TERRESTRIAL CONTAMINATION IN APOLLO LUNAR SAMPLES

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Abstract. The Apollo lunar samples were seen to offer a unique opportunity in the search for extraterrestrial organic matter without the ambiguity surrounding meteorite analysis due to their unknown contamination histories. The recognition that only a small amount of indigenous organic material was likely to be present in lunar samples combined with the extreme sensitivity of organic analysis methods made it clear that this opportunity could be realized only by carefully controlling the collection, processing, and analysis of the samples in order that they might remain free of significant levels of contamination. The contamination control procedures adopted are described and the analytical evidence obtained throughout the program on potential contamination sources is presented. The organic contaminants actually found in the lunar samples by the various investigators are summarized. It is shown that the program succeeded in providing investigators with samples containing less than 0.1 ppm total contamination.

1. Introduction

The significance of information on extraterrestrial occurrence of organic matter to an understanding of the basic principles of chemical evolution and the origin of life on earth has long been recognized. The return of lunar samples by the Apollo lunar landing missions was seen to offer a unique opportunity for the study of extraterrestrial material free of the ambiguity surrounding meteorite analyses because of their unknown contamination histories. The obtaining of information on the nature and extent of organic matter present in returned lunar samples has, therefore, been one of the primary scientific goals of the Apollo lunar exploration program since its inception.

Organic geochemists have recognized from the beginning of Apollo science planning that only a small amount of organic matter would be likely to be found indigenous to the moon. Even though the concentration of indigenous organic matter may be very small, it can be easily detected by such techniques as gas chromatography, mass spectrometry and emission-absorption fluorescence where detection limits are on the order of one nanogram (1×10^{-9} g). The combination of these two factors made it clear that the unique opportunity offered by lunar samples could be realized only by carefully controlling the collection, processing and analyzing of lunar samples so they might remain free of any significant levels of contamination. Significance in this case was established as the practical average laboratory detection capability of 10^{-9} g g^{-1} achieved by several organic geochemical techniques. (It is granted that absolute instrument sensitivities may be lower but the practical aspects of

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sample handling, including grinding or sieving, extraction, chemical treatment, etc. make it difficult to achieve lower blanks.) The importance of minimizing the number of different contaminating compounds was also stressed, since the presence of a few compounds in known and reproducible quantities is a situation much easier to deal with than a wide spectrum of compounds whose total concentration is of equal magnitude. Maintaining this type and level of contamination control would then allow definitive conclusions to be drawn concerning the true source of organic compounds present in less than part per million quantities. Of course, such low levels of indigenous organic matter have indeed been found to be the case for the lunar samples returned through the Apollo 14 mission (LSPET, 1969; LSPET, 1970a; Levinson, 1970; Levison, 1971; Oró *et al.*, 1971).

The primary purpose of this paper is to describe the contamination control procedures adopted to meet these requirements, to briefly present all analytical evidence obtained throughout the program on potential contamination sources, and to summarize the organic contaminants actually found in the lunar samples by the various principal investigators participating in the analyses.

2. Contamination Control

In order to achieve the goals outlined above it was necessary to (a) identify potential sources of contamination; (b) analyze the possibility of these various potential sources actually reaching the lunar sample in amounts sufficient to invalidate or seriously degrade organic geochemical investigations; and (c) specify the necessary requirements and procedures to assure that such contamination would be held within the specified amounts.

The several potential sources of organic contamination which exist throughout the entire lunar sample collection and analysis procedure beginning with the Apollo Lunar Sample Return Container (ALSRC), in which the samples are stored during return to earth; continuing through processing and distribution of these samples in the Lunar Receiving Laboratory and finally the actual laboratory analyses are given below and discussed further in the body of this report.

(1) Surface contamination of the ALSRC and its outbound contents.

(2) Surface contamination on the Apollo Lunar Hand Tools (ALHT) at the time they are used to obtain samples on the lunar surface.

(3) Exhaust products deposited on the lunar surface by the Lunar Module (LM) descent engine and reaction control system (RCS) engines.

(4) Contamination introduced by exposing the sample to the vacuum (or alternately, nitrogen) environment of the LRL sample processing chamber.

(5) Surface contamination on tools used to process lunar samples in the LRL vacuum (or alternately, nitrogen filled) processing chamber, including storage containers.

(6) Surface contamination on containers used to distribute samples to principal investigators for analysis.

(7) Outgassing of the LM and other lunar surface equipment deposited on the lunar surface.

(8) Astronaut suit leakage deposited on the lunar material prior to or during collection.

(9) Particulate material abraided from the astronaut's suit or other lunar sample equipment during lunar surface activities.

(10) Venting of the LM fuel and oxidizer tanks, cabin and waste system, and the Portable Lunar Logistics System (PLSS or back pack).

(11) Artifacts from laboratory analysis procedures and instrumentation.

Analyses of the various sources and consideration of their relative probabilities of introducing contaminants into the sample led to the conclusions that the first six sources were the most serious. It is fortunate that these sources are those for which one could also establish some control in the Apollo program and/or obtain good information on the types and quantities of contamination they might contribute. These early analyses also led to the conclusion that the major (and least controllable) source of organic contamination would be the LM descent and reaction control system engines. An experimental program was therefore, carried out to obtain information on the organic composition of the engine exhaust gases. Control of the last source listed was, of course, left to the individual principal investigators.

In general, the control of potential organic contamination of the lunar samples has consisted of (i) severe limitations on materials which 'see' or contact the lunar samples, (ii) isolation of the sample in controlled environments at all times, (iii) development of procedures to clean all surfaces which come into direct contact or may 'see' the samples, and (iv) strict controls on fabrication, processing and handling of all lunar sample hardware. Materials actually contacting the lunar sample have been limited to stainless steel, aluminum alloy, and teflon. Materials which may 'see' the lunar sample during collection, storage and processing have included those above plus Viton B, silicone rubber, Pyrex glass, indium-silver alloy and molybdenum disulfide lubricant. Plans called for sealing the samples under ultraclean vacuum or nitrogen environments at all times, although these conditions were not always achieved.

The development of cleaning methods capable of achieving total organic contamination levels on surfaces of less than 10^{-9} g cm^{-2} was accomplished. All surfaces coming into direct contact with the lunar sample were initially required to be cleaned to this level. This includes the ALSRC, and all tools used in LRL processing, and all containers used in the LRL or for distribution to the principal investigators. This level was established by considering the total surface area of the ALSRC including the York mesh packing material (a woven aluminum alloy) and assuming all surface contamination would be transferred to the lunar sample. The cleaning procedure developed to produce these levels includes degreasing where necessary, precleaning by ultrasonification in detergent solution, flushing in isopropyl alcohol, soaking in nanograde purity benzene-methanol solution, and final rinsing by pressurized spray using redistilled Freon. The cleaning steps are carried out in successively cleaner areas with the final rinse being conducted in a Class 100 clean room. After

passing the THC requirements the cleaned item is heat sealed into two successive teflon bags cleaned by the same procedure. Similar cleaning techniques for obtaining less stringent cleanliness levels were developed for cabinets and other equipment which would 'see' the sample but never come into direct contact.

The question of how the final state of cleanliness is determined was given considerable thought early in the program. Practical aspects related to the high volume and rates of cleaning which had to be carried out required that any cleanliness certification test used would be simple and fast. Use of test coupons which would be subsequently pyrolyzed was rejected because of insufficient time for development, even though it would apear to be the most valid test of surface cleanliness. The method actually used involves collecting aliquots of the final rinse solution used in the cleaning procedure, vacuum evaporation of the solvent at room temperatures, and determination of the amount of residue either by direct weighing or gas chromatography of an aliquot of the residue dissolved in a suitable solvent. The gas chromatograph response was correlated with the amount of residue by integrating the total area of all peaks eluted and multiplying them by a calibration factor and the appropriate dilution factor. Knowing the area rinsed then allows calculation of the residue weight per unit area. The direct weight of the residue has the disadvantages of not distinguishing between inorganic particulate matter or organic residues and low sensitivity which dictates evaporating large samples. The gas chromatographic method has the disadvantage of detecting only C_{12}–C_{30} hydrocarbons and other compounds with similar retention times on relatively nonpolar packed columns. It was felt, however, that these types of compounds would be the most likely contaminants and demonstrating their absence would give us a strong confidence that no other organic material was present.

A contamination monitoring scheme was put into effect to assist in the evaluation of cleaning procedures and to obtain the maximum analytical information possible concerning the quantity and type of organic contamination that finds its way into the samples in spite of the controls discussed. This scheme has involved the analysis of York mesh samples or aluminum foil processed with the flight ALSRC's; the analysis of solvent wash samples used to clean all LRL sample processing tools, cabinets, equipment and containers; the analysis of LM exhaust gas products; and the analysis of very clean samples of Ottawa sand exposed to the various processing cabinets in the LRL.

Analyses of the York mesh samples were accomplished by solvent extraction and subsequent gas chromatography, low resolution mass spectrometry, and high resolution mass spectrometry of the extracted residues. LRL hardware cleaning was evaluated by gas chromatographic and high resolution mass spectrometric analyses of the solvent washing residues. Analyses of the Ottawa sand monitors exposed during LRL processing were done by direct pyrolysis (500 °C)-mass spectrometry in the LRL, and additionally aliquots of the sand were provided the several investigators who received lunar samples. A special processing cabinet set up at the University of California at Berkeley for processing the Apollo 14 prime organic sample returned

in a special separate container used a Varian Aerograph Model 1732-20 Trace Gas Analyzer to monitor volatile gases in the nitrogen filled cabinet, a dichloromethane filled bubbler to monitor the nitrogen supply, and alumina placques in addition to Ottawa sand to monitor the actual processing operations (Burlingame *et al.*, 1971a). Detailed information on all these analyses has been published elsewhere (Simoneit *et al.*, 1969; Simoneit and Flory, 1970; Flory *et al.*, 1971: Burlingame *et al.*, 1971b). A summary of the results of these analyses, their evaluations and the conclusions drawn are given below.

(1) Virtually all LM engine exhaust products are of low molecular weight, the bulk being free and combined gas products, including NH_3, H_2O, CO, NO, O_2 and NO_2. As such, they do not constitute a significant lunar surface potential contamination source due to their rapid diffusion over large areas. The organic products are quite varied in composition and somewhat minor in concentration, accounting for only 12% of the exhaust products collected. A complete description of the engine products has been given by Flory *et al.* (1972). Of particular interest are the following compounds: acetylene, hydrogen cyanide, ethylene, formaldehyde, propadiene, ketene, cyanic acid, hydrazoic acid, various methyl amines, acetaldehyde, methyl nitrite, formic acid, nitrous acid, butadiene, various hydrazines, nitromethane and some nitrosohydrazines with traces of other oxidation derivatives of UDMH and hydrazine. The unsaturated and oxygenated organic compounds can be considered as potential starting materials for polymeric structures and the organics containing one to three nitrogen atoms as labels of the LM engine exhaust products in estimating contamination levels. The presence of fluorescent pigments in the exhaust products was detected by Hodgson (1970) in subsequent analyses. Gehrke *et al.* (1970) later examined the exhaust products for the presence of amino acids and detected a derivatizable peak corresponding to one of many observed in their Apollo 11 lunar samples. Hare *et al.* (1970) also examined the exhaust products and reported finding traces of amino acids. The amount of organic material deposited on the lunar surface in the vicinity (400 m radius) of the Apollo 11 landing site was estimated to be 10^{-8} gm cm^{-2}. The bulk sample box was filled with 4×10^3 gm of lunar dust and comprised approximately 10^3 cm^2 of surface area. This would result in organic contamination levels of 2.5×10^{-10} g g^{-1} which would be detectable by some of the most sensitive techniques or organic analysis, notably fluorescence emission-absorption methods and total high resolution mass spectrometry.

(2) Analyses of aluminum foil and York mesh samples from the Apollo 11 mission indicated orgamic contamination levels of about 1 microgram cm^{-2} in the ALSRC's, which could have produced contamination levels of approximately 10^{-6} g g^{-1} in the sample returned in the ALSRC.

(3) Results of Apollo 11 ALSRC monitors led to improvements in cleaning procedures which produced flight hardware for Apollo 12 through 15 with only 10–100 ng cm^{-2} organic contamination.

(4) The 10–100 ng cm^{-2} cleanliness level appears to be the lowest practical limit for York mesh type material. This 10–100 ng cm^{-2} figure is undoubtedly partially

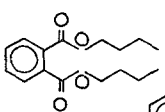
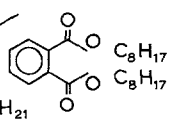
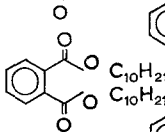
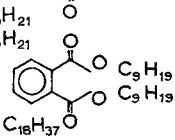
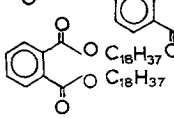
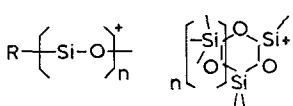
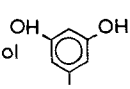
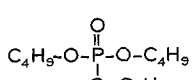
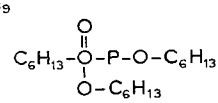
due to aluminum oxide in the extracted residue weights and also is strongly influenced by the adsorptive characteristics of the mesh.

(5) No amino acids were detected in the sand monitors exposed during simulations in the special processing cabinet at the University of California, Berkeley. Very small traces of light hydrocarbons (C_{10}–C_{12}) were detected in the nitrogen supply (Burlingame *et al.*, 1971a, b).

(6) The Sterile Nitrogen Atmosphere Processing (SNAP) line assembled at the LRL for Apollo 14 sample processing has introduced as much as 10 ppm of organic

TABLE I

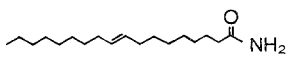
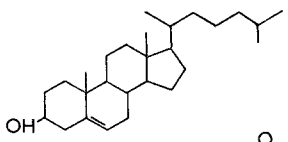
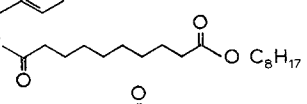
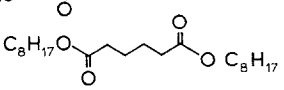
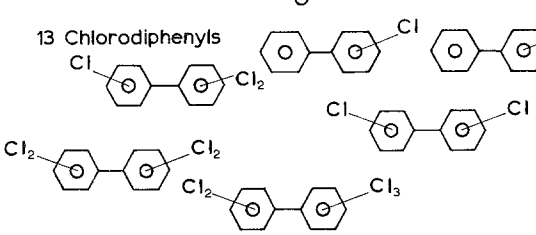
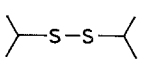
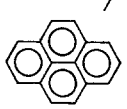
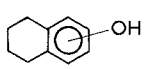
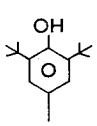
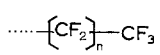
Frequently encountered organic contaminants

Compound Name	Compound structure	Apollo mission	Source
1. Hydrocarbons	$C_nH_{2n\pm z}$ $n = -35$ $z = +2$ to -16	11–14	Ubiquitous
2. Fatty Acids	$C_nH_{2n}O_2$ (some $C_nH_{2n-2,\ etc.}O_2$)	11–14	F-201 (LRL Vacuum proces). Chamber and Apollo Lunar Sample Return Container (ALSRC)
Palmitic Acid	$C_{16}H_{32}O_2$		
Stearic Acid	$C_{18}H_{36}O_2$		
3. 'Octoils'			
Dibutyl Phthalate		11–14	F-201
Dioctyl Phthalate		11–14	Ubiquitous
Didecyl Phthalate		12, 14	ALSRC
Dinonyl Phthalate		14	ALSRC
Dioctadecyl Phthalate		14	ALSRC
4. Silicones		11–14	F-201, Astronaut suit abrasion (Boots)
5. Ethylene oxide polymers		14	Nitrogen sample proces. Cabinet
Trimethylene oxide			
P-Dioxane			
1,3,5,-Trimethyl-2,4,6-Trioxane			
6. Orcinol		14	Nitrogen sample proces. Cabinet
7. Freons	$C_2F_4Cl_2$ (eg.)	11–14	NASA-WSTF Cleaning residual
8. Phosphates (Plasticizers)			
Tributyl Phosphate		11–12	F-201
Trihexyl Phosphate		11–12	ALSRC

contamination to Ottawa sand monitors in the cleanest simulation. These high levels are primarily due to the ethylene oxide sterilization of the cabinets and were comprised primarily of dioctyl pthalate and various polymerization products of ethylene oxide.

A listing of the most common and ubiquitous contaminants documented in this monitoring history is given in Table I, along with their sources. The Apollo mission for which the source is applicable is also given. Note that the number of compounds associated with the Apollo 14 mission is significantly reduced.

Tabel I (Continued)

Compound name	Compound structure	Apollo mission	Source
9. Oleamide		11–12	ALSRC
10. Cholesterol		11–14	ALSRC, York mesh monitor
11. Dibutylsebacate		11, 12	F-201
12. Dioctyladipate		12	*SESC Lid (Apollo 12)
13 Chlorodiphenyls		11, 12	ALSRC
14. Diisopropyldisulfide		12	ALSRC
15. Pyrene		11, 12	F-201
16. Tetrahydronaphthol		12	ALSRC
17. Ionol		11	Curator polypropylene bottles
18. Teflon		11, 12	Nitrogen processing chamber

*SESC is the Special Environmental Sample Container used for storage of the 'prime' organic sample. Attempts have been made on Apollo 12–15 to provide the cleanest possible sample by use of this special container.

3. Lunar Sample Contamination

The success of the organic contamination control program described in the previous section is determined solely by the quality of the lunar samples delivered to the principal investigators. It is important to stress that the numerous compounds identified in the LM exhaust and the various monitors are only potential lunar contaminants and may or may not find their way into the lunar sample. Two types of analytical data are available which allow us to assess the terrestrial organic contamination levels in the lunar samples: (1) the results of pyrolysis-mass spectrometry analyses conducted at the LRL, and (2) the organic compounds found by the various principal investigators during their respective investigations. In addition microscopic and direct visual examination of the lunar material may reveal the presence of any particulate contamination.

Pyrolysis-mass spectrometry analyses in the LRL were carried out during simulations and Apollo 11 and 12 sample processing as part of the preliminary examination as described by LSPET (1969) and (1970). Monitoring of the processing activities in the Lunar Receiving Laboratory during simulations prior to Apollo 11 indicated that organic contaminations levels as high as 1000 ppm might be introduced to the lunar samples. Improvements in handling techniques, cleaning procedures and orientation of laboratory personnel reduced this level to less than 1 ppm during processing of Apollo 11 lunar material. Further controls, more thorough cleaning and greater attention to potential sources of contamination reduced this level to less than 0.1 ppm during Apollo 12 sample processing. The small size of lunar samples analyzed and the low concentrations of organic material made it impossible to identify specific contaminants but in general the contamination resembled that associated with F201 and the ALSRC listed in Table I. Pyrolysis-mass spectrometry analysis data will not be available for the Apollo 13 mission and is not yet completed for Apollo 14 sample processing.

Several investigators have reported identification of contaminants in the lunar samples provided them for study from the Apollo 11 and 12 missions. Table II lists the contaminants identified in the Apollo 11 bulk fines, their estimated concentrations and the investigators involved. In general these contaminants can be correlated with the compounds in Table I or LM exhaust products with the possible exception of the sulfur containing compounds and ethylene glycol. The sulfur containing compounds may be derived from a polysulfide material used as a sealant in the LRL nitrogen atmosphere cabinets. The ethylene glycol can be attributed to the ethylene oxide employed as a sterilizing agent in the LRL. It is apparent from the data in Table II and from the published organic analyses (Levinson, 1970) that terrestrial contamination levels generally did not exceed 1 ppm in the Apollo 11 samples. Indeed, many samples were considerably less contaminated. The relatively high amount of particulate contamination is disturbing and may be largely responsible for different results obtained on seemingly identical samples. The general agreement between the pyrolysis-mass spectrometry analyses carried out at the LRL and the results of the various

TABLE II
Contaminants identified in Apollo 11 bulk fines sample

Contaminant(s)	Conc.	Principal investigator (reference)	Method of analysis
Diisopropyl disulfide	1–10 PPM	Oró (Oró *et al.*, 1970; Gibert *et al.*, 1971)	Solvent extraction-GC-MS
Diisopropyl disulfide, ethylene glycol, Isopropylthiol and dialkyl pthalates	< 1 PPM	Biemann (Murphy *et al.*, 1970)	Pyrolysis-high resolution MS
Fluorescent porphyrin like pigments (lunar exhaust)	0.1 PPB	Hodgson (Hodgson *et al.*, 1970)	Solvent extraction-fluorescent emission – adsorption
LM exhaust products, hydrocarbons to C_{10} and pthalates	< 10 PPB	Calvin (Burlingame *et al.*, 1970)	Solvent extraction-GC-high resolution MS
Octoil and lunar exhaust products	Trace	Ponnamperuma (Gehrke *et al.*, 1970)	Mass spectrometry
Toluene, C_2 alkyl benzene, phenol, diphenyl, a methyl ester	PPB	Nagy (Murphy *et al.*, 1970	Solvent extraction-GC-MS
Cellulose fibers Teflon and other organic materials	Many	Schopf (Schopf, 1970)	Optical microscope
Mylar and teflon	–	LSPET (LSPET, 1970b)	Optical microscope and pyrolysis-mass spectrometry

investigators indicates that most of the contamination was introduced prior to the sample being delivered to the investigator. This is further substantiated if the contaminants are found in Table I or in the LM exhaust products.

The contaminants identified in Apollo 12 samples and the investigators reporting them are listed in Table III. The number of contaminants is decreased compared to Apollo 11 even though many more samples were analyzed. This data combined with the published organic analyses (Levinson, 1971; Nagy *et al.*, 1971 and Harada *et al.*, 1971) of Apollo 12 samples indicate that terrestrial contamination levels were less than 0.1 ppm. This again is in agreement with the LRL pyrolysis-mass spectrometry results. The greatly reduced particulate contamination compared to Apollo 11 is especially encouraging and may be a significant factor in the overall reduction in contamination levels observed in the various organic analyses.

The question of amino acid contamination is one which cannot be settled from Apollo 11 and 12 data. Nagy *et al.* (1971) reported finding amino acids attributable to hand contamination and no others in Apollo 12 sample 12011 and 12023. Harada *et al.* (1971) detected amino acids in 12001 and 12033 which they feel are not typical of terrestrial contamination. Both of these groups detected amino acids in Apollo 11 sample 10086 which they consider indigenous. Oró *et al.* (1970) and Gehrke *et al.* (1970) searched for and did not find amino acids in the same Apollo 11 sample. Gehrke *et al.* (1971) also searched for and did not detect any amino acids in an unidentified Apollo 12 sample. These apparent discrepancies can in part or wholly

TABLE III
Contaminants identified in Apollo 12 lunar samples

Sample No.	Contaminant(s)	Conc.	Principal investigator	Methods of analysis
12001	Fluorescent pigments (lunar exhaust)	–	Hodgson *et al.* (1971)	Solvent extract – fluorescent absorption – emission
12025, 12028	Silicone rubber polystyrene teflon hydrocarbons to *m/e* 250	0.2–1.0 ppm	Burlingame *et al.* (1971c)	Pyrolysis-mass spectrometry
12011, 12023, 12032, 12042	Lunar exhaust products hydrocarbons	–	Calvin (Henderson *et al.* (1971)	Solvent extraction – direct probe high resolution mass spectrometry
12001, 12023	Amino acids	Low ppb	Nagy *et al.* (1971)	Ion-exchange chromatography
	Teflon			Helium pyrolysis
12001, 12032 12033, 12037 12042, 12023 12034, 12026 12028	Teflon and mylar	$<<$ Apollo 11	Schopf (1971)	Optical microscope

be due to contamination, differences in detection limits, inhomogeneity of the sample and differences in analytical procedures. A special sample was designated from Apollo 14 returned lunar material to resolve this problem. This sample was returned in a separate sealed container and processed in a special cabinet at the University of California, Berkeley.

Contamination by organic compounds can also affect the results of carbon isotope measurements. Some investigators (Kaplan *et al.*, 1970; Friedman *et al.*, 1970; and Epstein and Taylor, 1970) attributed some of their observed variations in ^{13}C content to contamination. It seems unlikely, however, that the small concentrations of organic contaminants detected could cause the observed variations and discrepancies.

Information is not yet available on organic contaminants in Apollo 14 samples, but preliminary results in our laboratory indicate the levels are as low or lower than those found in Apollo 12 samples.

In conclusion, it can be stated that a contamination control plan was developed and implemented which eventually resulted in providing investigators with lunar samples containing less than 0.1 ppm total organic contamination. It should be noted that this is as low or lower than the experimental blanks obtained in organic geochemistry research laboratories.

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