A Novel Chamber Method for the Soil CO₂ Flux Measurement Based on the Diffusion Equation

拡散理論に基づく土壌 CO₂ フラックス測定のための新しいチェンバー法

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A new chamber technique, based on the diffusion equation, is devised to measure gas fluxes emitted from the soil. The top of the chamber is made of a porous material of known diffusivity. We determine the fluxes passing through the porous media by measuring the concentration difference between the inside and outside of the chamber. The proposed method, the diffusion system or the porous-top chamber method, is a steady state method and able to measure the CO₂ fluxes from soil cores in the laboratory within the precision of the CO₂ sensor. Our system may provide a simple and economical means for continuous surface flux measurements in the field.

Key words: Porous media, Porous-top chamber, Gas diffusion, Steady state method

土壌から発生するガスフラックスを拡散理論に基づいて測定する新しいタイプのチェンバー測定法（拡散型チェンバー法）を開発した。このチェンバーは、その天板の一部にガス拡散係数が既知の多孔質媒体で作られた拡散板がはめ込まれており、土壌からのガスフラックスはこの拡散板を拡散して大気中に放出される。このときチェンバー内外に生じるガス濃度差を測定することにより、フラックス密度を求める。本測定法は通気式チェンバー法と同様に定常法の一種で、動的平衡状態を達成することにより、連続測定が可能である。本方法の妥当性を確認するため室内実験を行い、土壌サンプルから放出される CO₂ フラックスを、使用した濃度センサーの精度に応じた正確さで測定できることを示した。したがって、本方法は、簡易で安価なガスフラックスの連続測定手法となりうることが期待される。

キーワード：拡散媒体、拡散型チェンバー、ガス拡散、定常法

I. INTRODUCTION

Chamber methods have been extensively used for several decades to measure gas fluxes emitted from land surfaces. The operating principle of chambers is to restrict the volume of air with which gas exchange occurs, thereby magnifying changes in the concentration of the emitted (or absorbed) gas in the headspace (Denmead, 1994, Livingston and Hutchinson, 1994). In closed chamber systems (the non-steady state system), there is no air replacement in the headspace, so the gas concentration changes continuously. In open systems (the flowthrough, steady state system), a constant airflow through the headspace is maintained, and the gas concentration attains a steady difference from the background concentration in the ambient air. These changes are measured to determine emitted (or absorbed) gas fluxes based on mass balance.

Chamber methods, as seen from the operating principle, do not require fast-response nor high-precision gas sensors (Denmead, 1993). Furthermore, the land area required for their deployment is small, typically less than 1m². They are therefore useful for
process studies and experiments that cannot be approached with micrometeorological methods. However, there are some difficulties with current chamber methods.

Open systems are mechanically more demanding because of the need to maintain the airflow through the chamber. Moreover, they can be subject to errors resulting from over-pressurization or under-pressurization of the chamber. Such a pressure change, in turn, induces a mass flow of the gases from or into the soil, leading to falsely high or low estimates of gas emission rate (Kanemasu et al., 1974, Davidson et al., 2002).

Closed systems are much simpler and less demanding, and therefore more commonly used. However, continuous measurements for more than an hour or so are generally impossible to make, because measures must be taken to prevent changes in the gas concentration within the chamber from influencing the flux. Recently, an automated flow-through, non-steady state system has been developed in which the chamber is closed periodically when measurements are taken (Drewitt et al., 2002). This system has the advantage of high measurement frequency without disturbing the surface being measured, and is suitable for long-term, unattended remote operation. However, it requires a sophisticated mechanical and electric system to control opening and closing the chamber lid.

The objective of this study is to develop a chamber method by which the trace gas fluxes from the land surface can be measured more easily and reliably than by current methods. The system we propose is the one that can be called the diffusion system or the porous-top chamber method. Unlike conventional chamber systems that are based on mass balance, our system employs the integrated diffusion equation (the transfer equation) to determine fluxes. Thus, we shall first examine if the CO₂ flux can be measured accurately by our proposed method. We shall then examine if our system is capable of providing continuous surface flux measurements of CO₂ evolved from the soil.

II. METHODS

1. Principle of the diffusion system

The steady-state gas flux density (F) can be expressed by the following transfer equation:

\[ F = - \frac{\nabla C}{R} \]  

(1)

where \( R \) is the diffusive resistance and \( \nabla C \) the concentration difference across the resistance. Thus, we can determine the flux density \( F \) by measuring when the value of \( R \) is known.

To apply the above principle, we have fitted the top of the chamber with a porous material whose diffusive resistance is predetermined. When placed on a gas emitting surface, the chamber allows for gas fluxes to continually diffuse out of the chamber through the top. This process is assumed to be governed by the following diffusion equation:

\[ F = -D \frac{dC}{dz} \]  

(2)

where \( F \) is the flux density crossing the porous layer, \( D \) the diffusivity and \( C \) the gas concentration of this layer, and \( z \) the distance from the base of the porous layer. We further assume that the porous layer is thin enough so as to assure the constant flux with vertical distance. Then, the integration of eq (2) , with the conditions \( C = C_c \) at \( z = 0 \) and \( C = C_a \) at \( z = L \), yields:

\[ F = (C_c - C_a) \frac{D}{L} \]  

(3)

where \( C_c \) is the gas concentration in the ambient air, and \( L \) the thickness of the porous layer. This is equivalent to eq (1) where \( L/D \) is expressed as \( R \).

The soil CO₂ profiles below and the concentration in the chamber at equilibrium (when the surface flux balances the rate of production) are different from those in the pre-deployment field. Thus, when the flux changes with time, there will be a period in which the CO₂ concentrations in the soil and the chamber air adjust to their new equilibrium values. Estimates of the steady flux during this time will be erroneous (Denmead, 1979, Livingston and Hutchinson, 1994).

To estimate the time for our diffusion system to reach steady state, we shall make an additional as-
sumption that chamber deployment does not affect the emission rate, that is, \( F_f \) is constant with time. Then, from the mass balance over the time period \( dt \), we obtain
\[
\frac{dC}{dt} = (AF_f - A_cF) \frac{1}{A_c} \exp \left( \frac{A_cD}{LV} \right)
\]
(4)
where \( V \) is the head-space volume, \( dC \) the concentration increment in the head space over \( dt \), and \( A \) the basal area of the chamber, \( A_c \) the area of the porous material, \( F \) and \( F_f \) the flux densities out of and into the head space. Solution of eq (4) subject to the conditions of eq (3) and \( C = C_a \) at \( t = 0 \) gives
\[
C - C_a = \frac{AF_f}{A_c} \frac{1}{A_c} \exp \left( \frac{A_cD}{LV} \right)
\]
(5)
Thus, the time constant \( \tau \) of this system, period for the chamber concentration to reach 63% of its final equilibrium value, is given as
\[
\tau = \frac{LV}{A_cD} = \frac{A}{A_c} \cdot \frac{L}{D} \cdot \frac{V}{A} = \frac{A}{A_c} \cdot R \cdot \frac{V}{A}
\]
(3)
The equilibrium time is proportional to the effective diffusive resistance \( (R \cdot A/A_c) \) and the characteristic height \( (V/A) \) of the chamber.

Gasses evolved in the soil are transferred to the atmosphere by molecular diffusion through the soil, and by eddy diffusion through the surface boundary layer. Since eddy diffusivities are usually several orders of magnitude greater than their molecular counterparts, surface gas fluxes are governed by the diffusive resistance of the soil layer. This situation is disturbed to some extent as we enclose the surface with the chamber. By enclosing the surface with our porous-top chamber, we introduce an additional process, that is, molecular diffusion through the chamber top. Thus, the magnitude of diffusivity of the chamber top material, relative to that of the soil, should be large enough so that the chamber exercises no appreciable influence on the surface fluxes.

2. Design of the diffusion chamber

The diffusion system consisted of a chamber and a means to detect the gas concentration inside the chamber. We designed two chambers of different configuration and diffusive resistance. The one (Chamber 1) was aimed for ultimate field use, whereas another (Chamber 2) was designed for laboratory test only. Chamber 1 was an open-ended plastic container (Fig. 1). Four holes, each 0.1m in diameter, were cut through the top-board, and each sealed with a disc of sintered sand plate (JACOM Co., “porous stone”). Chamber 2 was also an open-ended plastic container, a modified wagner pot of 1/500 are. We reduced the height of the pot and replaced its bottom with a layer of 0.4mm glass beads. The diffusivity of each porous material was determined by the method described elsewhere (Komimm and Takami, 1996) Pertinent specifics of the chambers are given in Table 1.

We used an electrode (Toa Denpa, model CE-331) to measure CO₂ concentration inside the chamber. The 30mm-diameter sensor head was inserted through the top of the chamber. This sensor was designed to measure CO₂ concentration of the still air. The time period for the sensor to reach 90% of its fi-

<table>
<thead>
<tr>
<th>Components</th>
<th>Chamber 1</th>
<th>Chamber 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area of the end-opening (A)</td>
<td>0.0625 m²</td>
<td>0.020 m²</td>
</tr>
<tr>
<td>Height</td>
<td>0.2 m</td>
<td>0.11 m</td>
</tr>
<tr>
<td>Area of the porous-material (A_c)</td>
<td>0.0314 m²</td>
<td>0.0193 m²</td>
</tr>
<tr>
<td>Thickness of the porous-material (L)</td>
<td>0.005 m</td>
<td>0.035 m</td>
</tr>
<tr>
<td>Diffusivity of the porous-material (D) at 0 °C</td>
<td>2.80×10⁻⁶ m²·s⁻¹</td>
<td>2.32×10⁻⁶ m²·s⁻¹</td>
</tr>
</tbody>
</table>

*0.25 m × 0.25 m, **0.16 m diameter, ***Four 0.1 m diameter discs
nal value was about 2 min (at 20°C) and its precision was 20 ppmv. The electrode gas meter requires 9 volt electric power to operate, either from batteries or from commercial power supply.

We had to make a compromise between three factors that were involved in choosing the diffusive resistance of the chamber. These were: the sensitivity of CO₂ concentration measurements, the need for appropriate equilibration and the effect of an additional resistance on the surface fluxes. Considering the precision of the sensor, we determined the diffusive resistance of the chamber top in such a way that the chamber concentration would reach several hundreds ppmv above the ambient level.

The effective diffusion resistance of the Chamber 1 \( R \cdot A/A_c \) thus determined was 34 s · cm⁻¹, 10 to 100 orders of magnitude smaller than that of the soil.

The time constant of this particular design, estimated from eq (8) with dimensions given in Table 1, was about 20 min. The diffusion resistance of Chamber 2 was 151 s · cm⁻¹ and the time constant 29 min, both being estimated as for Chamber 1.

III. Testing

We carried out two tests on our system. The first one was aimed to validate the measurement principle of the system, using Chamber 1 and known CO₂ fluxes from the point source. The second one was intended to see if the system is potentially applicable to the field, using Chamber 2 and one dimensional surface fluxes from the soil.

1. Test against known fluxes

We tested our method against the fluxes that were known and independent of chamber operation. The fluxes were the ones evolved from the soil cores and were determined by an open air flow system (Fig. 1, Fig. 2). The system consisted of a box-type chamber (0.28m × 0.54m × 0.32m) and a non-dispersive infrared gas analyzer (IRGA, Licor Model LI6260). Outside air was drawn continuously by a pump through the chamber and its CO₂ concentration \( C_o \) was measured as it left. A second air stream was drawn by another pump from the air outside the laboratory, and its CO₂ concentration \( C_a \) was measured by the same IRGA. We connected a flow meter to each airflow pass before the pump. A three-way solenoid valve, placed after each pump, regulated the airflow pass to IRGA. Outside air was sampled for the first 2 minutes, and inside air for the next 8 minutes, this cycle being repeated over the measurement period.

Fig. 1 A porous-top chamber and the apparatus for its testing. The porous-top chamber was enclosed in a larger chamber through which a constant air flow was maintained.

Fig. 2 The assembly to test the diffusion system. CO₂ concentrations, one in the air outside of the laboratory \( C_a \) and another through the open-chamber \( C_o \) were measured with an infrared gas analyzer (IRGA) to determine the flux from the soil sample \( S \). The air samples for these measurements were independently drawn, via flow meters (not shown in the figure) by pumps \( P \) and were regulated by the three-way solenoid valves \( SV \) and needle valves \( E \) before being fed into the analyzer. CO₂ concentration inside the diffusion chamber \( C_c \) was continuously monitored with an electrode gas sensor \( E \) to estimate the flux through the porous disc \( PD \).
Fig. 3  An example of IRGA outputs. Two solenoids were energized alternately as indicated by a solid line ( □ ) and the corresponding concentrations ( □ ) were measured for 2 min for the ambient air ( C_{a} ) and for 8 min for the air passing through the open-chamber ( C_{o} ). The IRGA outputs were logged every 10 sec on a data collector.

( Fig. 3 ) Three needle valves were each adjusted when the air flow pass to the analyzer was altered to keep a constant airflow rate ( 3330 mm^{3} \cdot s^{-1} )

We fabricated a test chamber ( Chamber 1 ) as described in the previous section, and enclosed it with the chamber of the airflow system ( Fig. 1, Fig. 2 ). CO_{2} sources, soil cores filled in open containers, were placed inside the test chamber. The CO_{2} concentration of the air inside this chamber ( C_{c} ) was measured, as described earlier. Such arrangements allowed the test chamber to measure the same fluxes as determined by the open air flow system.

We increased the number of soil containers from one to seven to vary the source intensity and thus the fluxes. For each set of soil cores, fluxes were measured continuously over four to five hours, during which the room temperature was left uncontrolled. Mass fluxes from the soil \( Q ( kg \cdot s^{-1} ) \) were determined by the following mass balance equation

\[
Q = ( C_{o} - C_{a} ) \cdot f
\]

where \( f ( m^{3} \cdot s^{-1} ) \) is the flow rate through the air circulation chamber. The fluxes measured by the diffusion method were obtained from

\[
Q = ( C_{o} - C_{a} ) \cdot \frac{A_{c} D}{L}
\]

where \( A_{c} \) is the area of the porous material ( m^{2} ), and \( L ( m ) \) the thickness and \( D ( m^{2} \cdot s^{-1} ) \) the diffusivity of the porous layer, respectively. Diffusivity \( D \)

was corrected for the temperature according to

\[
D = D_{0} \left( \frac{T}{273.15} \right)^{1.83} = 2.80 \times 10^{-1} \left( \frac{T}{273.15} \right)^{1.83}
\]

All the CO_{2} sensor outputs, volume fractions of CO_{2}, were converted to mass fraction according to the following standard procedure:

\[
CmgCO_{2} \cdot cm^{-3} = \frac{C_{ppm} \cdot \sqrt{273.15}}{T \cdot 1.2} \cdot \frac{P}{1013} \cdot 1.963
\]

We did not make barometric correction, assuming the atmospheric pressure ( P ) remained constant at 1013 hPa over the measurement period.

2. Comparison with the closed system

We compared the diffusion chamber system ( Chamber 2 ) with the closed chamber system in the laboratory. The closed system consisted of a cylindrical container made of plastic and the same electrode gas sensor as in the diffusion system. The diameter of the chamber was 16cm and its height 11cm. The CO_{2} source was a clay soil mixed with organic matter. The soil was filled in a pair of wagner pots, whose cross sectional area was 200cm^{2} and height 24cm. Each of the wagner pots was sealed tightly with each chamber, so that all the CO_{2} evolved from the soil was led into the chamber head space without any leakage. In other words, we intended to supply each chamber with one-dimensional ( vertical ) surface fluxes of equal magnitude. Two sets of CO_{2} source-chamber apparatus thus arranged were left in the laboratory side by side for 14 hours without temperature control.

We measured CO_{2} concentration continuously in both systems. The fluxes were continuously determined in the diffusion system as before. In the closed system, they were estimated from the concentration difference every half an hour, using the following mass balance equation:

\[
Q = V \cdot \frac{dC}{dt}
\]

where \( Q ( kg \cdot s^{-1} ) \) is the CO_{2} mass flux from the soil, \( V ( m^{3} ) \) is the volume of the chamber head space, and \( dC ( kg \cdot m^{-2} \cdot s^{-1} ) \) the concentration difference of CO_{2} over the time period \( dt ( s ) \). The same procedures were applied to conversion of volume concentration to mass fraction and temperature cor-
rection for the diffusivity as in the test of the system against known fluxes (III.1). We did not measure CO₂ concentration in the ambient air, assuming it to be 330 ppmv.

IV. RESULTS

1. Test against known fluxes

Fig. 4 shows some examples of measurements. The CO₂ concentration outside the laboratory (Cₐ) varied slightly, reflecting a natural diurnal variation. Concentration in the airflow chamber (Cₜ) was 50-100 ppmv above Cₐ and closely followed the change in Cₐ. Concentration in the test chamber (Cₚ) was 100-150 ppmv above Cₚ, and showed more variation than the other two concentrations. The fluxes measured by the diffusion system were generally in good agreement with the ones determined by the mass balance (the open air flow system), although the diffusion system slightly overestimated the fluxes in the test with 4 soil cores. As expected, measured fluxes generally followed the temperature increase in both tests.

Fig. 4 also shows the dynamic response of the flux measurement. Compared to the airflow system, the diffusion system's response to a sudden change was much slower; took about 40 minutes for it to reach a steady state. In other words, the measured time constant of the system was about 25 min, comparable to the one, 20 min, predicted from eq (6).

Fig. 5 summarizes the results of a series of such tests. Fluxes, measured continuously over 4-5 hours

\[
\begin{align*}
\text{Flux by mass balance (mgCO}_2\text{·hr}^{-1}) & \quad 0 & \quad 20 & \quad 40 & \quad 60 \\
\text{Measured flux by diffusion system (mgCO}_2\text{·hr}^{-1})
\end{align*}
\]

Fig. 5 Comparison of flux measured with the diffusion chamber against flux determined as the mass balance. The number of soil cores was changed to create different CO₂ source intensities.

\[
\begin{align*}
\text{Flux from the soil (mgCO}_2\text{·hr}^{-1}) & \quad 0 & \quad 20 & \quad 40 & \quad 60 \\
\text{Errors in the measured flux by diffusion system (mgCO}_2\text{·hr}^{-1})
\end{align*}
\]

Fig. 6 Dependence of the errors on the flux level in the measured fluxes with the diffusion chamber. The errors are the difference between the fluxes measured with the diffusion chamber and the fluxes determined as the mass balance.
Fig. 7 Comparison between diffusion system (Chamber 2) and closed system. The fluxes measured with the diffusion system (·) were plotted every 10 min. The fluxes measured with the closed system (□) were calculated from concentration difference every 30 min.

for each set of soil cores, were averaged, and plotted in this figure. For example, the third smallest data point was the average flux from 3 soil cores and the forth the one from 4 cores (Fig. 4). The diffusion system slightly overestimated the fluxes ranging between 2 and 55 mg CO₂·hr⁻¹ (corresponding to flux densities ranging between 32 and 880 mg CO₂·hr⁻¹·m⁻²). The errors, the differences between fluxes determined by the airflow system and measured by the diffusion system, were independent of the flux magnitudes (Fig. 6).

2. Comparison with the closed system

The CO₂ fluxes used in this comparison were 10 to 100 orders of magnitude greater than the naturally occurring fluxes in the field (Fig. 7). They varied in response to changes in the ambient temperature. The diffusion system underestimated the fluxes for the first 60 minutes, during which steady state did not occur. Thereafter, the two measurements were in close agreement for about 10 hours. The closed system, then, began to show lower fluxes relative to the diffusion system.

V. DISCUSSION

The diffusion system is based on two premises: the flux passing through the chamber occurs solely by molecular diffusion, and the steady state is established during the measurement period. The results from the tests showed these requirements were met under our test conditions. The system proved to measure precisely and accurately the CO₂ fluxes from the soil cores, under slowly varying temperature and nearly still air (air speed of about 0.4 mm·s⁻¹). The system was also capable of providing reliable surface flux measurements continuously in the laboratory where horizontal gas transport was prevented.

The errors of the diffusion system may have primarily resulted from the accuracy of the electrode CO₂ sensor. Fig. 6 suggests that the errors are independent of the magnitude of fluxes. In other words, they appear not systematic but random and thus are probably not inherent to the system. Moreover, the square mean of the errors in the measured fluxes was 1.52mgCO₂·hr⁻¹. Using this value in eq (8), we estimated the error in CO₂ concentration measurements at 12.3ppmv. The electrode sensor resolution, 10ppmv, accounts for most of this error, and the IRGA may have contributed the remaining. Bias toward the overestimation of the flux and thus of the concentration was probably due to the sensor drift during measurement.

In the field, chambers may be operated under largely different conditions from the ones we used in the laboratory tests. Furthermore, all the chamber systems inevitably affect the normal emission process occurring in the field. Thus, the ultimate evaluation of the method should be made in the field.

The first thing to be considered for field application is the effects of chamber gas concentration on the flux. That is, the feedback effects on the flux through the adjustment of concentration profile to the altered surface concentration. The effects can be categorized into two: one the effect with horizontal gas transport and the other the effect without it. The latter can be considered not to occur in our system, because it is a one-dimensional steady-state system if the horizontal concentration gradient in the soil below the chamber is negligible. In one-dimensional steady-state systems, a new concentration gradient is so established to match the pre-deployment flux, once the equilibrium is attained. In fact, close agreement between our system and the closed system pro-
vides evidence that our system operates under continuous dynamic equilibrium.

The horizontal gas transport may become important and cause a significant underestimate of the vertical flux if the chamber gas concentration increases too high to develop a significant horizontal concentration gradient. Thus, the effect with horizontal gas transport will be likely to occur in the present design of our system, since the equilibrium gas concentration in the headspace reaches around 200ppmv above the ambient air. However, such situation is not inherent to the system. The concentration difference between the headspace and the ambient air can be minimized by optimizing the diffusive resistance (R) of the porous chamber-top relative to the sensitivity of gas concentration measurements. We could easily accomplish this by using a more sensitive sensor. IRGA for instance, with appropriate in-line gas sampling arrangements as employed by Denmead (1979) in a closed chamber system.

Our preliminary field trial indicated that the system was capable of operating continuously for several days to measure the CO₂ fluxes of 360-1600mg CO₂·hr⁻¹·m⁻² emitted from a cultivated field (Kominami and Taniguchi, unpublished data). Hourly changes in the fluxes generally followed changes in the soil temperature, but such a diurnal pattern was perturbed when the wind speed was high.

The eddy diffusion and mass flow may come into play in the gas exchange process between the ambient air and the upper pores of the porous material when the wind strengthens. This will, in turn, cause the apparent diffusivity of the material to increase. In fact, our laboratory experiment (Kominami and Murakami, unpublished data) showed that the measured diffusivity of several porous materials increased with wind speed. The materials with higher permeability relative to diffusivity were more affected.

VI. CONCLUSIONS

Our proposed method is the first to apply the transfer equation to the chamber technique for gas flux measurements (Kominami and Takami, 1998, Japan Patent 2754161). In principle, it is analogous to electric current measurements with a circuit tester in that a known resistor is applied to the circuit and the voltage drop across the resistor is measured. We can also see the similarity between the proposed method and the heat flow plate which measures conductive heat flux through the soil and other solid materials.

This study has demonstrated that, in principle, CO₂ fluxes from the soil can be measured by the method we have proposed. Our laboratory test has validated the system under the situation where rate of the production and emission of gases is not affected by chamber operation. Furthermore, we have provided theoretical considerations to suggest that our system can be designed so as to minimize perturbation of surface fluxes occurring in the field.

Our system has several advantages over the conventional systems. Unlike the closed systems, it maintains the equilibrium concentrations of CO₂ in the headspace. Thus, it can minimize errors in flux estimates due to alteration of natural concentration gradient within the soil profile. Unlike the open systems, our system does not require a constant airflow through the chamber. It is therefore mechanically much simpler. Furthermore, errors in flux estimates arising from pressure change in the chamber can be avoided.

Preliminary field tests have indicated the effect of wind on flux estimates. At present, we are uncertain about the nature of this effect, although it may imply the possible capability of our system to accommodate the effect of wind and pressure on the process of surface-atmosphere gas exchange. Further insight into the problem will require detailed theoretical and experimental studies not only on the measurement method itself, but also the nature of gas exchange processes between the soil surface and the atmosphere.

More research is needed for our system to be established as a reliable means of accurately measuring soil respiration in the field. In addition to alteration of diffusion gradient, pressure artifacts and wind effects, addressed in this study, other influences of chamber placement on soil respiration,
such as alteration of soil moisture, should be examined. Meantime, we propose the present method as a possible means for simple and economical flux measurements where many simultaneous treatments are involved and commercial electric supplies are not available. Though the present system is the one for the CO$_2$ flux, the system is applicable for measuring of other trace gas fluxes as well.

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