^{14}C in cropland soil of a long-term field trial – experimental variability and implications for estimating carbon turnover

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Abstract

Because of their controlled nature, the presence of independent replicates, and their known management history long-term field experiments are key to the understanding of factors controlling soil carbon. Together with isotope measurements, they provide profound insight into soil carbon dynamics. For soil radiocarbon, an important tracer for understanding these dynamics, experimental variability across replicates is usually not accounted for, hence, a relevant source of uncertainty for quantifying turnover rates is missing. Here, for the first time, radiocarbon measurements of five independent field replicates, and for different layers, of soil from the 66-years old controlled field experiment ZOFE in Zurich, Switzerland, is used to address this issue. ^{14}C variability was the same across three different treatments and for three different soil layers between surface and 90 cm depths. On average, experimental variability in ^{14}C content was 12 times the analytical error but still, on a relative basis, smaller than variability in soil carbon concentration. Despite a relative homogeneous variability across the field and along the soil profile, the curved nature of the relationship between radiocarbon content and modelled carbon mean residence time implies that the absolute error of calculated soil carbon turnover time increases with soil depth. In our field experiment findings on topsoil carbon turnover variability would, if applied to subsoil, tend to underweight turnover variability even if experimental variability of the subsoil isotope concentration is the same. Together, experimental variability in radiocarbon is an important component in an overall uncertainty assessment of soil carbon turnover.

1 Introduction

Long-term agricultural field trials have long been recognized as important sources for understanding long-term management effects on soil parameters such as soil organic carbon content and turnover (Jenkinson, 1991). Their special value lays in their controlled nature, the long-term record of management activities, reliable soil and crop parameter records as well as site climate data. Many experiments have indicated that soil carbon responds sensitively to agricultural management and have allowed identifying sustainable management practices. Hence, these data sets are valuable sources of information also for developing or testing soil and ecosystem carbon models (Smith et al. 1997; Franko et al. 2011).

Soil carbon feedback to management is controlled by organic matter input as well as turnover and hence loss. Isotopes play an important role for unraveling soil carbon turnover rates. In complementation to records on carbon storage change over time, they deliver information on how fast new carbon replaces old carbon (e.g., Trumbore 1993). When the isotopic signature, i.e. $^{13}C/^{12}C$ or $^{14}C/^{12}C$ ratio, of the input material to soil is not constant over time, it induces a directed shift in the soil's isotopic signature. For a known shift in input signature, the subsequent change in the soil's isotopic signature allows estimating the replacement rate of old by new carbon. For example, changes from C3 to C4 vegetation or vice versa alter the $^{13}C/^{12}C$ ratio of the input and allow for estimating turnover rates (Balesdent et al. 1988). Besides stable $^{13}C/^{12}C$, also the radioactive isotope ^{14}C has a long history of application in soil carbon studies (Harkness et al. 1986; Jenkinson et al. 1992; Trumbore 1993). The introduction of extra ^{14}C to the atmosphere via nuclear bomb testing in the 50ies and 60ies of the last century and the subsequent diffusion of that label into terrestrial ecosystems has triggered a vast amount of research that makes use of the ^{14}C signature of soil carbon. The beauty of ^{14}C is given by its ubiquity and its potential to cover the whole relevant time frame of soil carbon turnover, ranging from years to millennia.

Information from both, controlled long-term field experiments and the soil's ^{14}C signature have been combined previously with the aim to get better insight into soil carbon dynamics (Table 1). These data are particularly useful for model development as the isotope reduces the degrees of freedom in the modeling approach, i.e., it constrains the carbon turnover dynamics and reduces the risk of giving right answers for the wrong reasons. For example, Jenkinson and Coleman (2008) used ^{14}C from the famous Rothamsted field trials to extend the existing Rothamsted Carbon Model RothC by a subsoil module. Hsieh (1993) took advantage of the oldest cropland experiments from the U.S., Morrow plot and Sanborn field, to get insight into labile carbon turnover. These and other applications as listed in Table 1 have not yet, though, considered the variability of radiocarbon in the field. Experimental variability, both across the field and within the soil profile, adds an important component of uncertainty to any modeling of terrestrial carbon. This variability needs to be known for reliable estimates of management-carbon storage feedbacks. For radiocarbon, the relatively high costs of the nowadays mostly used measurement by accelerator mass spectrometry (AMS) is a major obstacle to addressing experimental variability questions.

To our knowledge, experimental variability of soil ^{14}C using independent treatment replicates has not yet been addressed in any of the world's long-term cropland experiments listed in Table 1. Here, we aim to fill that gap by using recent ^{14}C measurements of mineral soil from a 66-years temperate long-term cropland trial in Zurich, Switzerland. Two questions are studied: i) what is the variability in soil radiocarbon content in independent replicates of a long-term field trial both in top- and subsoil, and ii) what are possible implications of experimental variability for the interpretation of soil carbon turnover estimates?

2 Material and Methods

The Zurich Organic Fertilization Experiment ZOFE was commenced in 1949 at the Swiss federal research institute for agriculture, Agroscope in Zurich. It is located at 420 m asl, receives an annual precipitation of 1040 mm and has a mean annual temperature of 9°C (1949–2009). The soil is a well-drained, carbonate-and stone-free, homogeneous haplic Luvisol (IUSS 2006) (texture: clay 14%, silt 27%, sand 54%). ZOFE comprises 12 different fertilization treatments with five replicates each (Fig. 1), applied to a 8-years crop rotation. The experiment is arranged in a systematic block design. A detailed experiment overview is provided by Oberholzer et al. (2014).

Here we present data from three treatments (Fig. 1) that were analyzed for their radiocarbon content. Treatment 'Null' received no fertilizer since 1949, treatment 'FYM+PK' receives 2.5 t farmyard manure (dry organic matter) every second year plus annually 235 kg K and 35 kg P as mineral fertilizer. Treatment 'N2P2K2Mg' receives 56/139 kg N (before/after 1981), 318/167 kg K (before/after 1991), 61/38 kg P (before/after 1991), 12/6 kg Mg (before/after 1991), and no organic fertilizer. All mineral fertilizer units are in kg ha^{-1} a^{-1}. Differences in crop productivity resulted in different residual plant carbon input of 556 (Null), 1085 (FYM+PK), and 1255 (N2P2K2Mg) (kg ha^{-1} a^{-1}) (Oberholzer et al., 2014).

Soil samples were taken in April 2012 from the center of each plot using a powered rotating soil auger (Humax, Burch AG, Rothenburg, Switzerland) down to depth of 90 cm. The auger is equipped with an outer shaft hosting a PVC inlet that gets filled with a volumetric soil sample of diameter 5.0 cm during drilling. Samples were pooled into segments 0-20, 20-30, 30-60, and 60-90 cm. For the present study, samples from the plough pan 20-30 cm were not analyzed. After extraction, samples were sieved < 2 mm, dried at 105 °C, roots were removed by hand, and an aliquot was finely ground. Prior to radiocarbon analysis, samples were pretreated using acid fumigation with 0.5M HCl to remove possible remnants from liming or traces of pedogenic carbonate. Soil radiocarbon content was measured by accelerator mass spectrometry at two different facilities, the radiocarbon laboratory of ETH Zurich, and the radiocarbon laboratory of the University of Bern, Switzerland. Both systems operate following the protocol of Synal et al. (2007). Radiocarbon concentrations are given as percent Modern Carbon (pMC) as defined by Stuiver and Polach (1977).

To study effects of experimental ^{14}C variability on soil carbon dynamics, we applied a common, time-dependent steady-state soil carbon turnover model. This has been first described by Harkness et al. (1986) and later been used as single or multiple pool version in various studies (e.g. Baisden et al. 2013; Gaudinski et al. 2000; Harrison 1996; Trumbore et al. 1996). The model gives mean residence times (MRT's) of soil carbon. Because we have no ^{14}C time-series available the most simple version of that model is applied representing a single-pool assumption as described in Leifeld et al. (2013). Although soil carbon time-series are better described by multiple pool approaches (Baisden et al. 2013), the assumption followed here is sufficient to discuss possible consequences of experimental variability for the interpretation of soil carbon dynamics. Because our data represent a single point in time, they do not allow adequate parameterization of a more complex model. Hence, we do not claim the presented turnover estimates to represent the *in situ* situation most realistically but rather to allow discussion of variability effects.

The effect of depth on pMC and carbon mean residence time was tested by univariate ANOVA separately for each treatment and for the aggregated sample across treatments.

3 Results and Discussion

Radiocarbon contents in the ZOFE plots average 100.2 (± 1.8 (1 SD)), 88.0 (± 3.00), and 76.5 (± 4.2) pMC for 0-20, 30-60, and 60-90 cm, respectively (Table 2). Across all 15 plots as well as when grouped by treatment, the depth effect was highly significant ($p<0.001$). Declining pMC values with soil depth are indicative for longer carbon mean residence times in the deeper layer of soil and have been reported frequently for soils that were not prone to substantial inputs from fossil carbon (Budge et al. 2011; Gaudinski et al. 2000; Jenkinson et al. 2008; Toyota et al. 2010).

Table 2 also indicates that the coefficient of variation (CV) of pMC for five independent plots, representing mostly experimental variability, lays between 1 – 7 percent (mean over nine treatments-layer combinations: 3.15 %). This is, for the present data set, 3 – 23 times the CV of 0.3% pMC of the analytical precision of the AMS measurement. Notably, the CV for soil organic carbon concentration is, per treatment and layer, on average 9.5 % (data not shown), thus three times that of the radiocarbon content. There was no significant depth effect on the coefficient of variation ($p=0.16$), hence, ^{14}C variability does neither increase nor decrease with depth. At the same time, the CV grouped by treatment was statistically not different between 'Null', 'FYM+PK', and 'N2P2K2Mg' (p=0.64). The latter implies that experimental ^{14}C variability as measured in ZOFE is related to site – or soil inherent properties rather than to agricultural management

Although ^{14}C variability did not significantly change with depth, it influences the variability of the derived soil carbon MRT's differently in the three layers. This can be studied by calculating turnover for the range of pMC data expressed by their average confidence interval (CI). Here, we calculate the CI of MRT's from the average coefficient of variation of i) 0.30 % of pMC (analytical error) and ii) 3.15 % of pMC (experimental variability, average of nine individual sample sets with each n=5) over the measured data range. We use these average CV's for calculating CI's over the whole data range because neither treatment nor depth significantly influenced pMC variability in the field. Owing to the combination of i) a non-constant atmospheric radiocarbon concentration as a result from long-term and short-term $^{14}CO_2$ fluctuations and ii) exponential radioactive decay in the soil, the relationship between pMC and MRT is non-linear. This is illustrated for a series of homogeneous soil pools of different age. Radiocarbon signatures of such a pool series with range from 70 – 105 pMC (resembling the span found in the soil data, Table 2), convert to MRT's of between 3891 and 156 years (Fig. 2). The pMC-age curve becomes steeper at smaller radiocarbon concentrations. Whereas the central curve in Fig. 2 gives results for the mean pMC, the inner and outer bands represent the 95% CI of i) the average variability owing to analytical error only (inner band) and ii) the average experimental variability in the soil. These bands give upper and lower probability limits for the calculated MRT and they deviate the further from their mean the older the carbon is. For example, a MRT of 3891 (CI 3392 - 4453) years is assigned to a soil carbon pool with signature pMC = 70 (CI 67.50 – 72.50 pMC), whereas the same relative uncertainty for a mean pMC of 105 (Cl 101.20 – 108.80 pMC) converts to a MRT of 156 (CI 92 – 268) years.

Fig. 3 further illustrates the principle. Soil carbon from 60 – 90 cm, carrying a signature of e.g. 75 pMC, has a calculated MRT of 2947 years with deviations of + 477 and – 428 years, referring to the variability among five independent field replicates. The uncertainty range is reduced to +193 and - 129 years (mean MRT 321 years) for a pMC of 100, roughly representing the current topsoil. While the absolute uncertainty declines the younger the soil becomes, the relative uncertainty increases in the opposite direction (Fig. 3, right). Fig. 3 also exemplifies the wider uncertainty band, over the calculated pMC range, when experimental variability and not only measurement error is accounted for. At pMC 70, the uncertainty range of MRT's considering experimental variability is 4.2 times that of measurement error only. This factor increases to 6.5 at pMC 105, indicating that for younger soil carbon the omission of experimental variability introduces a larger relative uncertainty than for older soil carbon.

4 Conclusions

Soil radiocarbon dating from a long-term agricultural experiment indicates that experimental variability of this parameter is many times the analytical error. Experimental variability seems neither controlled by management nor by soil depth. Conversion of relative uncertainty in radiocarbon content to relative uncertainty in carbon turnover reveals a higher sensitivity of carbon turnover to ^{14}C variability in deeper soil layers that contain older carbon. Consequently, when soil samples from a long-term field trial are pooled per depth and treatment for ^{14}C analysis, the underestimation of the actual experimental variability of soil carbon turnover is larger for subsoil samples where long-lived C pools are more abundant.

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Table 1. Long-term agricultural field experiments where radiocarbon was used to derive soil carbon turnover estimates.

Experiment	Country	^{14}C time series	^{14}C available from independent and randomized treatment reps.	^{14}C measured in > 1 layer	Reference
Lethbridge	Canada	yes	no	No	Ellert and Janzen (2006)
Askov	Denmark	yes	no	No	Bol et al. (2005)
Bad Lauchstädt	Germany	yes	no	No	Ludwig et al. (2007)
Halle	Germany	no	no	yes	Rethemeyer et al. (2007); Flessa et al. (2008)
Rotthalmünster	Germany	no	no	yes	Rethemeyer et al. (2007); Flessa et al. (2008)
DOK	Switzerland	yes	no	No	Leifeld et al. (2009)
ZOFE	Switzerland	no	yes	yes	This study
Rothamsted	UK	yes	no	yes	Jenkinson et al. (2008)
Morrow Plots	USA	yes	no	No	Hsieh (1992)
Sanborn Field	USA	no	no	No	Hsieh (1992)

Table 2. Percent modern carbon (%) (±1 sigma uncertainty[1]) of organic carbon in soil samples from the ZOFE trial taken in 2012 for three treatments and three soil layers. Lines 'CV' indicate the coefficient of variation for each treatment/depth combination. Plot number according to Fig. 1. Sign 'x' refer to lab ETH, sign 'o' to lab Bern.

Treatment	Plot number	0-20 cm	30-60 cm	60-90 cm
Null	1	97.54 (0.36)x	84.24 (0.32)x	73.52 (0.33)x
Null	19	102.33 (0.22)o	85.44 (0.34)x	77.17 (0.32)x
Null	28	101.01 (0.36)x	84.74 (0.33)x	74.75 (0.33)x
Null	46	101.00 (0.22)o	89.90 (0.20)o	74.03 (0.17)o
Null	51	100.69 (0.22)o	83.48 (0.19)o	74.90 (0.39)x
CV (%)		1.77	2.96	1.87
FYM+PK	6	100.78 (0.22)o	91.26 (0.21)o	78.98 (0.18)o
FYM+PK	24	99.80 (0.22)o	89.55 (0.20)o	78.52 (0.18)o
FYM+PK	33	101.20 (0.22)o	90.05 (0.20)o	73.11 (0.17)o
FYM+PK	39	102.33 (0.38)x	90.52 (0.20)o	86.10 (0.19)o
FYM+PK	53	96.08 (0.21)o	88.95 (0.20)o	75.37 (0.18)o
CV (%)		2.39	0.98	6.27
N2P2K2Mg	12	100.13 (0.37)x	88.36 (0.33)x	74.93 (0.18)o
N2P2K2Mg	18	100.49 (0.22)o	85.36 (0.20)o	76.11 (0.18)o
N2P2K2Mg	27	97.79 (0.37)x	93.88 (0.35)x	85.01 (0.34)x
N2P2K2Mg	45	101.55 (0.46)x	86.52 (0.33)x	71.40 (0.31)x
N2P2K2Mg	59	101.06 (0.22)o	87.84 (0.20)o	73.16 (0.17)o
CV (%)		1.45	3.72	6.94

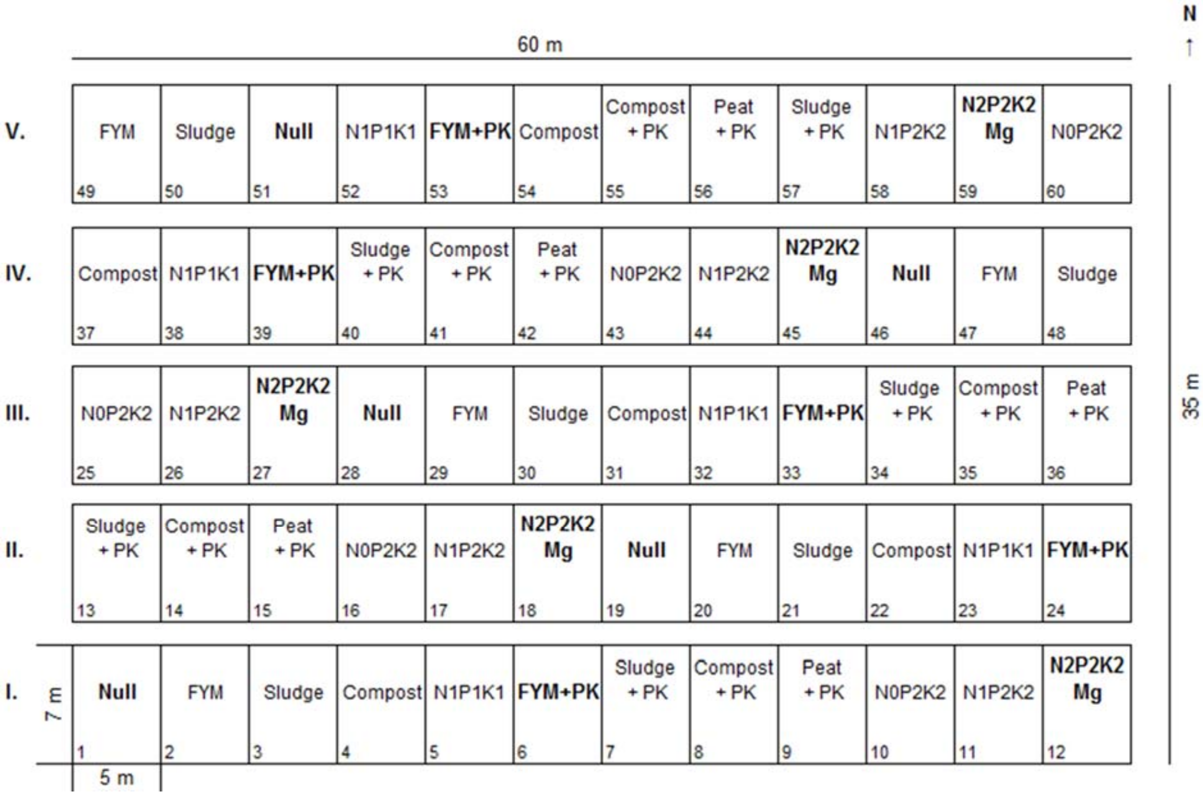
[1] sigma = standard deviation

Figure captions

Fig. 1. Spatial arrangement of the ZOFE field trial in Zurich indicating twelve treatments à five replicates arranged in five blocks (I. – V.). Plot numbers in lower left corner are listed together with measurements in Table 1. Treatments in **bold** (Null, FYM+PK, N2P2K2Mg) were used for the present study. For a detailed description of all treatments, please see Oberholzer et al. (2014).

Fig. 2. Relationship between percent modern carbon (pMC) and calculated carbon mean residence time (MRT) using a time-dependent steady-state single pool turnover model. The inner line refers to mean values; the inner, dashed band to the 95% uncertainty range related to the average ^{14}C analytical error, and the outer, solid band to the 95% uncertainty range related to the average experimental ^{14}C variability in the field.

Fig. 3. Comparison of absolute (left) and relative (right) deviation of calculated MRT's from the mean, expressed as 95 % confidence interval of i) average analytical errors (inner, dashed line) and ii) average experimental variability in the field (outer, solid lines). In the ZOFE trial MRT's of below 200 years resemble topsoil 0-20 cm, of c. 1200 years to 30 – 60 cm, and of c. 2600 years to 60 – 90 cm.



V.	FYM 49	Sludge 50	**Null** 51	N1P1K1 52	**FYM+PK** 53	Compost 54	Compost + PK 55	Peat + PK 56	Sludge + PK 57	N1P2K2 58	**N2P2K2 Mg** 59	N0P2K2 60
IV.	Compost 37	N1P1K1 38	**FYM+PK** 39	Sludge + PK 40	Compost + PK 41	Peat + PK 42	N0P2K2 43	N1P2K2 44	**N2P2K2 Mg** 45	**Null** 46	FYM 47	Sludge 48
III.	N0P2K2 25	N1P2K2 26	**N2P2K2 Mg** 27	**Null** 28	FYM 29	Sludge 30	Compost 31	N1P1K1 32	**FYM+PK** 33	Sludge + PK 34	Compost + PK 35	Peat + PK 36
II.	Sludge + PK 13	Compost + PK 14	Peat + PK 15	N0P2K2 16	N1P2K2 17	**N2P2K2 Mg** 18	**Null** 19	FYM 20	Sludge 21	Compost 22	N1P1K1 23	**FYM+PK** 24
I.	**Null** 1	FYM 2	Sludge 3	Compost 4	N1P1K1 5	**FYM+PK** 6	Sludge + PK 7	Compost + PK 8	Peat + PK 9	N0P2K2 10	N1P2K2 11	**N2P2K2 Mg** 12

Fig. 1.

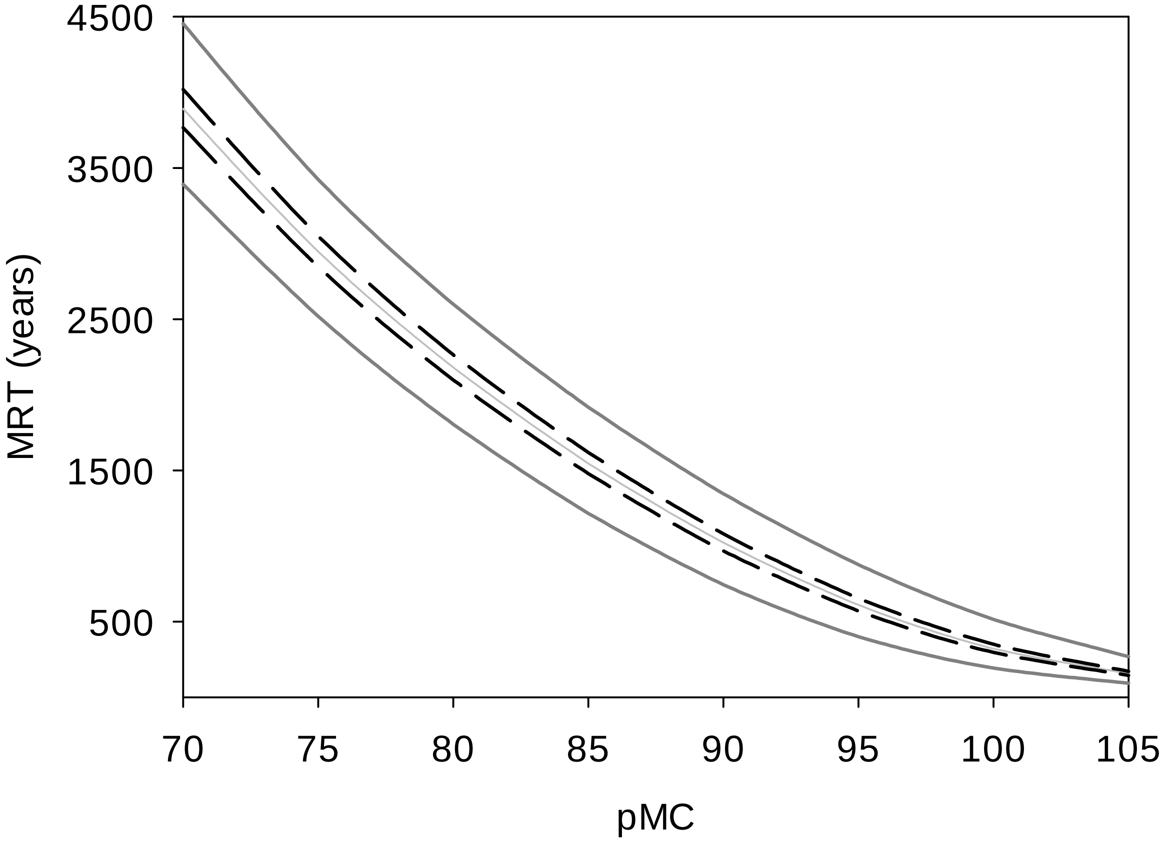


Fig. 2

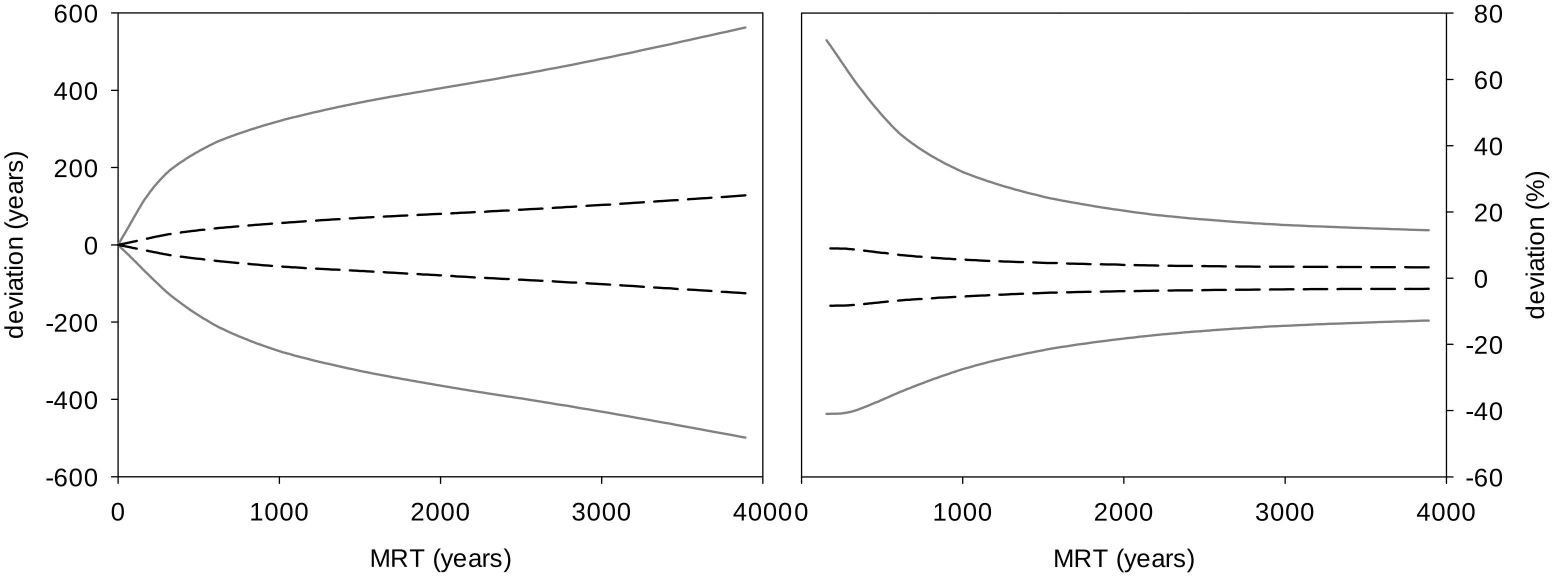

600
400
200
0
-200
-400
-600
deviation (years)
0
1000
2000
3000
4000
MRT (years)
80
60
40
20
0
-20
-40
-60
deviation (%)
0
1000
2000
3000
4000
MRT (years)

Fig. 3