

**COMPOUNDS OF THE ORGANOGENIC ELEMENTS
IN APOLLO 11 AND 12 LUNAR SAMPLES :
A REVIEW**

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Abstract. Investigations of low molecular weight compounds of the organogenic elements on lunar samples are reviewed. The three general techniques of vacuum pyrolysis, acid hydrolysis, and crushing have been employed by most investigators. Vacuum pyrolysis of lunar fines produce a variety of gaseous species which are either: (1) indigenous, (2) solar wind products and/or (3) chemical reaction products of mineral phases found in the lunar samples. Acid hydrolysis of lunar fines using deuterium-labeled acids yields evidence for indigenous methane and ethane. Methane and ethane found in the lunar fines are largely derived from the solar wind with only trace amounts indigenous to the samples. Crushing experiments with lunar fines and breccias produce methane, ethane, hydrogen, nitrogen, hydrogen sulfide and the rare gases.

1. Introduction

Investigations of low molecular weight compounds in lunar samples have been undertaken by a number of investigators. Three general techniques have been employed to release these compounds containing the organogenic elements for analysis. They are vacuum pyrolysis, acid hydrolysis, and crushing. Most of the analyses were made on dark mature lunar fines with a total carbon content in the range of 100 to 300 $\mu\text{gC/g}$ (Moore *et al.*, 1970, 1971; Epstein and Taylor, 1970; Kaplan *et al.*, 1970). Individual investigators used gas chromatograph and/or mass spectrometric techniques for their investigations. A qualitative list of species detected by the three techniques for the Apollo 11 and 12 lunar fines is given in Tables I–IV. It is important to note that the absence of a particular species in the analysis by an individual investigator may mean that he did not look for it rather than it was not present.

A. PYROLYSIS

The majority of the investigators performed vacuum pyrolysis on the lunar samples. Carbon dioxide was reported in each investigation where pyrolysis of the Apollo 11 and 12 fines were carried out (Tables I and II) and is released over the temperature range from 100°C to 1400°C (Oró *et al.*, 1970, 1971; Gibson and Johnson, 1971). There appear to be two distinct temperature ranges in which carbon dioxide is released from the lunar fines. A low temperature release of CO₂ occurs below 600°

to 700°C (Oró *et al.*, 1971; Gibson and Johnson, 1971). Oró *et al.* (1971) believe that the low temperature CO₂ is of terrestrial origin. Chang *et al.* (1971), Kaplan *et al.* (1970, 1971), and Oró *et al.* (1970) have noted differences in the isotopic ratios for the fractions of CO₂. It appears that the low temperature CO₂ may be either from contamination and/or be solar wind derived.

The release of carbon monoxide upon pyrolysis of the lunar fines is reported by a majority of the investigators (Tables I and II). The carbon monoxide is released over a wide temperature but the majority of the CO is released above 700°C. The carbon monoxide observed during the pyrolysis appears to be a reaction product of carbon containing phases (e.g. carbides) with the metal oxides and silicates found in the lunar fines (Gibson and Johnson, 1971; Oró *et al.*, 1971; Chang *et al.*, 1971). The carbon monoxide temperature release pattern observed for the lunar fines from both Apollo 11 and 12 has been duplicated by pyrolysis of synthetic lunar analogs to which have been added small amounts of meteoritic carbides (cohenite) (Gibson and Johnson, 1971).

Vacuum pyrolysis of lunar fines has shown that trace amounts of methane are produced (Abell *et al.*, 1970a, b, 1971; Chang *et al.*, 1970, 1971; Nagy *et al.*, 1970, 1971; Oró *et al.*, 1970, 1971; Funkhouser *et al.*, 1971). The concentration levels of the methane observed are generally below 6 ppm CH₄ (Abell *et al.*, 1970b; Chang *et al.*, 1971). The methane observed in the Apollo 11 and 12 lunar fines has several origins: (1) indigenous to the samples, (2) solar wind derived and/or (3) solar wind alteration of indigenous carbon to produce methane (Abell *et al.*, 1970b). Methane abundances may be directly correlated with other chemical abundances or physical properties associated with the solar wind (Abell *et al.*, 1971). In lunar fines from core tubes, those layers which have longer exposure to the solar wind are enriched in methane as compared to layers with shorter exposure periods (Abell *et al.*, 1971). Thus, it appears that the majority of the methane observed in the lunar fines can be accounted for as having a solar wind origin with only trace amounts of CH₄ indigenous to the samples (Abell *et al.*, 1971).

In addition to methane, ethane has also been observed from pyrolysis of lunar fines (Abell *et al.*, 1970a, b, 1971; R. C. Murphy *et al.*, 1970; S. Murphy *et al.*, 1970; Nagy *et al.*, 1970, 1971; Funkhouser *et al.*, 1971; Oró *et al.*, 1971). The concentration levels of ethane are generally below 0.1 ppm C₂H₆ (Abell *et al.*, 1970b, 1971). Additional hydrocarbons have been reported in trace amounts and it is questionable as to whether they are indigenous to the sample or result from terrestrial contamination (R. C. Murphy *et al.*, 1970; Henderson *et al.*, 1971; Nagy *et al.*, 1970). R. C. Murphy *et al.* (1970) and Preti *et al.* (1971) report an extensive list of trace hydrocarbon compounds observed during their high resolution mass spectrometric pyrolysis of lunar fines. They reported that although a number of organic hydrocarbon compounds or compound types have been detected, few appear to be definitely indigenous to the lunar samples.

Pyrolysis of lunar fines at temperatures below 500°C generally produces gases with carbon containing species in the following order of abundance: CO₂ > CO > CH₄.

TABLE I
 Pyrolysis of Apollo 11 lunar fines - X = species detected

| Investigator Species Sample No. | Abell 10086 | Burlingame 10086 | Chang 10086 | Epstein 10084 | Friedman 10084 | Funkhouser 10084 | Gibson 10086 | Kaplan 10086 | Lipsky 10086 | R. Murphy 10086 | Nagy 10086 | Oró 01086 |
|---------------------------------------|----------------|---------------------|----------------|------------------|-------------------|---------------------|-----------------|-----------------|-----------------|-----------------------|---------------|--------------|
| H ₂ | X | | | X | X | X | X | | | | X | X |
| CH ₄ | X | X | | | | | | X | | | X | |
| NH ₃ | X | | | | | | | | X | | | |
| H ₂ O | | X | | X | | | X | | | | | X |
| C ₂ H ₂ | | X | | | X | | X | X | | | | X |
| CO | X | X | | X | | | X | | | | | X |
| N ₂ | X | | | | | | X | | | | | X |
| CO-N ₂ | | | | | | | X | | | | | X |
| C ₃ H ₄ | X | | | | X | | | | X | | X | |
| HCN | X | X | | | | | | | | | | |
| C ₂ H ₆ | X | | | | X | | | | X | | X | |
| O ₂ | X | | | | | | X | | | | | |
| H ₂ S | | X | | | | | X | | X | | X | |
| C ₃ H ₈ | | X | | | | | | | X | | X | |
| CO ₂ | X | | X | | X | | X | X | | X | X | |
| COS | | | | | | | | | | X | | |
| SO ₂ | | | | | X | | | | X | X | | X |
| CS ₂ | | | | | | | | | X | X | | |
| Hydrocarbon > C ₃ | X | | X | | | | | | X | X | | |

TABLE II
 Pyrolysis of Apollo 12 lunar fines -- X = species detected

| Investigator Species Sample No. | Burlingame 12025 12028 | Chang 12023 | Freidman 12030 | Funkhouser 12070 | Gibson 12023 | Henderson 12023 12028 | Kaplan 12032 | Lipsky 12001 | Nagy 12023 | Oró 12023 | Preti 12028 |
|---------------------------------------|------------------------------|----------------|-------------------|---------------------|-----------------|-----------------------------|-----------------|-----------------|---------------|--------------|----------------|
| H ₂ | | | X | X | X | | | | X | X | |
| CH ₄ | X | X | | X | | X | | | X | X | X |
| NH ₃ | | | | | | | | | | X | X |
| H ₂ O | | | X | | X | | | X | | | |
| C ₂ H ₂ | | | | | X | | | | | X | X |
| CO | X | X | X | X | X | X | X | | X | X | X |
| N ₂ | | | | X | X | | | | X | X | |
| CO-N ₂ | | | | X | X | | | | X | X | |
| C ₂ H ₄ | | | | X | | | | X | | X | |
| HCN | | | | | | | | | X | X | X |
| C ₂ H ₆ | | | | X | | | | | X | X | |
| O ₂ | | | | | | | | | | | |
| H ₂ S | | | | | X | X | | | | | X |
| C ₃ H ₈ | | | | | | | | | | X | X |
| CO ₂ | X | X | X | X | X | X | X | | X | X | X |
| COS | | | | | | | | | | | |
| SO ₂ | X | | | | X | | | | | | X |
| CS ₂ | | | | | | X | | | | | X |
| Hydrocarbons > C ₃ | X | | | | | X | | | | | X |

Pyrolysis at temperatures above 750°C results in carbon monoxide abundances three to five times greater than CO₂ (Chang *et al.*, 1971; Gibson and Johnson, 1971; Oró *et al.*, 1971). For the most part, methane was not detected at temperatures above 750°C. The gas release data along with chemical abundance information on the carbon species reflects differences in binding energies and nature of the carbon containing species in the lunar fines.

Molecular nitrogen has been observed during pyrolysis of lunar fines by various investigators (Oró *et al.*, 1970; Nagy *et al.*, 1970, 1971; Gibson and Johnson, 1971; Burlingame *et al.*, 1970; Abell *et al.*, 1970a; Lipsky *et al.*, 1970). The concentration of nitrogen in the lunar fines has been reported by Moore *et al.* (1970, 1971) to range from 80 to 150 µgN/g. However, there is evidence that some of the nitrogen measured may be from atmospheric contamination and the true nitrogen abundances may be lower. Gibson and Johnson (1971) presented evidence for nitrogen being adsorbed on the lunar fines. The molecular nitrogen (presumably from the atmosphere) was released during vacuum pyrolysis at temperatures below 150°C. Oró *et al.* (1970) showed that nitrogen was released sporadically during pyrolysis of lunar fines at temperatures near their melting points (1000 to 1100°C). Several investigators using low resolution quadrupole mass spectrometers have reported combined carbon monoxide and nitrogen abundance data (Oró *et al.*, 1970; Gibson and Johnson, 1971). However, the contribution to the mass 28 peak from CO and N₂ can be distinguished by using their corresponding fragment peaks (masses 12 and 16 for CO and mass 14 for N₂).

Additional compounds containing nitrogen have been reported from vacuum pyrolysis of lunar fines. Ammonia (NH₃) was detected by R. C. Murphy *et al.* (1970) and Abell *et al.* (1970a). Hydrogen cyanide (HCN) was reported to be released from Apollo 11 fines by Burlingame *et al.* (1970) while Preti *et al.* (1971) reported the presence of HCN in the Apollo 12 fines.

Hydrogen containing phases, in addition to the hydrocarbons and nitrogen species, released during vacuum pyrolysis of the lunar fines include molecular hydrogen (H₂), water (H₂O), and hydrogen sulfide (H₂S). Molecular hydrogen was reported by Abell *et al.* (1970a, 1971); Epstein and Taylor (1970, 1971); Friedman *et al.* (1970, 1971); Gibson and Johnson (1971); Nagy *et al.* (1970, 1971); Oró *et al.* (1970, 1971) and Funkhouser *et al.* (1971). The water released from the lunar fines may be from terrestrial contamination or may possibly have a solar wind origin (Epstein and Taylor, 1970, 1971; Friedman *et al.*, 1970, 1971; Gibson and Johnson, 1971).

The most abundant sulfur containing phases observed during pyrolysis of lunar fines are H₂S and SO₂ (Gibson and Johnson, 1971; R. C. Murphy *et al.*, 1970; Burlingame *et al.*, 1970; Friedman *et al.*, Oró *et al.*, 1970, 1971; Henderson *et al.*, 1971). The two sulfur containing molecules, H₂S and SO₂, have been shown to be the reaction products of sulfide phases in the fines (e.g. troilite) with metal oxides and silicates (Gibson and Johnson, 1971). The gas release pattern observed for the lunar fines has been duplicated by using lunar soil analogs to which small amounts of troilite have been added (Gibson and Johnson, 1971). Additional sulfur containing

species generated during pyrolysis of the lunar fines include: CS₂ and COS (R. C. Murphy *et al.*, 1970; Henderson *et al.*, 1971).

B. ACID HYDROLYSIS

The second major method employed to detect the presence of low molecular weight compounds in the lunar fines is acid hydrolysis. Each investigator seems to have a favorite technique or procedure which he employs to carry out the acid hydrolysis. The acids used for the hydrolysis included: HCl, DCl, HF, DF, H₃PO₄, H₂SO₄ in addition to H₂O and D₂O (Abell *et al.*, 1970a, b, 1971; Chang *et al.*, 1970, 1971; Oró *et al.*, 1970, 1971; Henderson *et al.*, 1971; Kaplan *et al.*, 1970, 1971). The low molecular weight species released by acid hydrolysis are given in Table III.

TABLE III
Acid hydrolysis of lunar fines - × = species detected

| Sample | Abell 10086 | Abell 12023 | Burlingame 10086 | Chang 10086 | Chang 12023 | Henderson 12023 | Kaplan 10086 | Murphy 10086 | Oró 10086 | Oró 12023 |
|-----------------------------------|----------------|----------------|---------------------|----------------|----------------|--------------------|-----------------|-----------------|--------------|--------------|
| H ₂ | × | | | | | | | | | |
| CH ₄ | × | × | × | × | × | × | × | | × | × |
| NH ₃ | | | | | | | × | | | |
| H ₂ O | | | | | | | | | | |
| C ₂ H ₂ | | × | | × | | | × | | × | |
| CO | | × | × | | | | | | | × |
| N ₂ | × | | | | | | | | | × |
| C ₂ H ₄ | | × | | × | × | × | × | | × | × |
| HCN | | | | | | | | | | |
| C ₂ H ₆ | × | × | | × | | × | × | | × | × |
| O ₂ | | | | | | | | | | |
| H ₂ S | | | | × | × | × | | × | × | × |
| C ₃ H ₈ | | | | × | | × | | | | |
| CO ₂ | | | | | | × | | | × | × |
| COS | | | | | | | | | | |
| SO ₂ | | | | | | | | | | |
| CS ₂ | | | | | | × | | | | |
| Hydro- carbons >C ₃ | | | | × | × | × | | | | |

Carbon dioxide was reported to be produced by acid hydrolysis of both Apollo 11 and 12 lunar fines (Burlingame *et al.*, 1970; Henderson *et al.*, 1971; Oró *et al.*, 1970, 1971). However, Chang *et al.* (1971) did not find CO and CO₂ released from acid hydrolysis of lunar fines and noted that this is consistent with the absence of organic compounds, carbonyls, and carbonates. Therefore, mineral phases containing carbonates, carbonyls and hydrolysable carbon must be absent from the lunar fines despite the claims of Gay *et al.* (1970) and Oró *et al.* (1970).

Every investigator who carried out an acid hydrolysis of lunar fines reported the presence of methane. The quantities of methane produced from the hydrolysis are generally below 20ppm CH₄, similar to the quantities of methane liberated from the

vacuum pyrolysis (Abell *et al.*, 1970a, b; Chang *et al.*, 1970, 1971; Henderson *et al.*, 1971). Acid hydrolysis with deuterium-labeled acids (e.g. DCl, DF) has produced evidence of indigenous CH₄ present in the lunar fines (Abell *et al.*, 1970a, b, 1971; Chang *et al.*, 1970, 1971; Cadogan *et al.*, 1971). The unlabeled methane, produced from the hydrolysis with DCl and DF appears to be indigenous to both the Apollo 11 and 12 lunar fines and its concentration is estimated to be around 2 μg per gram of sample (Chang *et al.*, 1970, 1971; Abell *et al.*, 1970a, b, 1971; Cadogan *et al.*, 1971). In addition to unlabeled methane, acid hydrolysis with DCl also released isotopically labeled species (e.g. CH₃D, CD₄, etc.) indicating that hydrolysable carbide phases were present in the lunar fines. Detailed mineralogical studies of the lunar fines have confirmed the presence of carbides in the lunar samples (Fron del *et al.*, 1970; Anderson *et al.*, 1970; Carter and MacGregor, 1970).

Carbon monoxide was produced by acid hydrolysis and reported by several investigators (Burlingame *et al.*, 1970; Abell *et al.*, 1970a, 1971; Henderson *et al.*, 1971; Oró *et al.*, 1971). Abell *et al.* (1970a) and Oró *et al.* (1971) detected the presence of molecular nitrogen after acid hydrolysis in Apollo 11 and 12 soil samples respectively. Kaplan (personal communication, 1971) reported the possible presence of ammoniacal nitrogen which was generated during acid hydrolysis. The hydrogen cyanide reported during the vacuum pyrolysis of the lunar fines was not detected during the acid hydrolysis of the samples.

Sulfur containing species which were released during acid hydrolysis were reported by several research groups. Burlingame *et al.* (1970) and Henderson *et al.* (1971) noted that H₂S, COS, CS₂, and SO₂ were released from acid hydrolysis, whereas Kaplan *et al.* (1970, 1971) and Oró *et al.* (1970, 1971) reported only the presence of H₂S. Because of the presence of troilite (FeS) in the lunar fines, it would seem that more investigators should have noted the presence of H₂S from acid attack on the fines.

Hydrocarbons, other than methane, released from hydrolysis include C₂H₂, C₂H₄, C₂H₆, C₃H₈ along with hydrocarbons greater than C₃ (Chang *et al.*, 1970, 1971; Henderson *et al.*, 1971; Oró *et al.*, 1970, 1971; Burlingame *et al.*, 1970). Abell *et al.* (1971) and Chang *et al.* (1971) have resolved the hydrocarbons produced from acid hydrolysis into indigenous species and chemical reaction products by the use of deuterium-labeled reagents. Meinschein *et al.* (1970) and Mitchell *et al.* (1971) failed to find detectable quantities of C₁₅-C₃₀ alkanes in the lunar fines at a detection level of 1 part per billion by weight.

C. CRUSHING EXPERIMENTS

The third major method of attack used in determination of possible indigenous low molecular weight species found in the lunar fines is crushing the sample under vacuum and analyzing the gaseous species released. Only three investigators reported results from crushing experiments (Funkhouser *et al.*, 1971; Abell *et al.*, 1970a, Chang *et al.*, 1971). The gaseous species detected upon crushing the lunar samples are given in Table IV.

The three investigators reported the presence of methane from their crushing experiments, while two of the groups noted the presence of ethane along with the methane (Funkhouser *et al.*, 1971., Abell *et al.*, 1970a). The concentration levels of the methane are around 1 ppm CH₄ and 0.1 ppm C₂H₆ (Abell *et al.*, 1970a). Funk-

TABLE IV
Crushing experiments on lunar samples - × = species detected

| Investigator Species Samples | Abell 10086 | Abell 12023 | Chang 12023 | Funkhouser 10046 12070 | |
|------------------------------------|----------------|----------------|----------------|------------------------------|---|
| H ₂ | | | | × | × |
| CH ₄ | × | × | × | × | × |
| N ₂ | | | | × | × |
| C ₂ H ₆ | × | × | | × | × |
| H ₂ S | | | | × | × |
| C ₃ H ₈ | | | | | × |

houser *et al.* (1971) have also reported ppm quantities of H₂, N₂, and H₂S released from crushing a lunar breccia (10046). The efficiency of the crushing process is very low (1–10%) with regard to the release of the total gaseous phases found in the samples (Funkhouser *et al.*, 1971; Abell *et al.*, 1971).

D. NATURE OF THE LOW MOLECULAR WEIGHT SPECIES IN THE LUNAR FINES

The specific nature and origin of the low molecular weight species observed from vacuum pyrolysis, acid hydrolysis, and crushing of the lunar fines is difficult to determine. However, at present, certain pieces of experimental information stand out. The lunar fines are samples which have complex and variable histories with regard to their origin and make-up: they are composed of lithic fragments, individual mineral grains, glass, and meteoritic components. The past history of each of these fragments or components is complex and each has an important role to play in interpreting the observed results from the analysis of the lunar fines for the organogenic elements and compounds.

Carbon containing phases and species are found in the lunar fines in the form of carbides (Fron del *et al.*, 1970; Carter and MacGregor, 1970; Anderson *et al.*, 1970; McKay, personal communication, 1971). Some of the lunar carbides are undoubtedly of meteoritic origin. Chang *et al.* (1971) and Abell *et al.* (1970a, 1971) have presented chemical evidence for carbides in the lunar fines. They believe that the carbides observed possibly represent indigenous lunar carbon, or the product of interaction of solar wind carbon with metal oxides and mineral phases, or meteoritic material on the lunar surface. At the present time, it is difficult to determine the exact nature of the lunar carbides. Wood *et al.* (1971) reported the presence of a single 1–2mm piece of a type-II carbonaceous chondrite in a coarse fines fraction of an Apollo 12 soil sample.

Moore *et al.* (1970, 1971) have theorized that the majority of the carbon found in the lunar fines can be accounted for as having a solar wind origin. They noted that the finest grain size fraction contained the greatest carbon concentrations. Gibson and Johnson (1971) presented evidence for trace quantities of carbon monoxide and carbon dioxide possibly residing within glass and rock vesicles. Abell *et al.* (1971), Chang *et al.* (1971), and Cadogan *et al.* (1971) noted that the hydrocarbons formed during acid hydrolysis probably arise from carbide-like materials contributed by meteoritic impact and solar wind implantation. Zeller *et al.* (1970) had previously shown that proton irradiation of the lunar fines should result in the formation of the observed hydrocarbons and water.

Nitrogen containing compounds are found in the lunar fines in the form of molecular nitrogen trapped within vesicles (Funkhouser *et al.*, 1971). Oró *et al.* (1970) and Gibson and Johnson (1971) have shown that nitrogen is released at elevated temperatures during vacuum pyrolysis of the lunar fines indicating that nitrogen may be dissolved within the individual mineral and glass phases. Nitrogen is also added to the lunar fines from the solar wind. Moore *et al.* (1970, 1971) pointed out that the nitrogen abundances found for the lunar fines correlate with other solar wind derived species.

Sulfur containing species are found in the lunar fines in the form of sulfide minerals. Troilite is a mineral found in minor amounts in the lunar rocks. However, Kaplan *et al.* (1970) reported that sulfur abundances for the Apollo 11 lunar fines (average 650ppmS) are around three times lower than the sulfur abundances found in the mare basalts (average 2200ppmS). The sulfur enrichment in the basalts relative to the fines is also observed in the Apollo 12 samples, however the depletion of sulfur in the fines is not as great (average fines = 615ppmS; average basalts = 780ppmS) (Kaplan and Petrowski, 1971). Thus, sulfur has apparently been lost from the lunar basalts during the formation of the lunar regolith. The addition of sulfur to the lunar fines from meteoritic impact and solar wind addition is apparently very minor. The sulfur phases in the impacting meteorites are apparently vaporized during the crater forming process. The sulfur phases found in the lunar fines are predominantly from breakdown of mare basalts. The sulfur containing species produced by pyrolysis and hydrolysis of the lunar fines, therefore, appear to be the reaction products of the sulfide phases with the metal oxides and silicates or from the acid used in the hydrolysis process.

Some hydrogen gas released from the lunar fines may have been trapped as molecular hydrogen during solidification of magma, (Funkhouser *et al.*, 1971) but in most cases, the solar wind is the major source of hydrogen. The hydrogen gas released from the lunar fines could have been generated during the heating by the reaction of water with metallic iron or ferrous iron present in the silicates.

Another important organogenic element to which little attention has been paid in this review is phosphorus. Phosphorus is present in all lunar samples in trace amounts. Mason and Melson (1970) noted that the range of P contents in Apollo 11 material is quite limited, being about 200 to 900ppm. The major phosphorus-bearing phase is apatite, $\text{Ca}_5(\text{PO}_4)_3(\text{F}, \text{Cl})$, although whitlockite, $\text{Ca}_3(\text{PO}_4)_2$, has been recorded

along with traces of meteoritic schreibersite $(\text{Fe,Ni})_3\text{P}$. KREEP glass contains a relatively high phosphorus content of about 0.26% P (Meyer *et al.*, 1971).

The experimental results obtained from the analysis of lunar samples from three lunar missions indicate that those organogenic elements required for the production of compounds necessary to support an exobiology regime are depleted in the lunar samples. Considering the harshness of the lunar environment (temperature extremes, high vacuum, meteoroid bombardment, lack of water and other volatiles, high solar and cosmic radiation) one is not encouraged in the search for lunar organic compounds.

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