Reports

Carbon, Carbides, and Methane in an Apollo 12 Sample

Abstract. *Total carbon in the Apollo 12 sample 12023 fines was 110 micrograms per gram of sample with a carbon isotopic abundance $\delta^{13}C$ (relative to the Pee Dee belemnite standard) of +12 per mil. Hydrolysis of the fines with deuterium chloride yielded undeuterated methane along with deuterated hydrocarbons, thus confirming the presence of 7 to 21 micrograms of carbon per gram of sample as carbide and about 2 micrograms of carbon per gram of sample as indigenous methane. After vacuum pyrolysis of the fines to 1100°C the following gases were detected in the relative abundance: carbon monoxide $\gg$ carbon dioxide $>$ methane. Variations of the $\delta^{13}C$ value with the pyrolysis temperature indicated the presence of carbon with more than one range of isotopic values. The observed $\delta^{13}C$ value of +14 per mil for lunar carbide is much higher than that of carbide in meteorites. These results suggest that lunar carbide is either indigenous to the moon or a meteoritic contribution that has been highly fractionated isotopically.*

Guided by the Apollo 11 results (*1–6*) and the similarity in carbon content of Apollo 11 and 12 samples (*7*), we investigated the amount, nature, and isotopic composition of carbon in a sample of Apollo 12 fines (sample 12023) (*8*). The results of acid hydrolysis (*9*), vacuum crushing, and vacuum pyrolysis experiments indicate that this sample contains indigenous carbide and methane in amounts comparable to those found in Apollo 11 fines (*2, 10*). The bulk of the carbon does not appear to be present as carbonyls, carbonates, or organic carbon, but rather seems to be in elemental form or in the form of unhydrolyzable carbides, or both; upon vacuum pyrolysis to 1100°C most of the carbon is converted in the mineral matrix to CO and CO_2 with the former predominating.

We determined the values given in Table 1 for total carbon concentration and carbon isotopic composition in 12023 fines by following procedures used previously (*4*). The first sample listed seems high in carbon concentration and light in isotopic composition, probably because of slight contamination. The second and third determinations are believed to be representative of the concentration and isotopic composition of carbon in sample 12023 (*11*).

By means of procedures described earlier (*2*), the 12023 fines were treated with 6*N* HCl and the gaseous products were collected. High-resolution mass spectrometric analysis showed the presence of C_1 to C_4 hydrocarbons, but no CO or CO_2 (detection limit, approximately 4 nanomoles of a gas). Gas chromatography revealed amounts of C_1 to C_4 hydrocarbons corresponding to 16.8 μg of carbon per gram of sample (Table 2, experiment 1), values similar to those obtained for Apollo 11 sample 10086 (*2*). In an experiment modeled after that of Abell *et al.* (*10*) we confirmed the presence of indigenous carbide in Apollo 12 sample 12023 by heating the sample with 6*N* DCl in D_2O, which resulted in the release of deuterated hydrocarbons. Although 69 percent of the methane could be accounted for as CHD_3 and CD_4, 31 percent was obtained as CH_4 (Table 2, experiment 2) (*12*).

Since CH_4 apparently was released by partial acid destruction of the lunar mineral matrix, an alternative means (*10*) of breaking up the matrix was examined. A sample of 12023 fines in a Pyrex ball mill equipped with Lucalox (General Electric) balls was evacuated at 150°C and 10^{-4} torr for 2 hours, then pulverized for 8 hours. In another experiment a second sample of 12023 fines was mixed with 1 ml of D_2O (99.8 percent D) in the ball mill (to exchange hydroxyl hydrogen in the sample) and then evaporated to dryness at 10^{-4} torr and 150°C. The procedure (*10*) was repeated twice. Undeuterated methane was identified in both crushing experiments; however, 36 percent of the methane from the D_2O-exchanged sample was released as CHD_3 and CD_4 (Table 2, experiments 3 and 4). In a separate experiment 25 mg of iron carbide (Fe_3C) was mixed with a 1.06-g sample from a batch of basalt, which previously had yielded no CH_4, CO, or CO_2 when crushed, and was pulverized in the ball mill. Traces of methane were produced (*13*). Carbides in the samples can be hydrolyzed during crushing experiments, possibly by evolved water, as indicated by the formation of CHD_3 and CD_4 from the D_2O-exchanged samples. Similarly, most of the hydrocarbons generated during acid treatment of sample 12023 fines are produced by carbide hydrolysis (*14*). Significantly, however, some CH_4 was released in the crushing and hydrolysis experiments involving D_2O and DCl, respectively. The unlabeled methane appears to be indigenous to Apollo 12 fines, and its concentration is estimated to be about 2 μg per gram of sample (*15*). Similar results were reported for Apollo 11 samples (*2, 10*).

Table 1. Amount and isotopic composition (relative to the Pee Dee belemnite standard) of carbon in samples of Apollo 12 (12023) fines.

Total carbon (μg of carbon per gram of fines)	$\delta^{13}C$ (per mil)
140	+3.4
112	+11.3
109	+12.4

Since lunar breccia presumably is derived from the fines, it was of interest to examine a sample of breccia for carbides and indigenous hydrocarbons. The crushing of Apollo 11 breccia sample 10009 was accomplished in two stages (combined in Table 2 as experiment 5). The first stage (8 hours) yielded C_1, C_2, and C_3 hydrocarbons, but the second stage (8 hours) yielded only methane (160 nanomoles per gram of sample) and, notably, CO_2 (11 nanomoles per gram of sample). Subsequent treatment of the pulverized breccia sample with 6*N* DCl resulted in the production of primarily deuterated hydrocarbons arising from carbide hydrolysis, but no additional CO_2. However, 31 percent of the evolved methane was undeuterated and, therefore, this methane is regarded as indigenous to the sample (Table 2, experiment 6) (*16*). It is not clear whether the trace amount of CO_2 released during the crushing of the breccia is indigenous

Table 2. Hydrocarbons and deuterated hydrocarbons produced during HCl or DCl treatment and vacuum crushing of Apollo 12 fines and Apollo 11 breccia; 0, not detectable; –, present but not measured.

Experiment	Sample*	Amounts of gases released (nanomoles per gram of sample)								
		CH_4	CHD_3	CD_4	C_2H_6	C_2D_6	Other undeuterated C_2 to C_4 species	Other deuterated C_2 to C_4 species	Indigenous CH_4 (μg per gram of carbon)	Carbide (μg per gram of carbon)
1. 6*N* HCl	12023 fines	978	0	0	29	0	153 (C_2, C_3, C_4)	0	3.7†	13.1‡
2. 6*N* DCl	12023 fines	133	102	187	0	51	0	–	1.6	6.7‡
3. Crushing in vacuum	12023 fines	40	0	0	0	0	0	0	0.48	
4. Crushing in vacuum after D_2O exchange	12023 fines	18	8	2	0	0	0	0	0.22	
5. Crushing in vacuum	10009 breccia§	214	0	0	–	0	–	0	2.6	
6. 6*N* DCl after crushing in vacuum	10009 breccia	336	301	401	0	185	0	–	4.0	18.1‡

* Sample sizes for experiments 1 through 5, respectively, (in grams) are as follows: 0.973, 1.251, 2.081, 2.087, and 1.801. † Calculated under the assumption that the amount of indigenous methane relative to that produced by carbide hydrolysis is the same as that in experiment 2. ‡ Calculated from the total amount of hydrocarbon or deuterated hydrocarbon, as determined by gas chromatography, minus the amount of indigenous methane. § Carbon dioxide, 11 nanomoles per gram of sample, was also detected.

lunar gas or contamination. The fact that CO_2 emerged only after extensive crushing supports the former view, whereas its absence after hydrolysis (*15*) suggests the latter.

The procedure used in pyrolysis experiments was as follows: (i) samples were initially outgassed at 150°C and 10^{-4} torr for 8 to 10 hours; (ii) then samples were pyrolyzed under vacuum in stepwise fashion at 500°, 750°, and 1100°C; (iii) then the sample was combusted at 1100°C in oxygen. At each pyrolysis stage about half of the evolved gases were converted to CO_2 for determination of the total carbon concentration and carbon isotope composition, and the remaining portion was analyzed by mass spectroscopy. The results of three pyrolysis experiments are summarized in Table 3. Included are data obtained from the pyrolysis of iron carbide, a sample of which was intimately mixed with a previously pyrolyzed (to 1100°C) basalt sample. The results obtained in trials 2 and 3 were quite similar, but the results of trial 1 differ because a faulty thermocouple introduced uncertainties into the determination of the actual temperatures. Generally, however, pyrolysis at 500°C yielded gases in the following order of abundance: $CO_2 > CO > CH_4$. Methane, the only hydrocarbon detected, was obtained in amounts that could be accounted for as indigenous gas. At temperatures above 750°C, CH_4 no longer appeared and CO greatly predominated over CO_2.

Data from the pyrolysis of iron carbide indicate that 95 percent of the carbide is converted to CO and CO_2 at temperatures from 500° to 1100°C. Thus, in lunar samples, although a fraction of the gases can be attributed to oxidation of hydrolyzable carbides, most of the CO and some of the CO_2 evolved at 750° and 1100°C must be derived from some other source or sources. The absence of comparable quantities of organic pyrolysis fragments and the lack of detectable CO or CO_2 in hydrolysis experiments is not consistent with the presence of organic compounds, carbonyls, and carbonates. The products, however, are consistent with the conversion of elemental carbon, in whatever form, or unhydrolyzable carbides, or both, to CO and CO_2 by interaction with minerals in the lunar sample (*2, 6, 7*). Thus indigenous carbon in this sample of Apollo 12 fines appears to occur as methane (2 to 4 μg per gram of sample), hydrolyzable carbides (7 to 13 μg per gram of sample), and possibly elemental carbon or unhydrolyzable carbides, or both (93 to 101 μg per gram of sample) (*17*).

Consistently low $\delta^{13}C$ values at 500°C probably reflect the presence of isotopically light indigenous CH_4 (*18*). The possibility of contamination in the form of strongly adsorbed terrestrial CO_2 cannot be eliminated as a source of isotopically light carbon. At 750°C

Table 3. Carbon-containing gases produced during the stepwise pyrolysis of Apollo 12 fines: relative abundance, total carbon, and carbon isotopic composition; –, not measured; 0, not detectable.

Temperature (°C)	Gases released (mole %)			Total C (μg per gram of sample)	$\delta^{13}C$ (per mill)
	CO	CO_2	CH_4		
			Trial 1		
500	37	46	18	25	−10.0
750	>98	<0.08	<0.7	49	+13.6
1100	94	6	0	61	+12.6
1100(O_2)*				–	–
			Trial 2		
500	15	68	17	16	−17.7
750	>98	<1.1	<0.4	27	+22.0
1100	86	14	0	73	+8.0
1100(O_2)*				20	+7.0
			Trial 3		
500	32	38	30	16	−16.5
750	95	4	1	24	+26.4
1100	93	7	0	69	+7.4
1100(O_2)*				20	−7.0
			Iron carbide		
500	–	–	–	5†	–
750	73	27	0	18†	−26.2
1100	70	30	0	77†	−19.6
1100(O_2)*				0	

* Sample combusted in oxygen at 1100°C. † Percentage of total carbon originally in Fe_3C sample.

the isotope value of the gases increased sharply, then decreased at 1100°C. The data clearly indicate that the CO evolved at 750°C differs isotopically from the CO evolved at 1100°C. Although $\delta^{13}C$ values of products obtained at the mid-temperature range could be interpreted as the result of isotopic fractionation at lower temperature, involving preferential production and evolution of compounds containing ^{12}C, the dramatic decrease in the $\delta^{13}C$ range at the highest temperature is not in agreement with such a possibility and presents strong evidence for the presence in lunar fines of carbon with more than one range of isotopic values (*4, 19*). Combustion at 1100°C of the material remaining yielded 20 μg of carbon per gram of sample (trials 2 and 3), but the $\delta^{13}C$ value of this unpyrolyzed carbon was +7 per mil in trial 2 and −7 per mil in trial 3. The significance of the combustion results is not clear, although sample heterogeneity may exist.

Carbon on the lunar surface probably exists as a mixture of native material, solar wind particles, and unvolatilized meteorite debris (*4, 6, 20, 21*). The carbide in lunar samples has been generally attributed to a meteoritic origin (*2, 3, 22*). Since the carbon isotopic composition of meteoritic carbides has been measured, it appeared that examination of the $\delta^{13}C$ value for lunar carbide could resolve the question of its possible meteoritic origin. Such a value could not be measured directly, but could be inferred from the isotopic composition of hydrocarbons produced during the acid treatment of lunar material.

In two experiments hydrocarbons released by HCl treatment were collected as described earlier (*2*), then converted to CO_2 for isotopic determination. The gases amounted to 21 μg per gram of sample with $\delta^{13}C = +14$ per mil in one case and 26 μg per gram of sample with $\delta^{13}C = +5$ per mil in the second. The latter sample appears to be high in carbon content and is isotopically light (*23*). Therefore, the isotope value of +14 per mil is thought to be representative of the carbon isotopic composition of the hydrocarbon gases evolved from sample 12023. Experiments with Fe_3C indicated that hydrocarbons produced by hydrolysis were depleted rather than enriched in ^{13}C. Thus, the $\delta^{13}C$ value of +14 per mil obtained for the hydrocarbons derived from sample 12023 apparently represents a minimum value. Although the $\delta^{13}C$ value of the indigenous methane is not known, it is most probably less than zero (*18*), and the small amount of indigenous gas can make only a minor isotopic contribution to the $\delta^{13}C$ value of the total gas mixture.

The most significant fact that emerges from the carbon isotope data is that hydrolyzable carbide in Apollo 12 sample 12023 is much heavier, isotopically, than any known carbide phase in meteorites; isotope values for meteoritic carbide fall in the range −4 to −8 per mil (*24*). In contrast, hydrolyzable carbides in 12023 fines have a minimum isotope value of +5 per mil and a most probable isotope value of +14 per mil or slightly higher. The large difference between isotope values for lunar and meteoritic carbide strongly suggests that the former was either native to the moon or deposited by meteorites and subsequently subjected to isotope fractionation processes on the lunar surface.

A solar wind "hydrogen-stripping" mechanism for ^{13}C enrichment in the carbon on the lunar surface has been proposed (*4, 21*) to account for the unusually heavy carbon in Apollo 11 samples. After long exposure on the lunar surface, meteoritic carbide with an initial isotope value of −4 to −8 per mil could be sufficiently depleted in ^{12}C to attain a value of +5 to +14 per mil. The action of the solar wind may also provide an explanation for the occurrence of carbon with several isotope ranges. Bond formation between solar wind carbon and many other elements on the lunar surface and subsequent "hydrogen-stripping" could be accompanied by isotope effects, thus leaving assemblages of carbon atoms in the mineral matrix having different degrees of enrichment in ^{13}C. These differences become manifest during pyrolytic oxidation as variations in the isotopic composition of CO at several temperatures (see Table 3), as well as possible isotopic variations between CO and CO_2 at the same temperature (*18*).

If meteorites are not the source of lunar carbide, then we are left with the possibilities that the carbides represent primordial indigenous lunar carbon, however formed, or the product of interaction between solar wind carbon and metals or metal oxides or minerals on the lunar surface. Less ambiguous information regarding primordial lunar carbide (and carbon) could be obtained by careful examination of the interiors of lunar crystalline rocks where exposure to solar and cosmic radiation and intermixing with lunar fines and breccia is minimized. In contrast to values obtained from fines, carbon isotope values from Apollo 11 rocks ranged from −20 to −28 per mil, but there was some ambiguity regarding contamination (*4*). Evidence also exists for the presence of carbides in Apollo 11 rocks (*2*) but the amounts were too low for isotopic measurements. If the carbon was indeed indigenous to the lunar rocks, the observed range of isotopic composition would be more representative of primordial lunar carbon.

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7. Lunar Sample Preliminary Examination Team, *Science* **167**, 1325 (1970).
8. Sample 12023, a fine-grained, brownish-black powder, was obtained from the Sea of Storms at a location about 440 m from the Apollo 12 lunar module and 500 m from the site of Surveyor 3 (*7*). The fines were taken from a trench 20 cm deep and packaged in the lunar environment sample container. Because of its fine grain size, the sample was not pulverized but rather was analyzed as received.
9. In preliminary experiments, ammonia in HCl hydrolyzates was detected and measured by the phenol-hypochlorite method [L. Solorzano, *Limnol. Oceanogr.* **14**, 799 (1969)]; the amount of ammonia detected was about 40 μg per gram of sample for 12023 fines (two experiments) and 63 μg per gram of sample for 10009 breccia. No ammonia was detected in solvent blanks or in a blank obtained by the acid hydrolysis of a basalt sample (all basalt samples used in this work were collected freshly crystallized in February 1970, from the Aloi crater, Hawaii). Identification of NH_3 was confirmed by ion-exchange chromatography by means of an automatic amino

acid analyzer. Possibly ammonia was indigenous and freed by partial acid breakdown of the mineral matrix, or it may have been a product of the hydrolysis of lunar nitrides, or a combination of both.

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11. The carbon isotope abundance is defined as follows (std refers to the Pee Dee belemnite standard):

$$\delta^{13}\mathrm{C} = \left[\frac{(^{13}\mathrm{C}/^{12}\mathrm{C})\mathrm{sample} - (^{13}\mathrm{C}/^{12}\mathrm{C})\mathrm{std}}{(^{13}\mathrm{C}/^{12}\mathrm{C})\mathrm{std}}\right] \times 1000$$

The carbon concentration falls within the range of values determined for six different samples of Apollo 12 fines, that is, 23 to 200 μg per gram of sample (*7*), but is less than the two unpublished results of 135 and 166 μg per gram of sample determined by C. B. Moore, Arizona State University, on a portion of sample 12023. Our results indicate that sample 12023 contains slightly less carbon than Apollo 11 sample 10086 [157 μg per gram of sample, $\delta^{13}C = +18$ per mil (*2*)], and that the carbon is isotopically lighter.

12. It is not clear why hydrocarbon yields differ in experiments 1 and 2; sample inhomogeneity may be a factor.
13. When fused silica or a sample of basalt was crushed in the ball mill, neither hydrocarbons nor CO and CO_2 were released in detectable amounts.
14. Hydrolysis of Fe_3C and meteoritic cohenite, $(Fe,Ni,CO)_3C$, produces similar mixtures of hydrocarbons (*2*).
15. Crushing under vacuum appears to be a relatively inefficient means of releasing the gases, which may be trapped as crystalline inclusions or in glassy spherules [see E. Roedder and P. W. Weiblen, *Science* **167**, 641 (1970)] or between inorganic crystal boundaries. Treatment with hydrochloric acid, on the other hand, appears to be very effective because digestion of the residue from HCl treatment with 48 percent HF yielded no additional hydrocarbons, CO, or CO_2.
16. Hydrolysis of carbides can account for the evolution of C_2 and C_3 hydrocarbons during the first crushing stage. Failure to detect hydrocarbons other than CH_4 during the second crushing stage and subsequent DCl treatment supports the view that the C_2 and C_3 hydrocarbons produced during the initial crushing were not indigenous to the breccia (*15*).
17. Because the total carbon balance (129 to 136 μg per gram of sample) was always high (by about 20 μg per gram of sample) and the isotope balance was low (+6 to +9 per mil), it is likely that contamination at each stage of pyrolysis occurred at concentrations (up to 4 μg per gram of sample) below our manometric detection limit.
18. From data obtained in three pyrolysis experiments, it is possible to formulate and solve three simultaneous equations for the unknown $\delta^{13}C$ values of CO, CO_2, and CH_4 evolved at 500°C. The calculated values are: CO, +22 per mil; CO_2, −18 per mil; and CH_4, −56 per mil. Similar calculations based on only the more reliable data from trials 2 and 3 indicate that, at 1100°C, $\delta^{13}C$ values for CO and CO_2 are +6 and +19 per mil, respectively. All of these values, however, should be viewed as qualitative because they arise from small isotopic and compositional variations between experiments which may well represent the uncertainty or reproducibility in the analytical methods.
19. S. Epstein and H. P. Taylor, Jr., *Science* **167**, 533 (1970).

→ P. I. Abell, G. H. Draffan, G. Eglinton, J. M. Hayes. J. R. Maxwell, C. T. Pillinger, *ibid.*, p. 7→ C. B. Moore, C. F. Lewis, E. K. Gibson, W. Nichiporuk, *ibid.*, p. 495; evidence of solar wind implantation of noble gases in Apollo 12 samples has been reported (*7*), but the amounts were two to five times less than those observed in Apollo 11 samples.

21. R. Berger, *Nature* **226**, 738 (1970).
22. A. T. Anderson, Jr., A. V. Crewe, J. R. Goldsmith, P. B. Moore, R. C. Newton, E. J. Olsen, J. V. Smith, P. T. Wyllie, *Science* **167**, 587 (197→ C. Frondel, C. Klein, Jr., J. Ito, J. C. Drake, *ibid.*, p. 681.
23. In the second experiment the CO_2 obtained after combustion of the hydrocarbons was impure and had to be purified by a second passage through the combustion train. This additional handling is believed to have introduced some contamination. Terrestrial contamination amounting to 5 to 6 μg of carbon per gram of sample with a $\delta^{13}C$ value of −30 per mil [see J. W. Smith and I. R. Kaplan, *Science* **167**, 1367 (1970)] could account for the apparently low value of +5 per mil.
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25. We thank Mrs. C. Petrowski, E. Ruth, and R. Presley of the University of California, Los Angeles, and M. Romiez of the Ames Research Center for valuable assistance.

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Endogenetic Craters Interpreted from Crater Counts on the Inner Wall of Copernicus

Abstract. *Collapse craters formed in terrestrial basalt flows exhibit size-frequency distributions that are similar to distributions for craters located in terraces in the inner wall of the lunar crater Copernicus. These distributions and surface morphology suggest that the interior terraces are basalt lava flows containing collapse craters as well as impact craters.*

Lunar crater counts have been employed previously in interpretations of lunar surface processes and geology (*1–3*). Most applications are based on the assumption (probably correct) that the vast majority of lunar craters are of impact origin. In some applications (*1, 3*), determination of crater size-frequency distributions for apparently different lunar surfaces permits relative age determinations for those surfaces. In essence, old surfaces have more and larger craters than young surfaces. However, the presence of lunar lavas, confirmed by Apollo samples (*4*), lends support to the possible existence of endogenetic craters. On the basis of crater counts, lunar surfaces with both endogenetic and exogenetic craters would erroneously appear to be "old." Results of this study indicate, however, that crater size-frequency distributions provide a means for recognizing and evaluating the presence of anomalous (endogenetic) craters.

Several types of endogenetic craters may be present on the moon. Collapse craters were proposed by Kuiper *et al.* (*5*) to be present in significant numbers on some mare surfaces. As terrestrial analogs, he cited examples of collapse craters formed in young basalt flows near Grants, New Mexico. Selection of basalt collapse craters as analogs to lunar structures is reasonable in view of the basaltic composition of mare rocks.

Volcanic collapse craters are produced by two main mechanisms: drainage of surface debris into subsurface

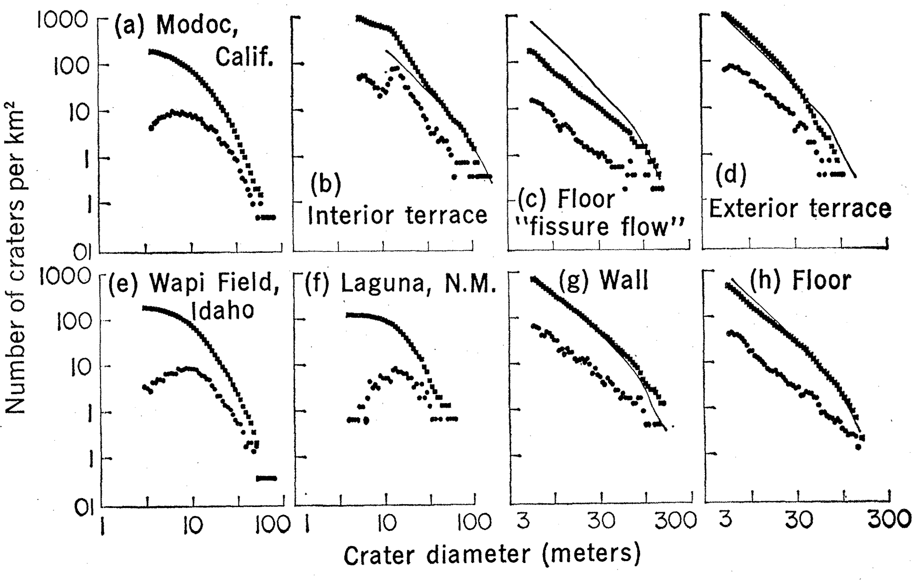

Fig. 1. Crater size-frequency distributions for terrestrial basaltic collapse craters (a, e, f) and five "formations" (b, c, d, g, h) associated with the lunar crater Copernicus. The datum for the lunar counts is a line representing the cumulative crater count for the continuous ejecta blanket from Copernicus. ●, Average number of craters per square kilometer; ■, cumulative number of craters per square kilometer.