Response to reviewers' and Editors' Comments

Reviewer 1 comments.

I have the following suggestions that I urge the authors to consider in their final version.

1. Q: A major issue in this paper is that a regional reaction-transport model is not used. It has been demonstrated that in Beijing, under many meteorological conditions, a large quantity of the atmospheric pollutants can be transported in from the south. Thus, the in-cloud water and aerosol water parameters that were responsible for the secondary atmospheric sulfate formation in Beijing are in fact conditions a couple of days older and of different locations. This point is not considered in the text and should be added to the discussion and to be mentioned as a caveat in the abstract.

A: Thanks for the comment. It's true that the polluted air mass could have been processed under haze conditions in Beijing and its south area for a couple of days before reaching the sampling site. In this case, since haze is a regional phenomenon with similar meteorological conditions in Beijing and its south area (Zheng et al., 2015a; Zheng et al., 2015b; Wang et al., 2014), atmospheric parameters observed during haze at the sampling site should be typically representative for Beijing and its south area within the previous 2–3 days. As the reviewer mentioned, secondary sulfate formation in Beijing can be via the oxidation of atmospheric pollutants during transport and from local reactions. In the situation that secondary sulfate formation occurs during transport, since haze is a regional phenomenon with similar meteorological conditions in Beijing and its south area, our local atmospheric conditions-based calculations should be representative for secondary sulfate formation during transport, e.g., secondary sulfate formation within the previous 2-3 days in the south area. In fact, the overall sulfate production rate calculated on the basis of local atmospheric conditions increased from NPD to PD and basically coincided in time with the observed sulfate levels (Fig. 4 in the main text), which supports our local atmospheric conditions-based calculations being representative of secondary sulfate formation locally and during transport. To remind readers that our calculation is based on local atmospheric conditions rather than a regional reaction-transport model, we have changed the expression "our calculations" into "our local atmospheric conditions-based calculations" in the discussion and the abstract. We note that our local atmospheric conditions-based calculations may be not as robust as a regional reaction-transport model, which inspires future modelling work with the constraint of isotope data reported here to further improve the understanding of secondary sulfate

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formation during Chinese haze. Therefore, we have added the expression "Our local atmospheric conditions-based calculations illustrate the utility of $\Delta^{17}O(SO_4^{2^-})$ for quantifying sulfate formation pathways, but this estimate may be further improved with future regional modelling work." as a caveat in the abstract in lines 36-38.

2. Q: O_3 is counted as one of the oxidants in the heterogeneous pathway in the manuscript (Eq. 14). This is debatable. It is very likely that in mineral dust surface or in aerosol water, O_3 concentration can be negligible. If O_3 is not counted, the estimated NO₂ pathway would become much less important in the conclusion. In fact, this would be consistent with the possibility that NO₂ may not be playing an important role in S(IV) oxidation after all, as some has suggested. At least, this point should be discussed in the manuscript.

A: Thanks for the comment. Our Δ^{17} O observations are highly sensitive to ozone oxidation, and suggest a minor but significant role for this sulfate formation pathway. The Δ^{17} O of sulfate produced via heterogeneous reactions ($\Delta^{17}O_{hec}$) was calculated to be respectively 1.8 ‰, 3.1 ‰, 1.4 ‰, 0.1 ‰ and 0.8 ‰ for PD of Case I–V, which has been described in lines 308-309 in the present manuscript to replace the former expression "the Δ^{17} O of sulfate produced via heterogeneous reactions ($\Delta^{17}O_{hec}$) was calculated to range from 0.1 ‰ to 3.1 ‰ in our study." in the last manuscript. We also have added the description that "Since $\Delta^{17}O(SO_4^{-2})$ produced via H₂O₂ oxidation is 0.7 ‰, smaller than $\Delta^{17}O_{het}$ in Case I–III and V, O₃ oxidation must contribute to heterogeneous sulfate production." in lines 309-311. As for whether O₃ oxidation is negligible, our $\Delta^{17}O$ -constrained calculation suggests heterogeneous O₃ oxidation contributes 14 ‰, 11 ‰, 9 ‰, 11 ‰, 8 ‰, 0 % and 5 % under metastable assumption. The relatively high fraction in PD of Case I–II and low fraction in PD of Case III–V is consistent with the relatively high O₃ values observed in PD of Case I–II and low O₃ values observed in PD of Case III–V (Fig. 1c in the main text). Our $\Delta^{17}O$ -constrained calculation should be more reliable than purely assuming O₃ oxidation is negligible.

3. Q: Avoid claiming something like "the first observations of the oxygen-17 excess of..." in the manuscript. Many journals' instructions to authors specifically ask that you avoid using phrases like "we provide the first evidence" or "this is the first discovery". This is because it is more effective to

spell out how your work provides new knowledge and what important implications your discovery has. A: Thanks for the reminding. The word "first" has been removed from "the first observations of the oxygen-17 excess of..." and similar expressions throughout the manuscript.

4. Q: Mention the quantity (mg) of Ag₂SO₄ used for isotope measurement.

A: Thanks for the reminding. We have added the expression "The typical amount of O_2 for each run is 0.4–0.8 µmol." in line 118 in the method, which corresponds to 125–250 µg of Ag₂SO₄.

Reviewer 2 comments.

Q: I appreciate the authors' detailed response to my comments. This is a very interesting paper utilizing D17O measurements to understand multiphase chemistry in Beijing haze. The clarity of this paper is improved. I only have a minor suggestion. As for the role of heterogeneous sulfate production (the main topic of this paper), Table S7 is very clear. I suggest moving this table to the main text. I agree that heterogeneous oxidation dominated in cases I, IV, and V. However, in case III, fcloud (46%) is slightly greater than fhet (42%). Therefore, both production pathways were important in this episode. I suggest the authors to present this in a careful way throughout the manuscript (including abstract). I recommend this paper to be published in ACP as it is, or with minor modification as suggested above. **A:** Thanks for the comment. Table S7 has been moved to the main text as Table 2. We have changed the former expression "However, heterogeneous sulfate production (*P*,) on aerosols was estimated to

the former expression "However, heterogeneous sulfate production (P_{het}) on aerosols was estimated to dominate sulfate formation during PD of other cases, with a fractional contribution of (48±5) %." in the abstract into "During PD of Case I and III–V, heterogeneous sulfate production (P_{het}) was estimated to contribute 41–54 % to total sulfate formation with a mean of (48±5) %." in line 24-26. And we have changed the former expression "Heterogeneous reactions were found to dominate sulfate formation during PD in four out of the total five cases (except for Case II) with fractional contributions of 42 to 54 % and a mean of (48±5) % (Fig. 4)." in the discussion into "Heterogeneous reactions were found to contribute 41–54 % to total sulfate formation during PD of Case I and III–V, with a mean of (48±5) % (Fig. 4)." in lines 295-297.

Editor Comments:

Dear authors,

Many thanks for your revised submission. Please take note of the reviewers' comments and my

comments below when you prepare a revised manuscript.

A: Many thanks for your notice and comments. We prepare the revised manuscript following the reviewers' comments and your comments, and reply to these comments one by one.

Q: There are various problems with the dimensions of quantities in equations 1, 4, 5 and 8, and in case of equations 5 and 8, this may result in large changes to some of your results.

A: Thanks for your comment. We have corrected errors in these equations and errors in results from these equations. We reply to this comment in detail under your following specific comments.

Q: Section 2.7 does not state how the various fractions *f* have been calculated. Please give explicit equations that clarify this, including what the input terms (reaction rates, Δ (¹⁷O) values) are and how these input terms have been calculated themselves.

A: Thanks for your comment. We have added how the various fractions f was calculated in the present manuscript. It reads "By using Eq. (6) and the definition $f_{S(IV)+O3} + f_{S(IV)+H2O2} + f_{zero-d17O} = 1$, we have $f_{S(IV)+O3} = (d^{17}O_{obs}-0.7\%\times f_{S(IV)+H2O2})/6.5\%$ and $f_{zero-d17O} = (6.5\%-d^{17}O_{obs}-5.8\%\times f_{S(IV)+H2O2})/6.5\%$. Since $f_{S(IV)+O3}$, $f_{S(IV)+H2O2}$, and $f_{zero-d17O}$ should be in the range of 0 to 1 at the same time, $f_{S(IV)+H2O2}$ is further limited to meet $f_{S(IV)+H2O2} < \min\{d^{17}O_{obs}/0.7\%, (6.5\%-d^{17}O_{obs})/5.8\%\}$. Therefore, possible range of $f_{S(IV)+O3}$ and $f_{zero-d17O}$ can be obtained at different $f_{S(IV)+H2O2}$ assumptions." in lines 175-179 and " $f_p = c(p-SO_4^{2-})/c(SO_4^{2-})$, $f_{het} = \{P_{het}/(P_{het}+P_{cloud}+P_{SO2+OH})\} \times (1-f_p)$, $f_{cloud} =$ $\{P_{cloud}/(P_{het}+P_{cloud}+P_{SO2+OH})\} \times (1-f_p)$ and $f_{SO2+OH} = \{P_{SO2+OH}/(P_{het}+P_{cloud}+P_{SO2+OH})\} \times (1-f_p)$." in lines 184-186, where $c(p-SO_4^{2-})$ refers to the mass concentration of primary sulfate.

Q: The reviewers have also raised a number of points that need to be addressed. In particular, points 1 and 2 of reviewer 2 (source of the air; role of O_3 as oxidant) need some careful discussion. Table S7 should be moved to the main text.

A: Thanks for your reminding. We have replied to the reviewers' comments in the above section.

Q: There are still a few problems with missing units in equations – please refer to the first chapter of the IUPAC Green Book (https://www.iupac.org/fileadmin/user_upload/publications/e-resources/ONLINEIUPAC-GB3-2ndPrin

ting-Online-Sep2012.pdf) or chapter 5 of the SI brochure (https://www.bipm.org/en/publications/si-brochure/) for examples of correct quantity notation.

A: Thanks very much for recommending these books. We have corrected errors in the equations throughout the manuscript.

Q: The term "concentration" is not interchangeable with "mole fraction". Please use the term "mole fraction" where you refer to the latter (e.g. l. 197 and 198).

A: Thanks for your comment. Throughout the manuscript, we have changed "concentration" into "mole fraction" where it refers to the latter.

Q: Data availability: Please include a table with the data from Figures 1, 4, 5 and 6 and the individual input values used for each sample in the ISORROPIA model.

A: Thanks for your reminding. The data from Figures 1, 4, 5 and 6 and the individual values used for each sample in the ISORROPIA model are now available in the supplementary Excel file.

Q: 1. 67: A quantity symbol (e.g. *R*) must be used to define the isotope ratios and the index must follow immediately after the quantity symbol, e.g. $R_{sample}(^{x}O/^{16}O)$. "*X*" should be written in italics because it is a quantity symbol.

A: Thanks for your comment. We have changed the former expression "wherein $\delta^{X}O = ((^{X}O/^{16}O)_{sample}/(^{X}O/^{16}O)_{VSMOW} - 1)$ with X = 17 or 18 and VSMOW referring to Vienna Standard Mean Ocean Water" into "wherein $\delta = (R_{sample}/R_{reference} - 1)$ with *R* representing the isotope ratios of ¹⁷O/¹⁶O or ¹⁸O/¹⁶O in the sample and the reference Vienna Standard Mean Ocean Water, respectively" in lines 66-68 in the introduction.

Q: 1. 92: Please replace "ppb" with the corresponding SI unit "nmol mol⁻¹", throughout the manuscript. Atmospheric Chemistry and Physics requires the use of SI units. Also, please write the equation in line with the rules of quantity algebra, i.e. $[H_2O_2] / (nmol mol^{-1}) = 0.1155e^{0.0846T/C}$.

A: Thanks for your suggestion. We have replaced "ppb" with "nmol mol⁻¹" throughout the manuscript, and have rewritten the equation as you suggested in line 92.

Q: 1. 125/Eq. 1: Please use quantity algebra for all equations, see IUPAC Green Book

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(https://www.iupac.org/fileadmin/user_upload/publications/e-resources/ONLINEIUPAC-GB3-2ndPrin ting-Online-Sep2012.pdf). Where quantities are given as explicit values, they must carry units (e.g. 96 g mol⁻¹, 3600 s h⁻¹).

A: Thanks for your reminding. The quantities are with units now throughout the manuscript when they are given as explicit values.

Q: Eq. 1 is dimensionally not correct; it has units of g $m^{-3} h^{-1} atm^{-1}$, but is supposed to have g $m^{-3} h^{-1}$. Presumably the equation needs to include atmospheric pressure.

A: Thanks for your comment. Atmospheric pressure is included in equation 1 and 4 now. And we have corrected results from equation 1 and 4 throughout the manuscript. The following shows how we get equation 1 (similar for equation 4): The rate constant k (s^{-1}) for heterogeneous loss of SO₂ is determined by $k = (R_p/D_g+4/v\gamma)^{-1}S_p$ (Jacob, 2000), therefore, the heterogeneous sulfate production rate $P_{het} = k[SO_2]$ is in the unit of nmol (mol of air)⁻¹ s⁻¹ as the unit of SO₂ (nmol mol⁻¹) is indeed nmol (mol of air)⁻¹. 1 nmol SO₂ heterogeneous loss equals to 96 ng SO₄⁻² heterogeneous production, 1 s equals to 1/3600 h and 1 mol of air equals to the volume of $(1mol \times RT)/p$ by using the ideal-gas law, so 1 nmol (mol of air)⁻¹ s⁻¹ = 96 ng $((1mol \times RT)/p))^{-1}$ (1/3600 h)⁻¹. When *R* is 0.082 atm L K⁻¹ mol⁻¹, *p* is in unit of atm and *T* is in the unit of K,1 nmol (mol of air)⁻¹ s⁻¹ = 96 ng $(\frac{1mol \times RT}{p})^{-1}$ ($\frac{1}{3600}$ h)⁻¹ = $\frac{3600 \times 96 \times (p)}{0.082(T)}$ µg m⁻³ h⁻¹, where {*Q*} refers to the numerical value of a physical quantity *Q*. During our sampling period, the atmospheric pressure *p* ranged from 0.98 to 1.01 atm with a mean of (1.00±0.01) atm, so the corrected *P*_{het} and *P*_{SO2+OH} are both in the range of 98 % to 101 % of the former numerical values.

Q: 1. 127: The non-SI unit "atm" should be replaced with an SI-accepted unit, e.g. bar or Pa (or a derivative of them).

A: Thanks for your reminding. The non-SI unit "atm" have been replaced with an SI-accepted unit "Pa" in line 129.

Q: 1. 129: The uptake coefficient has the unit "1"; it is not "unitless".

A: Thanks for your reminding. The express "unitless" has been changed into the unit "1" in line 131.

Q: 1. 132: Again, units are missing from this equation. Also, the quantity that "PM_{2.5}" refers to must be identified, e.g. $\gamma(PM_{2.5})$ or $\rho(PM_{2.5})$, if it is a mass concentration. Both symbols are not ideal because they clash with the uptake coefficient and the bulk density. Perhaps the uptake coefficient should be given a different symbol than γ .

A: Thanks for your reminding. The former expression " $R_p = (0.254 \times PM_{2.5} + 10.259) \times 10^{-9}$ " has been changed into " $R_p/m = (0.254c(PM_{2.5})/(\mu g m^{-3}) + 10.259) \times 10^{-9}$ " in line 134, where $c(PM_{2.5})$ refers to $PM_{2.5}$ mass concentrations in the unit of $\mu g m^{-3}$.

Q: 1.136: The quantity that PM2.5 refers to must be identified, e.g. γ (PM2.5). The extraneous factor 10⁻⁶ and the multiplication symbols (×) should eliminated from the equation.

A: Thanks for your comment. The quantity " $PM_{2.5}$ " in equation 2 refers to $PM_{2.5}$ mass concentration, which has been replaced by " $c(PM_{2.5})$ " now.

Q: l. 137 & 205: Please choose a suitable single-letter symbol for relative humidity in these equations,
e.g. Ψ.

A: Thanks for your comment. The expression "RH" has been changed into " Ψ " in lines 140 and 212. And " Ψ " has been identified as "where Ψ refers to relative humidity with the unit of %." in line 141.

Q: 1. 152: see 1. 125: "3600 s h⁻¹", "96 g mol⁻¹"; correct dimensions (presumably multiplication by atmospheric pressure).

A: Thanks for your comment. Atmospheric pressure is included in equation 4 now. " 3600 s h^{-1} , 96 g mol⁻¹," are used to replace "3600" and "96" in line 157.

Q: 1. 160 & 246: These equations is wrong in a bad way. The units on the right hand side are " $g^2 m^{-3} h^{-1}$ ", but are supposed to be " $g m^{-3} h^{-1}$ ". Again, it should be "3600 s h⁻¹" and "96 g mol⁻¹". Finally, the SI requires quantity symbols to consist of a single (Latin or Greek) letter, so LWC is not an acceptable symbol in an equation and should be replaced by a suitable one. (LWC as an abbreviation is fine, just not as a quantity symbol). These errors suggest that *P*cloud values may be fundamentally wrong. Please discuss, using numerical examples, the impact of correcting the equation on your results.

A: Thanks for your comment. Equation 5 is derived from subsection 7.4 of Seinfeld and Pandis (2006)

at pp. 306, which reads:

The moles per liter of air can be then converted to equivalent SO_2 partial pressure for 1 atmosphere total pressure by applying the ideal-gas law to obtain

$$R_a'' = 3.6 \times 10^6 \, LRT \, R_a \quad (\text{ppb h}^{-1}) \tag{7.75}$$

where L is in g m⁻³, R = 0.082 atm L K⁻¹ mol⁻¹, and T is in K. For example, an aqueousphase reaction rate of 1 μ M s⁻¹ in a cloud with a liquid water content of 0.1 g m⁻³ at 288 K is equivalent to a gas-phase oxidation rate of 8.5 ppb h⁻¹. A nomogram relating aqueous-

It needs to be explained that R_a is the reaction rate in M s⁻¹, L is cloud liquid water content in g m⁻³ in the above equation 7.75. Since 1 ppb $h^{-1} = 1$ nmol (mol of air)⁻¹ $h^{-1} = 96$ ng $(\frac{1 \text{mol} \times RT}{p})^{-1} h^{-1} = 1$ 96 ng $\left(\frac{1 \mod \times 0.082 \operatorname{atm} L \operatorname{K}^{-1} \operatorname{mol}^{-1} \times \{T\} \operatorname{K}}{\{p\} \operatorname{atm}}\right)^{-1} \operatorname{h}^{-1} = \frac{96 \times \{p\}}{0.082 \{T\}} \mu g \operatorname{m}^{-3} \operatorname{h}^{-1}$, 8.5 ppb h⁻¹ (an approximate value of 8.502 ppb h^{-1}) in the above case equals to 34.56 μ g m⁻³ h^{-1} of sulfate production under 1 atmosphere total pressure. When we calculate the case (LWC = 0.1 g m⁻³ = 100 mg m⁻³, $R_{S(IV)+oxi} = 1 \mu M s^{-1} = 10^{-6}$ M s⁻¹) by using our equation 5, it turns out $P_{\text{cloud}} = 3600 \times 96 \times 100 \times 10^{-6} = 34.56$, which is the same as value calculated by above equation 7.75. In fact, the unit of right hand side of our equation 5 is truly " μ g m⁻³ h⁻¹" as 1 mg m⁻³ of LWC equals to 10⁻³ ml m⁻³ when ρ (H₂O) = 1g ml⁻¹ was used, which is also used by Seinfeld and Pandis (2006) to get the unit of ppb h^{-1} in the above equation 7.75. Here shows how we get the unit of P_{cloud} in equation 5 (similar for P_{het} in equation 18): (s h⁻¹)×(g mol⁻¹)×(mg m⁻¹) ³)×(M s⁻¹) = (s h⁻¹)×(g mol⁻¹)×(10⁻⁶ L m⁻³)×(mol L⁻¹ s⁻¹) = 10⁻⁶ g m⁻³ h⁻¹ = μ g m⁻³ h⁻¹. For your review, we shows how to obtain equation 5: $P_{\text{cloud}} = LWC \times R_{S(IV)+\text{oxi}} = \{LWC\}(\text{mg m}^{-3}) \times \{R_{S(IV)+\text{oxi}}\}(M \text{ s}^{-1}) = \{LWC\}(M \text{ s}^{-1})$ $\{LWC\} \times \{R_{S(IV)+oxi}\} (mg m^{-3}) \times (M s^{-1}) = \{LWC\} \times \{R_{S(IV)+oxi}\} (10^{-6} L m^{-3}) \times (mol L^{-1} s^{-1}) = 0$ refers to the numerical value of Q. In addition, we have changed "LWC" and "AWC" into " L_c " and " L_a " in equations 5 and 18 respectively, to meet the requirements of SI.

Q: 1. 167 & 1. 180: Include "%" after 6.5 and 0.7.

A: Thanks for your reminding. We have added "‰" after 6.5 and 0.7 in equations 6 and 8 in lines 172 and 189.

Q: 1. 196: This equation requires quantity symbols for the mole fractions and the unit "nmol mol^{-1} "

needs to appear in the right place, e.g. " $y(NH_3) = 0.34y(NOx) + 0.63 \text{ nmol mol}^{-1}$ ".

A: Thanks for your suggestion. We have changed "NH₃ (ppb) = $0.34 \times NO_X$ (ppb) + 0.63" into "[NH₃]/(nmol mol⁻¹) = $0.34[NO_X]/(nmol mol^{-1}) + 0.63$ " in line 203.

Q: 1. 197: Replace "concentration" with "mole fraction" – also other occurrences of the word "concentration" in the text may need to be replaced with "mole fraction". Concentration implies an amount per volume.

A: Thanks for your suggestion. Throughout the manuscript, we have changed "concentration" into "mole fraction" where it refers to the latter.

Q: 1. 205: "MF" should be replaced with a suitable single-letter symbol, e.g. "*x*(metastable)".

A: Thanks for your suggestion. "MF" has been replaced by "x(metastable)" in equation 9 in line 212 and identified as "where x(metastable) is the fraction of metastable aerosols to total aerosols in the unit of %." in line 213.

Q: 1. 211: The terms involving logarithms of concentrations and ion strengths in equations need to be divided by the standard concentration ($c^{\ominus} = 1 \mod \text{dm}^{-3}$), to make them dimensionally correct. The units of β^* need to be identified.

A: Thanks for your comment. The terms involving logarithms of concentrations and ion strengths in equations have been divided by " $c \ominus$ " in line 218-222. The unit of β^* is (mol L⁻¹)², which has been identified in line 225.

Q: 1. 258: Please use appropriate symbols, e. g $c(SO_4^{2-})$ for sulfate concentrations.

A: Thanks for your suggestion. c(X) has been used as the mass concentration of species X throughout the manuscript. The expression "SOR = $nSO_4^{2-}/(nSO_4^{2-}+nSO_2)$, where nSO_4^{2-} and nSO_2 represents the molar concentration of SO_4^{2-} and SO_2 , respectively" has been changed into "SOR, which equals to SO_4^{2-} molar concentration divided by the sum of SO_4^{2-} and SO_2 molar concentration" in line 264-265.

Q: p. 11: Remove unnecessary brackets around pH expressions, e. g. 7.6 ± 0.1 . The brackets are only required where similar such expressions have units.

A: Thanks for your reminding. Unnecessary brackets around pH expressions have been removed throughout the manuscript.

Q: Figure 2: In the figure caption, please include an explicit link to the newly added equations in the main text that give explicit solutions for the fractions shown in this figure.

A: Thanks for your suggestion. We have added the expression " $f_{S(IV)+H2O2}$ is in the range of 0 to $min\{\Delta^{17}O_{obs}/0.7\%, (6.5\%-\Delta^{17}O_{obs})/5.8\%\}, f_{S(IV)+O3} = (\Delta^{17}O_{obs}-0.7\%\times f_{S(IV)+H2O2})/6.5\%$ and $f_{zero-\Delta^{17}O} = (6.5\%-\Delta^{17}O_{obs}-5.8\%\times f_{S(IV)+H2O2})/6.5\%$. See equation 6 and its caption in Sect. 2.7 for details." in the end of Figure 2 caption in lines 578-580.

Q: Table S1: $k_{0\text{low}} = 3.3 \times 10^{-31} (T/300 \text{ K})^{-4.3} \text{ cm}^6 \text{ s}^{-1} [T \text{ has units of K; molecule is not a unit}]$ $k_{0\text{high}} = 1.6 \times 10^{-12} \text{ cm}^3 \text{ s}^{-1} [\text{since } (T/300 \text{ K})^0 = 1]$

A: Thanks for your suggestion. We have improved the expressions in Table S1 based on your suggestion.

Q: Table S4: Did you only use these mean values in your thermodynamic calculations? Or did you use sample-specific input parameters?

A: We used sample-specific input parameters. And these input has been presented in the supplementary Excel file.

References

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