Reports

Gas Bubbles in Fossil Amber as Possible Indicators of the Major Gas Composition of Ancient Air

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Gases trapped in Miocene to Upper Cretaceous amber were released by gently crushing the amber under vacuum and were analyzed by quadrupole mass spectrometry. After discounting the possibility that the major gases N_2, O_2, and CO_2 underwent appreciable diffusion and diagenetic exchange with their surroundings or reaction with the amber, it has been concluded that in primary bubbles (gas released during initial breakage) these gases represent mainly original ancient air modified by the aerobic respiration of microorganisms. Values of $N_2/(CO_2 + O_2)$ for each time period give consistent results despite varying O_2/CO_2 ratios that presumably were due to varying degrees of respiration. This allows calculation of original oxygen concentrations, which, on the basis of these preliminary results, appear to have changed from greater than 30 percent O_2 during one part of the Late Cretaceous (between 75 and 95 million years ago) to 21 percent during the Eocene-Oligocene and for present-day samples, with possibly lower values during the Oligocene–Early Miocene. Variable O_2 levels over time in general confirm theoretical isotope-mass balance calculations and suggest that the atmosphere has evolved over Phanerozoic time.

AMBER IS ANCIENT TREE RESIN THAT has hardened and been preserved in sedimentary rocks; its age ranges from Carboniferous (~300 million years ago) to Pleistocene-Recent (*1*). Besides bearing paleobiological information in its chemical structure and in its embalmed insects and spores (*1*), amber commonly contains gas bubble inclusions. From preliminary data it has been suggested (*2–4*) that these bubbles may represent mainly ancient air trapped at the time the original resin issued forth from its host tree. We present data that support this hypothesis and suggest that the major gas composition of air (N_2/O_2 ratio) has changed appreciably during the past 90 million years.

Amber samples studied include: (i) Dominican amber (from the Dominican Republic) of probable late Oligocene–to–Early Miocene age (*5*), (ii) Baltic amber of probable Oligocene-Eocene age (*1, 6*), and (iii) Upper Cretaceous amber from Cedar Lake, Manitoba, whose age (*6, 7*) is estimated to be "Senonian" (75 to 95 million years ago). Hardened modern pine tree resin from New Zealand (*Agathis australis*) was also studied as a check on our method. The Dominican amber consisted of centimeter-sized chunks containing millimeter-sized bubbles (Dominican 1 and 3), and a 4- to 5-cm diameter, transparent, gem-quality sample (Dominican 2) with a few large millimeter-sized bubbles trapped deeply within the clear amber (*8*). The Baltic material and New Zealand resin consisted of centimeter-sized chunks with bubbles readily visible under a binocular microscope. The Upper Cretaceous amber consisted of small pieces of clear amber, 2 to 4 mm in size, which in thin section were found to contain small bubbles ranging in size from about 10 to 100 μm.

Analysis of the amber was done by crushing it under vacuum and analyzing the released gases by time-resolved quadrupole mass spectrometry. The detection limit was about 10 ppm and the accuracy was about 3 to 5% of the amount of each gas present (*4, 9*). We used two crushing methods. The first method, applied to Dominican samples 1 and 2 only, consisted of placing bubble-rich chunks (with diameters of 1.5 to 2 cm) in stainless steel tubes that were attached to the mass spectrometer and evacuated to less than 10^{-6} torr. The tubes were sealed, removed, and the contained amber broken to mainly sand-sized fragments by gently crushing the tube in a hydraulic press. The released gases in the still-evacuated tubes were then transferred to the mass spectrometer for analysis. Because of the large bubble size, this crushing technique resulted in the release of much larger quantities of gas (~10^{-7} mole/g) as compared to the second method. The second method, applied to all other samples, involved successive, but very gentle, crushes in a small, evacuated (10^{-9} torr initial pressure), piston-cylinder crushing chamber that was attached at all times to the mass spectrometer. By slowly turning a crank, the degree of breaking and gas release could be controlled and small amounts of gas, on the order of 10^{-10} to 10^{-12} moles (except when unusually large bubbles were intersected), were sampled after each crush. Successive crank turnings eventually released more tightly held gases and allowed separation of gases from different sites within the amber.

All of the amber samples were examined in polished section to verify the presence of suitable gas inclusions. Gas is present in amber as "primary" bubbles entrapped between and within successive flows of tree resin, as irregular cavities in dessication cracks and secondary fractures, and as matrix gas. The primary bubbles are entrapped by resin flow over irregularities, by the struggling motions of insects trying to free themselves from the sticky resin, or by other means. Matrix gas refers to gas dissolved in the amber or dispersed as dense clusters of tiny submicroscopic bubbles (milky amber). This gas we believe arose from the uptake of tree-processed gases while the original resin resided within the tree. To avoid problems of interpretation, in this study we excluded from consideration both milky amber (*3*)

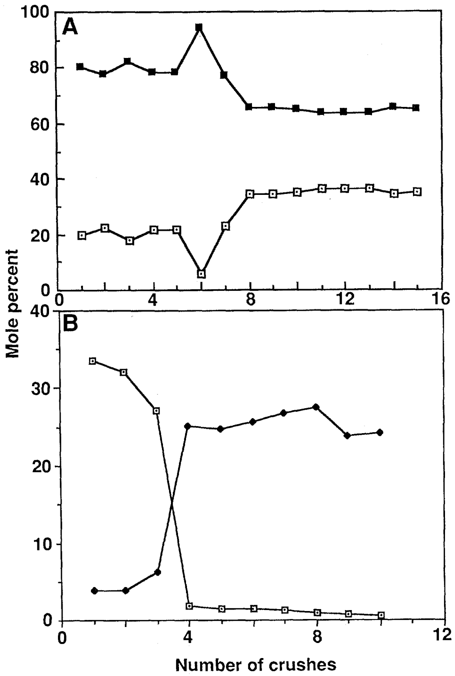

Fig. 1. Gas release from sequential gentle crushes of Baltic amber and Cretaceous (Cedar Lake) amber containing several microscopically observed primary gas bubbles. Percentages are on a water-free, molar basis and are based on the sum of $N_2 + O_2 + CO_2$. The amount of gas released per crush is on the order of 10^{-10} to 10^{-12} moles. The later, uniform composition, data we interpret as representing matrix gas derived from the tree. (**A**) Baltic amber, crush sequence I (see Table 3); (■), N_2; (□), $O_2 + CO_2$; (**B**) Cretaceous (Cedar Lake) amber, crush sequence III (see Table 2); (◆), CO_2; (□), O_2.

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and samples that showed appreciable fracturing.

We believe that primary bubbles represent mainly entrapped ancient air (which has been subjected to microbial respiration—see below). Gas from these bubbles, along with variable but usually minor amounts of H_2 and CH_4 probably from small cracks, was released easily as initial breakage occurred during the first few sequential crushes of an analysis. Subsequent crushes released mixtures of primary bubbles with (more tightly held) matrix gas. Eventually only matrix gas was released. The data for our Baltic amber analyses (Fig. 1A) illustrate these points (see also Fig. 1B for Cretaceous amber). In this example, we believe primary ancient atmospheric gas samples were obtained from the first seven crushes (excluding crush number 6), whereas for crushes 9 through 15 only matrix gas with a different composition was released. Horibe and Craig (*10*) report gas data obtained by pulverizing Baltic amber in a ball mill and analyzing large amounts of released gases (orders of magnitude more gas than in our work). Thus they most likely were unable to distinguish the small amount of gas in primary bubbles from the much more abundant (and masking) matrix gas. In fact, our results for later sequential crushes of Baltic amber (Fig. 1A) indicate a matrix gas composition essentially identical to their reported results. We also find, as they did, little or no O_2 in the matrix gas of all of our samples (for example, Fig. 1B). Furthermore, sequential analysis of our samples indicates that the major gas composition of matrix gas is highly variable from one amber sample to another as a result of complex life processes operating on the original resin. For example, results for Upper Cretaceous amber from Cliffwood, New Jersey, show a uniform matrix gas of nearly 98% N_2 (on a $N_2 + O_2 + CO_2$ basis) compared with 65% N_2 for the matrix gas in Baltic amber.

In two analyses (Dominican 1 and 2) we used cryogenic techniques to separate water and to better resolve the other gases (*4*). We also used cryogenic separation (at −105°C) during the analysis of an extra sample of modern resin to reduce interferences from abundant organic gases on the mass 40 peak for Ar and thus obtain N_2/Ar ratios. All of the samples contained appreciable water, ranging from a few percent up to 95% of total gases, but in no case was there enough water present that dissolved gases contributed more than a negligible proportion to the total gas analyzed. There was little difference for the gases of major interest (N_2, O_2, and CO_2) between results obtained with or without cryogenic separation (compare results for Dominican 1 with Dominican 3 in Table 1).

Table 1. Mean concentrations of "primary bubble" gas from amber compared to modern air and sediment gas. Values on a mole percent water-free basis; *n* = number of individual-crush gas samples or individual analyses for glacial drift and marine sediment. Analyses for glacial drift and marine sediment from (*11*) and (*12*), respectively. Small concentrations of hydrocarbons other than methane were also present in all of the ancient amber samples.

Gas	Modern air	Modern resin*	Dominican amber			Baltic amber	Cretaceous (Cedar Lake) amber	Glacial drift	Marine sediment
			1†	2†	3				
N_2	78.1	77.8	79.0	84.3	80.4	50.4	59.6	12	14
O_2	20.9	4.6	3.5	0.8	0.4	6.5	23.0	~0	0
CO_2	<0.1	16.7	12.5	12.1	11.8	7.4	6.8	2	<55
Ar	0.9	0.9	1.4	1.9	3.7	1.5	1.5	0.2	0.4
CH_4	<<0.1		0	0	1.4	11.9‡	4.0	86	>30
H_2	<<0.1		2.8	0.8	1.9	20.0‡	5.1		<<0.1
n		4	1	1	28	10	9	5	32

*Values represent percentage of sum of ($N_2 + O_2 + CO_2$ + Ar) only (Ar/N_2 via separate analysis using cryogenic separation); the gas samples actually average ~70% hydrocarbons. †Data from (*4*). ‡Averages for CH_4 and H_2 include two unusually high results for CH_4 (28 and 42%) and for H_2 (69 and 80%). Without these the remaining samples average 5.9% CH_4 and 6.4% H_2.

Table 2. Individual gas analyses of primary bubbles in Cretaceous (Cedar Lake) amber. The symbols I, II, and III refer to separate amber samples; letters to separate crushes of each sample. Values represent mole percent on a water-free basis.

Gas	Sample								
	IA	IB	IIA	IIB	IIC	IID	IIIA	IIIB	IIIC
N_2	63.4	65.0	64.4	63.3	54.8	64.5	60.8	49.1	51.0
O_2	25.3	22.2	17.8	19.1	27.5	17.2	32.6	24.8	20.8
CO_2	8.2	8.3	10.5	9.3	4.8	8.3	3.7	3.0	4.7
Ar	1.9	1.8	1.7	1.7	1.7	1.8	0.9	1.0	1.3
CH_4	0.4	0.7	4.4	4.6	8.5	4.6	1.9	4.4	6.1
H_2	0.9	2.1	1.1	2.0	2.7	3.4	0	17.7	16.1
$\frac{N_2}{O_2 + CO_2}$	1.9	2.1	2.3	2.2	1.7	2.5	1.7	1.8	2.0

Results of gas analysis ("primary bubbles" only), reported on a water-free basis, are shown in Tables 1, 2, and 3. Also shown for comparison are the compositions of modern air, gas in glacial drift (*11*), and dissolved gas in shallowly buried near-shore marine sediments (*12*). The composition of easily released gas bubbles from amber resembles air much more than it resembles sediment gas. The principal gas in the amber is N_2 and not CH_4, which is the dominant gas in marine and nonmarine sediments. Also, sediments at depth contain no O_2, whereas the amber samples exhibit various concentrations of O_2. Thus, bubbles in amber have not simply equilibrated with gases in the sediments in which they have been buried for millions of years.

There are also several lines of evidence that the amber gases have not exchanged with the modern atmosphere. They are:

1) The Dominican 2 sample contained sufficiently large bubbles that their total volume could be measured under a microscope. From this volume and the total amount of gas released during analysis, the average pressure in the bubbles was calculated to be about ten times the value for the modern atmosphere. This excess pressure was probably caused by compression of the amber during burial in the sediments. (Finding of strain halos around bubbles in other amber samples suggests that excess pressure is a common phenomenon.) Such retention of excess pressure militates against equilibration with modern air.

2) The O_2/N_2 ratios for several of the Cretaceous samples are greater than the present-day value and cannot simply represent gas contamination by modern air.

3) Extensive pumping of all samples down to pressures less than 10^{-6} torr in the tube crush experiments, and less than 10^{-9} torr in the successive crush experiments, was done to remove all traces of modern air prior to crushing.

4) The CO_2/O_2 ratio and H_2 concentration of gas samples from successive crushing of, for example, the Cretaceous amber (Table 2 and Fig. 1B) shows point-to-point variability that would not be expected if the material had undergone diffusion and consistent exchange with modern air.

5) The presence of H_2 in all of the samples, probably derived from organic reac-

Table 3. Individual major gas analyses (mole percent) of primary bubbles (from initial crushes) in modern resin and in Baltic amber. Percentages based on sum of $N_2 + O_2 + CO_2$. Roman numerals refer to separate amber samples; letters to separate crushes of each sample.

Sample	Gas			
	N_2	O_2	CO_2	$\frac{N_2}{O_2 + CO_2}$
		Modern resin		
IA	78.8	1.2	20.0	3.7
IB	76.1	2.7	21.1	3.2
IC	79.3	1.3	19.4	3.8
IIA	79.7	13.1	7.2	3.9
		Baltic amber		
IA	80.1	6.5	13.3	4.0
IB	77.8	5.1	17.1	3.5
IC	82.3	4.0	13.7	4.6
ID	78.6	0.3	21.1	3.7
IE	78.4	0.4	21.2	3.6
IF	76.8	0.9	22.3	3.3
IIA	78.4	18.2	3.3	3.6
IIB	77.3	19.8	2.9	3.4
IIC	79.9	17.1	3.0	4.0
IID	78.1	15.0	6.9	3.6

tions in the amber, indicates inhibited diffusive loss of this gas. Hydrogen would be expected to be the most diffusible gas in the amber and the most likely one to be lost to the air.

6) Finally, there is the simple geological argument that the amber was exposed during burial to sediment gas for much longer times than it has been exposed to the modern atmosphere. Since the amber has neither lost N_2 to the sediment gas nor picked up appreciable CH_4 over millions of years of burial (the small and variable amount of CH_4 found in most samples we believe is due mainly to organic reactions within the amber), it is unlikely that exchange with modern air gases has occurred during the much shorter time since it was eroded or removed from the host sediment.

Based on these considerations we conclude that the easily released gas from primary bubbles in our amber samples represents mainly ancient air trapped at the time the original resin was exuded from the host tree. However, there is still a major problem in that O_2 levels in our samples are highly variable and CO_2 concentrations are too high to represent original atmospheric levels. Values of CO_2 in the atmosphere greater than ~0.6% would result in impossibly high greenhouse temperatures (*13*). We originally thought (*4*) that the low values of O_2 and high values of CO_2 in the Dominican 1 and 2 samples represented reaction between original-air-entrapped O_2 and the amber. However, the high O_2 values in the Cretaceous amber and low values in modern resin have caused us to abandon this hypothesis. [Also, O_2 reaction with amber should not result in CO_2 production (*14*).] If O_2 readily reacts with amber (*14*) over geological time, why should the oldest samples we have studied have the highest O_2 contents and why do so many samples, even after several crushes, exhibit measurable O_2?

A better interpretation of the O_2 and CO_2 results is that the gas samples represent a mixture of original pure air and original respired air in varying proportions. As an air bubble is originally trapped, it could include microorganisms within it which respire some of the O_2 within the bubble before dying. In carbohydrate metabolism, for every mole of O_2 consumed, ~1 mole of CO_2 would be produced. If this approach is correct, then the original air concentration of O_2 can be calculated from the sum of the measured concentrations of O_2 and CO_2. Application of this idea to four analyses of modern resin results in a calculated original concentration of O_2 of 21%, which agrees well with the composition of modern air (Tables 1 and 3). This provides a check on the respired-air hypothesis and also provides evidence against other respiratory reactions with different stoichiometries, such as succinate metabolism. More importantly, calculated values of the ratio $N_2/(O_2 + CO_2)$ for each of the amber samples are relatively constant, despite highly variable O_2/CO_2 ratios (see Tables 2 and 3).

We have entertained the possibility that the O_2 measured in "primary bubbles" represents contaminant air that has diffused from the modern atmosphere into more accessible (crushable) sites in the amber which, nevertheless, do not give up this gas during initial vacuum pumping before crushing. However, this hypothesis does not explain: (i) summing of CO_2 plus O_2 concentrations to a constant value for a variety of different gas and amber samples from the same locality, (ii) the lack of a simple mixing trend between matrix gas and modern air, and (iii) higher concentrations of O_2 in Cretaceous amber than that found in other age ambers or in the modern atmosphere. (These results also do not agree with the idea of consistent differential diffusion of modern air gases into the amber samples.) Because of this we feel that our explanation as original entrapped air, although more audacious, constitutes a better explanation of the data.

If these preliminary findings are correct, then we suggest that the atmospheric concentration of O_2 sometime during the Senonian (Late Cretaceous) was appreciably higher than today, in excess of 30%. [Preliminary data for amber from another location also suggests higher O_2 levels at this time (*15*).] Because the residence time of N_2 in the atmosphere is on the scale of hundreds of millions of years, compared with about 7 million years for O_2 (*16, 17*), the lower N_2/O_2 ratio at this time was probably due to higher absolute O_2 levels and not lower N_2 levels (*18*). (There is no obvious reason why total atmospheric pressure had to have been held exactly at its present value over geologic time.) This conclusion is in qualitative agreement with calculations based on carbon and sulfur isotopic measurements (*17, 19*), especially those of Arthur *et al.* (*19*), that suggest that atmospheric O_2 levels at this time were elevated because of increased worldwide burial rates of both organic matter and reduced sulfur.

Our data for Baltic amber indicate similar O_2 concentrations to today during the Eocene-Oligocene, whereas the Dominican amber data suggests a lower O_2 level during the Oligocene–Early Miocene. However, because of consistently low measured O_2 concentrations and a strong similarity between the composition of early and late crushes in the Dominican 3 sample (n = 28 in Table 1), it is possible that the Dominican amber has undergone some diagenetic exchange between primary bubbles and matrix gas. This means that the sum of O_2 plus CO_2 in Table 1 probably represents minimum values.

Much more data on amber, including better stratigraphic (time) resolution, needs to be obtained to check these preliminary data and to extend the measurements to older samples. Stable isotope analyses on amber gases need to be performed, which we so far have been unable to do with our technique because of contributions to the peaks for rarer isotopes from the multitude of minor organic gases sampled by the quadrupole method. There is also a problem with argon. Although the N_2/Ar ratios of primary bubbles in modern resin exhibit modern air–like values (N_2/Ar of the bubbles ~80), for primary bubbles in amber the N_2/Ar ratios are highly variable and always lower (N_2/Ar values range from 15 to 70). The average N_2/Ar ratio for our amber matrix gas is approximately 30, in general agreement with Horibe and Craig (*10*), but it suggests to us control by differential gas solubility in the original resin, rather than in water, as they state. Also, for some primary bubbles, excess Ar can be explained by radiogenic decay of ^{40}K to ^{40}Ar, at our measured levels in the amber of total potassium (1.1% by weight in a sample of Dominican 1), if the potassium is heterogeneously distributed. However, for Dominican 1 and 2 there is too much excess Ar to be explained simply by ^{40}K decay (*4*); perhaps here diagenetic exchange between primary bubbles and matrix gas has occurred, as discussed above.

In conclusion, we believe that atmospheric evolution, and its possible effect on organic evolution (*20*), is an integral part of Phanerozoic earth history, and that gas bubbles in fossil amber, with much more work, may provide an indication of ancient atmospheric composition.

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The 1987 Whittier Narrows Earthquake in the Los Angeles Metropolitan Area, California

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The Whittier Narrows earthquake sequence (local magnitude, $M_L = 5.9$), which caused over $358-million damage, indicates that assessments of earthquake hazards in the Los Angeles metropolitan area may be underestimated. The sequence ruptured a previously unidentified thrust fault that may be part of a large system of thrust faults that extends across the entire east-west length of the northern margin of the Los Angeles basin. Peak horizontal accelerations from the main shock, which were measured at ground level and in structures, were as high as 0.6*g* (where *g* is the acceleration of gravity at sea level) within 50 kilometers of the epicenter. The distribution of the modified Mercalli intensity VII reflects a broad north-south elongated zone of damage that is approximately centered on the main shock epicenter.

THE MODERATE-SIZED ($M_L = 5.9$) Whittier Narrows main shock, which occurred in the east Los Angeles metropolitan area at 7:42 (PDT) on 1 October 1987, caused three direct fatalities and substantial damage in many communities in Los Angeles and Orange counties (*1*). The main shock was located at 34°3.0'N, 118°4.8'W, 3 km north of the Whittier Narrows (*2, 3*) and at the northwestern end of the Puente Hills (Fig. 1). The depth of focus of the main shock is estimated to have been 14 ± 1 km. The epicenters of the aftershocks form an approximately circular pattern that is centered at the epicenter of the main shock and has a diameter of 4 to 5 km (Fig. 2); the hypocenters define a surface that dips gently to the north. The spatial extent of the aftershock zone is smaller than for most $M_L = 5.9$ main shocks. By comparison, the 1986 North Palm Springs earthquake ($M_L = 5.6$) had an aftershock zone of 9 km by 16 km (*4*). The focal mechanism of the main shock, which was derived from first motion polarities, has two nodal planes, that is, possible fault planes, that strike east-west and dip 25° to the north and 65° to the south. The maximum compressive stress is estimated to trend 163° (south-southeast) and plunge 6° (*5*). The spatial distribution of the hypocenters of the main shock and aftershocks as well as the focal mechanism of the main shock indicates that the causative fault is a gently north-dipping thrust fault with an east-west strike that is located at depths from 11 to 16 km. No surface rupture for the Whittier Narrows earthquake has been documented (*6*).

The Whittier Narrows earthquake was located near the subsurface intersection of the west-northwest striking Whittier fault and the east-west striking thrust faults of the southern margin of the Transverse Ranges. Where it is best exposed in the Puente Hills, the Whittier fault is dominantly a northeast-dipping strike-slip fault with a small reverse component. The Whittier Narrows main shock was not caused by slip on the Whittier fault because it ruptured a gently dipping thrust fault with an east-west strike. The seismogenic potential of such thrust faults has long been recognized farther to the north along the exposed frontal fault system of the Transverse Ranges. For instance, the 1971 ($M_L = 6.4$) San Fernando earthquake occurred along the frontal fault system (*7*).

The spatial distribution of hypocenters of the main shock and aftershocks indicates that the Whittier Narrows earthquake sequence ruptured a thrust fault beneath the uplifted Elysian Park–Montebello Hills and Puente Hills. These uplifts are surface expressions of anticlines that are part of a large and mostly buried antiform (an anticline in which the stratigraphy is uncertain).

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