 0

These values and volumetric water content with respective matric pressures be submitted with form .SWA.

- derived data from SWRC
  - bulk density (kg  $m^{-3}$ ; to be submitted with .SWC)
  - moisture content at field capacity  $(m^3 m^{-3})$
  - moisture content at permanent wilting point  $(m^3 m^{-3})$
  - Van Genuchten model parameters  $\theta r$ ,  $\theta s$ ,  $\alpha$ , n
  - Predicted Ksat (cm day<sup>-1</sup>)
- Data quality indicators (specific file for submission of laboratory QA information, XX2009SW.LQA, still to be defined)
  - Lab ID (laboratory that analysed SWRC)
  - Lab quality indices (to be defined) 0

#### 6.2. Data processing guidelines

Soil water retention curve models will be fitted to the raw data (Table 3). For forest soils, one of the best performing functions is the Van Genuchten equation defined by its empirical parameters  $\theta r$ ,  $\theta s$  and empirical constants  $\alpha$ , n and m = 1-1/n.

Calculation of these parameters can be done using the public domain RETC programme which may be downloaded from: <u>http://www.pc-progress.cz/Pg\_RetC.htm</u>. This software enables to predict Ksat from the SWRC measurements.

The Van Genuchten model parameters are also stored in the FutMon soil physical databases.

#### 6.3. Reporting guidelines

Data will be reported as foreseen in the data submission forms .SWC and .SWA.

#### 7. References

ISO 11274:1998(E). Soil Quality – Determination of the water-retention characteristic – Laboratory methods. International Organization for Standardization. Geneva, Switzerland. 20 p. (available at <u>www.iso.ch</u>)