Author's response to referee comments on: "Radiocarbon dating of alpine ice cores with the dissolved organic carbon (DOC) fraction" by Fang et al.,

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We would like to thank the reviewers for their constructive comments that helped us to improve the accuracy of our evaluation of the potential of DOC for radiocarbon dating. Our responses to their comments are in blue.

Anonymous Referee #2

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This manuscript presents the first results of a technique that utilizes 14C of DOC to date alpine ice cores. The basal sections of alpine ice cores are difficult to date because of high degree of ice thinning (making layer counting impossible) and complex ice flow. The approach used in this study is very analytically challenging, and in my opinion the method has been carefully developed and tested. The analytical precision on the F14C values is impressive considering the small sample sizes. Considering that this approach requires smaller ice samples (_250 g) than the earlier approach developed by the same group that uses insoluble organic carbon, this now seems like the most promising technique for dating alpine basal ice. Overall, I think that this is an exciting study that is in principle well suited for The Cryosphere. However, I think the study and manuscript also have some weaknesses that should be addressed.

Major Comments:

One of the major goals of this study / manuscript is validation of the 14C-DOC technique. In my opinion the manuscript doesn't fully achieve this. The main approach for this evaluation is comparison with the WIOC-14C results. But those results seem to be affected (to varying degrees) by 14C interference from carbonate dust in the samples. Are the authors able to measure a few samples from a layer-counted Greenland ice core, for example, to provide a more robust validation? I realize that this may be difficult, both because of lower DOC concentrations and ice availability, but perhaps a core from a coastal ice cap such as Renland could be a good target?

As we discuss in the manuscript, the carbonate effect on WIOC is small and within the range of the analytical uncertainty. The ages of ice obtained with WIOC were validated before by comparison with independently dated ice (by annual layer counting or conventional ¹⁴C dating of microfossils contained in the ice) and this was published and we cite that (Uglietti et al., 2016). We therefore don't see the necessity to measure further samples from Greenland to validate the WIOC dating method used as benchmarker in this study.

I think it would be valuable to provide a more complete analysis of the overall dating uncertainties. If 14C-WIOC is the benchmark measurement that is being used for validation of 14C-DOC, then the uncertainties in the 14C-DOC ages need to fully reflect the uncertainties associated with us 14C-WIOC (see more on this below). Alternatively, if the authors consider 14C-DOC to be an inherently superior approach (as compared to 14C-WIOC), then a more clear argument needs to be made for this. I think the uncertainties associated with the correction for carbonate dust (for WIOC-14C) need to be more thoroughly considered. The authors provide some helpful discussion of this in the supplement (starting on line 33), but I'm not convinced that the uncertainties are fully accounted for. For example, it seems to me that F14Ccarb could in principle range from 0 to 1 depending on the source of the carbonate. One could imagine a situation with seasonally-drying lakes in arid regions, for example, where the carbonate dust at the surface would be close to modern in its 14C signature. The C /Ca ratio in dust derived from dolomite would be twice as large as what is being used in Supplement equation 2. The effect of these additional uncertainties may be visible in figure 3b – while the

correction makes the Chongce samples look more reasonable, two of the Belukha samples now fall off the trend.

Regarding the main issues raised by both reviewers, we do understand the concerns and would like to thank for the careful evaluation of the manuscript. While details can certainly be discussed, we however are a bit surprised that the reviewer here asks for an even more detailed analysis of the overall dating uncertainties. This considering the fact, that we discuss discrepancies, which are barely statistically significant (below the analytical detection limit for 4 out of 3 sites). Since the method of ¹⁴C-WIOC dating has been validated previously (see Uglietti et al., 2016) we also think that it is justified to use this as a benchmark. Anyway, in the revised version of the manuscript, we will include the valuable suggestions of the two reviewers to even further improve this in-depth discussion (see comments related to in-built ages and insitu production). Consequently, with the new consideration of potential in-situ contribution to $DO^{14}C$, this fraction will no longer be considered to be the superior approach. Instead, for both fractions, we are confident to be able to provide more precise and accurate guidelines about potential limitations in the accuracy for both approaches.

In the new calculation about in-situ production, we find about 50% of the offset between F¹⁴C DOC-WIOC can be explained by in-situ production, see related comment. Although numbers of carbonates contribution to WIOC will thus change, the related modeling approach will still be part of the manuscript. First, we would like to stress, that this modeling results should not be viewed as a mean for correction of WIOC F¹⁴C results. Instead, the aim was to test the hypothesis that a less than 100% efficient carbonate removal procedure could potentially explain the observed offset between F¹⁴C of WIOC and DOC with the required level of efficiency being plausible (high and only slightly less than 100%). As we stated, the future aim would be to improve the carbonate removal process, not to correct WIO¹⁴C for a potential carbonate bias. Under this aspect, we agree with the reviewer, that the selection of the parameter space for F¹⁴C_{carb} and the carbonate-to-calcium ratio is critical. This is what we already stated in the supplement. Our opinion is that therein (lines 33-42 as pointed out by the reviewer), the range of possible F¹⁴C values for carbonates or carbonate-to-calcium ratios was already reasonably explored. We discussed the robustness of the modeling results, provided an idea of the associated uncertainty by exploring the parameter space. However, not intending to apply a correction for (a potential) carbonate bias, we thereby did not focus to precisely quantify uncertainties but considered an evaluation of those to be sufficient for determining if the model results are robust in terms of the estimated removal efficiency required for explaining the observed offset. For explanation in this response only, we will summarize below the reasoning behind our approach and point out what was already addressed in the supplement:

(1) The carbonate removal efficiency by the acidification step very likely depends on the source and transport of the mineral dust (geological form, particle size) affecting the solubility. Thus, tuning the model for a common carbonate removal efficiency (x_{eff}) instead of allowing this parameter to vary for individual sites will likely will not yield the best possible approximation between the observations and the modeled data as reflected in Figure 3b. In the Supplement, line 38-45, we thus discussed results with a model set-up allowing the carbonate removal efficiency to be different for the individual sites (within +- 4% for all sites). This showes, that the estimated value for x_{eff} may not be a precise, best value for each site but a robust average estimation.

(2) As pointed out by the reviewer, the abundance of carbonates in their different geological forms is likely different for each site (similar to point (1) dependent on the source region) and thus the value for the C/Ca ratio is likely not a fixed single value. However, we do not know what value would be most appropriate for each of the sites. Allowing this parameter

to be free in the model, i.e. allowing it to tune to a "best" value for each individual site would thus be speculative and over-tuning of the model. Anyhow, please note, that in the Supplement we did some evaluation of changes to this parameter by using a different value for this ratio (0.8 instead of 0.5), which again provided an estimate about the robustness/uncertainty of the final result for x_{eff} .

(3) $F^{14}C_{carb}$ very likely differs dependent on the site (again relates to the source). Best, $F^{14}C_{carb}$ would be selected individually for each sample, based on the difference in age of the contemporary atmosphere to the assumed age of the source carbonate at that time. But also here, the available literature is sparse and without speculation we cannot assume "precise" values. The value suggested by the reviewer ($F^{14}C_{carb} = 1$) would certainly not be a reasonable value to assume, it reflects the year 1950 AD. We think his idea was that at the time when a certain snow/ice layer now at some depth in the glacier was on the surface, mineral dust (carbonates) with an $F^{14}C$ being contemporary at that time were deposited as impurities in this layer. In this case the upper limit for $F^{14}C_{carb}$ would be equal to the $F^{14}C$ corresponding to the age of this layer. It is clear, that a close to contemporary $F^{14}C$ would not introduce a bias and carbonates then could not explain the observed offset between DOC and WIOC. However, layers from "input from seasonally-drying lakes in arid regions" may occur as individual, special events but as such do not represent the norm (also for some sites, this can be excluded as a possibility entirely). Anyway, in the Supplement we already calculated using a different value for $F^{14}C_{carb}$ (0.05).

To conclude, we are very much aware that if the intention would be to model the observed offset as closely as possible to come up with a correction, the model-set up and parameter space could be explored in even more detail (note, that in principle, by tuning each parameter for individual sample, we can reproduce the offset to nearly 100%). However, in our opinion this would only be justified if knowing with absolute certainty that a contribution from carbonate carbon to F¹⁴C of WIOC exists and one could start to quantify and investigate the individual parameter values in detail. Again, we would like to point out that we look at discrepancies very close to the detection limit (or even below). Here we can thus only provide evidence that such an effect exists, causing a systematic offset in the direction observed. As mentioned in the beginning, now being aware that in-situ ¹⁴C contribution to DOC cannot be completely ruled out, the potential "carbonate bias" becomes even smaller (i.e. the removal efficiency even higher) than what we estimated before.

Minor Comments:

Line 73. For water-soluble organics sourced from biomass burning, there may be an age offset due to the older ages of the burned material. While this is probably small compared to the measurement uncertainties, it would still be worth mentioning briefly. Similar comment for organics sourced from oceanic emissions (affected by ocean radiocarbon reservoir effect). We agree with the reviewer that there is an in-built radiocarbon age from old carbon reservoirs to be assumed, actually both, for the DOC and WIOC fractions. Depending on the region, the mean reservoir age of these potential sources is likely variable. For the reservoir age from biomass burning the mix of tree ages in the forests along the moisture source/transport pathways may differ and for organics sourced from oceanic emissions, both continentally of the site and the strength of upwelling/mixing with ocean deep waters in the source region will have an effect. In any case, the reviewer is correct when assuming that these potentially inbuilt ages are small, compared to the analytical uncertainty. In the study of Uglietti et al. (2016) where WIO¹⁴C ages were compared to ages derived by independent methods, no bias was identified.

The mixed age of trees in Swiss forests today is estimated to be slightly less than 40 years (Mohn et al., 2008). Back in time, prior to extensive forest management (e.g. cutting of trees in Europe was likely older. However, the most abundant European tree species reach ages of less than 100 years only. Zhang et al. (2017) found a relatively young stand age for Chinese forests mostly due to the large proportion of newly planted forests (0–40 years old), which are more prevailing in south China. Older forests (stand age>60 years old) are more frequently found in east Qinghai-Tibetan Plateau and the central mountain areas of west and northeast China, where human activities are less intensive. The oldest mean stand age was found to be 136 years old, and the youngest 18 years. For old-growth forests, e.g. in British Columbia (USA) with very limited human forestry management (old, dead tree logs still present), average in-built ages in charcoal of around 200-300 years were determined (Gavin, 2001). Note that hard-wood trees in this region can become exceptionally old (several hundreds of years) but maybe can assumed to be also representative for Siberian forests.

While charcoal is associated 100% with biomass burning (as can be assumed also for EC), WIOC is estimated to equally originate from direct biogenic emissions (similar in ¹⁴C content as the contemporary atmosphere, i.e. atmospheric CO₂) and biomass burning with around 50% (before the use of fossil fuels, Minguillon et al., 2011). For biogenic DOC, May et al. (2013) estimated a turnover-time of around 3-5 years for a study site in Switzerland. This corresponds to a contribution of around 20% from biomass burning which can easily be derived by using the mixed age of trees in Swiss forests of 40 years (see above). Note that a living tree is build up by rings with the outermost, youngest ring having contemporary age and thus, if a living tree of e.g. 40 years is completely burned, the released carbon will have an average $F^{14}C$ corresponding to a mean age of 20 years only. Biomass burning also includes contribution from burning of grasslands and/or bushfires (young in age). Based on the above, a potential in-built age from biomass burning to the WIOC and DOC fraction can be estimated. When considering a value of 150 ± 100 years for the mean age of burned material (aged wood plus grass and bushes), the potential in-built age from biomass burning for WIOC and DOC results with around 75 ± 50 years and 30 ± 20 years, respectively.

Collle Gnifetti is located more than 850 km away from the Atlantic Ocean and 250 km from the Mediterranean Sea (not in the main transport trajectory) and the other sites are even much more continental. For ice cores from the Alps the concentrations of methanesulfonate (MSA), a marine organic tracer, are more than one order of magnitude lower compared to the terrestrial organic tracers (e.g. formate, acetate).

In conclusion, regarding the analytical uncertainty of around 10-20% for radiocarbon dating by WIOC and DOC, these in-built ages are insignificant, at least for samples being older than a few hundred years. We will add a sentence or two to the manuscript as suggested and briefly summarize what we outlined in detail here.

Line 191. Why is the Libby half-life of 14C being used here instead of the more accurate value of 5730 yrs?

Figure 3b legend. Use a label that's more descriptive than "corr" for the corrected data; perhaps just say "corrected results"

The definition of the conventional ¹⁴C age is calculated from -8033 * ln ($F^{14}C$) defined by Stuiver and Polach (1977). This equation is based on the very original Libby half-life of 5568 years. This value was revised in the early 1960s to 5,730 ± 40 years, which meant that many calculated dates in papers published prior to this were incorrect. For consistency with these early papers, it was agreed at the 1962 Radiocarbon Conference in Cambridge (UK) to use the "Libby half-life" of 5568 years. Radiocarbon ages are thus still calculated using this half-life,

and are known as "Conventional Radiocarbon Age". Since the calibration curve (IntCal) also reports past atmospheric ¹⁴C concentration using this conventional age, any conventional ages calibrated against the IntCal curve will produce a correct calibrated age.

Figure 3b legend will be modified to "w/o carbonate contribution" indicated as without carbonate contribution.

Line 278. "As described in Section 3.2, no significant difference between F14C of DOC and WIOC was observed for the ice samples from Colle Gnifetti, Belukha and SLNS (Figure 3)." This is incorrect. Based on the figure, the differences seem significant at the 1-sigma level for several samples, and at the 2-sigma level for at least 1 sample.

Our data set, with the corresponding uncertainties does not allow us to conclude that there is a significant difference between $F^{14}C$ of DOC and WIOC particularly for the data set from these three sites. We applied a Mann Whitney u-test (U=79.5, n1=n2=14, p=0.41>0.05) indicating the $F^{14}C$ (DOC) and $F^{14}C$ (WIOC) to be not significantly different. Easiest to see that this is true and that there is no statistical evidence of a difference is the fact that the 95% confidence interval in Figure 3 includes the 1:1 line. Of course, individual data points are expected to lie outside the 1 or 2 sigma level of the Gaussian distribution of the entire data set. This basically is the definition of these levels. Around 3 out of 10 individual data points are expected to be outside the one sigma range (68%) and around 1 out of 10 outside the 2 sigma range (95%). Therefore, the observation made by the reviewer is certainly correct but also exactly what is expected.

Anyhow, our formulation might not have been entirely clear and we will change to:

"As described in Section 3.2, there is no statistical evidence of a significant difference between $F^{14}C$ of DOC and WIOC for the data-set from the Colle Gnifetti, Belukha and SLNS ice samples (Figure 3)."

Table 5 / Figure 4 and associated text. I think that the discussion of the limitations of this comparison to the previous age estimates should be expanded to provide some more detail / caveats associated with the comparison. For example, how close were the CG and Chongce cores to each other / do we even expect the basal ice to be of similar age? Is the comparison at Belukha still meaningful given the different core locations?

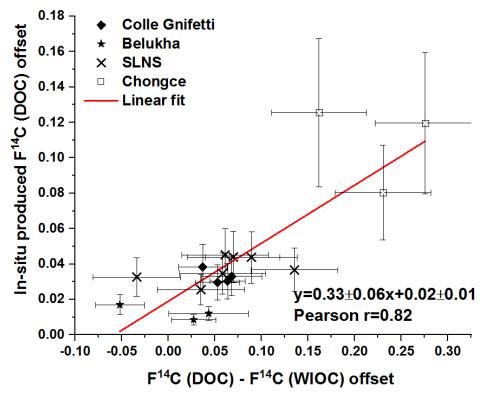
The two cores from CG were collected in 16 m distance from each other, the ones from Chongce at less than 2km distance from each other on this extended ice cap. Therefore we expect similar ages (for CG the age presented is not from basal ice). For Belukha we would also expect a similar glacier history, since both sites are only about 2 km apart and at the same elevation. We will add this information.

While I think that the authors' conclusion that there is no evidence that in situ 14C is affecting the 14C-DOC measurements is likely correct, the authors should do a better job of supporting this conclusion. For example, how can you be certain that the higher 14C-DOC as compared to 14C-WIOC in most samples is not due to in situ 14C? The largest offsets are observed at Chongce, which has the highest altitude and therefore should in principle have the highest in situ 14C production rates.

Thank you for this comment. We excluded an effect from ¹⁴C in-situ production in the initial version since no obvious super modern values were measured, in contrast to the findings of May (2009). However, thanks to the comments of both reviewers we realized, that this observation alone is not sufficient for such a conclusion.

We followed the suggestions of the reviewer and will add a section in the revised manuscript to more carefully estimate the potential of ¹⁴C in-situ production on DO¹⁴C dating for each site.

For the production rate P_0 we used the literature values for different altitudes from Lal et al., 1987 in combination with the estimates for the latitudinal dependence of P_0 from Lal 1992. The annual accumulation rates for the new cores from CG, Belukha, and Chongce are not available at this point. Therefore, the according values were approximated based on previous studies for these sites. For CG from Jenk et al., 2009, for Belukha from Henderson et al., 2006, and for Chongce from Hou et al., 2018 for core3 (Table S1 and Table S2, see below). All these cores were drilled closeby of the new sites (see response below) and although some variation cannot be excluded, the potential difference is assumed to be relatively small with a negligible effect for the calculations here. For the SLNS core the annual accumulation rate has not been determined yet. Instead we estimated the annual accumulation rate ($0.21 \pm 0.11 \text{ m w.e./yr}$) by using a 2-dimensional glaciological flow model (2p model, Bolzan, 1985; Thompson et al., 1989) to fit the DO¹⁴C dates. We find an estimated average offset of DOC-F¹⁴C values due to in-situ production of 0.044 ±0.033. Generally, we find a good correlation between the observed F¹⁴C (DOC)-F¹⁴C (WIOC) offset and the calculated ¹⁴C in-situ contribution to DO¹⁴C with the in-situ production explaining about 50% of the observed difference (R=0.82, see Figure below).



Further, as shown in this Figure, it is evident that the potential effect of in-situ production is strongest for the samples from Chongce. Based on the calculation, this is explained by the high altitude in combination with a low annual accumulation rate of this site (Table S1 and Table S2). For sites from lower altitude and/or characterized by higher accumulation rates, the contribution of ¹⁴C in-situ production to the DOC fraction is small and within the analytical uncertainty. In addition, the effect of in-situ production also depends on the carbon concentration, being lower the higher the concentration. In conclusion, under most conditions, in-situ production is not significant. Only for ice samples from extrem altitude, especially in combination with low accumulation rates, DO¹⁴C dating results should be carefully interpreted. Under these conditions a potential contribution from ¹⁴C in-situ production cannot be excluded and could introduce an age bias exceeding the analytically derived age distribution.

Changes to manuscript:

New section about in-situ production (partly in supplement), new figures, adapted section about carbonates (discussion, also in abstract and conclusion – effect likely even smaller than estimated before), new section combining in-situ and carbonate effects.

Site	Coordinates Elevation	Location	Total Length (m)	Accumulation (m w.e.year ⁻¹)	References
Colle Gnifetti	45°55'45.7''N, 7°52'30.5''E 4450 m asl.	Western Alps Swiss-Italian border	76	0.45*	Sigl et al., 2018
Belukha	49°48'27.7"N, 86°34'46.5"E 4055 m asl.	Altai Mountains Russia	160	0.5*	Henderson et al., 2006
SLNS	38°42'19.35"N, 97°15'59.70"E 5337 m asl.	Shulenanshan Mountain China	81	0.21#	Hou et al., submitted
Chongce core1	35°14'5.77"N, 81°7'15.34"E 6010 m asl.	Kunlun Mountain China	134	0.14+	Hou et al., 2018

Table S1. Characteristics of the study sites.

*Accumulation rate from previous publication for the core collected in 2003 from the same drilling sites with 16 m distance.

&Accumulation rate from core collected in 2001 at 90 m distance from the drilling site in 2018.

[#]Accumulation is estimated from 2p model using DO¹⁴C dates.

⁺Accumulation rate is from Chongce core 3 that is located at the same plateau with less than 2 km distance.

Table S2. Estimated in-situ production contribution to the DOC fraction.

Core section	Ice	Carbon	Depth	Po	¹⁴ C in-situ	Change of	DOC-WIOC
	mass	mass	(m	(¹⁴ C atom/g ice year)	(no. of atoms)	F ¹⁴ C in DOC	F ¹⁴ C Offset
	(g)	(µg)	w.e.)			fraction	
CG110	171	18.9	55.8	328	1197	0.033±0.011	0.068 ± 0.032
CG111	207	25.5	56.3	328	1197	0.030 ± 0.010	0.053 ± 0.024
CG112	248	23.6	56.7	328	1197	0.038 ± 0.013	0.037 ± 0.026
CG113	246	29.5	57.0	328	1197	0.030 ± 0.010	0.064 ± 0.019
Belukha412	172	28.5	142.7	286	921	0.017 ± 0.006	-0.052±0.026
Belukha414	128	41.9	143.9	286	921	0.009 ± 0.003	0.027 ± 0.024
Belukha415	102	23.7	144.5	286	921	0.012 ± 0.004	0.043 ± 0.043
SLNS101	238	44	47.9	345	2666	0.044 ± 0.011	0.070 ± 0.050
SLNS113	213	39.4	54.4	345	2656	0.044 ± 0.011	0.089 ± 0.050
SLNS122	234	57.9	58.1	345	2651	0.033 ± 0.008	-0.034±0.047
SLNS127	183	57.8	60.5	345	2647	0.026 ± 0.006	0.029 ± 0.047
SLNS136	220	48.3	64.7	345	2641	0.037 ± 0.009	0.135±0.047
SLNS139	208	48.1	66.5	345	2638	0.035 ± 0.008	0.058 ± 0.046
SLNS141-142	246	43.8	67.7	345	2636	0.045 ± 0.011	0.061 ± 0.047
CC237	208	28.5	113.7	497	5371	0.120 ± 0.040	0.275±0.054
CC244	167	21.7	117.6	497	5353	0.126 ± 0.042	0.161±0.051
CC252	120	24.3	120.2	497	5341	0.080 ± 0.027	0.231±0.051

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