

ARTICLE

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Deoxyribose and deoxysugar derivatives from photoprocessed astrophysical ice analogues and comparison to meteorites

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Gaurav Vishwakarma 02/11/2019

Meteorite: a piece of rock or metal that has fallen to the earth's surface from outer space like a meteor.



- Formose reaction: involves the formation of sugars from formaldehyde. The term formose is a portmanteau of *form*aldehyde and ald*ose*
- (+)-Butanol/TFAA = trifluoroacetic anhydride
- BSTFA = N,O-bis(trimethylsilyl)trifluoroacetamide
- MTBSTFA = N-tert-butyldimethylsilyl-N-methyltrifluoroacetamide

Stochastic: having a random probability distribution or pattern that may be analyzed statistically but may not be predicted precisely.

Importance

Astrophysical ice analogues Sugars, **Deoxysugars** amino acids, nucleobases, amphiphiles

Meteorites Sugars, amino acids, nucleobases, amphiphiles

Background Work

letters to nature

Carbonaceous meteorites as a source of sugar-related organic compounds for the early Earth

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* NASA Ames Research Center, Moffett Field, California 94035, USA † International Research School of Planetary Sciences (IRSPS), Universita' d'Annunzio Viale Pindaro, 42, 65127 Pescara, Italy has hitherto been no conclusive evidence for the existence of polyols in meteorites, leaving a gap in our understanding of the origins of biologically important organic compounds on Earth. Here we report that a variety of polyols are present in, and indigenous to, the Murchison and Murray meteorites in amounts comparable to amino acids. Analyses of water extracts indicate that extraterrestrial processes including photolysis and formaldehyde chemistry could account for the observed compounds. We conclude from this that polyols were present on the early Earth and therefore at least available for incorporation into the first forms of life.

Our study of Murchison and Murray polyols uses a relatively definitive means of detection, gas chromatography-mass spectro-

	Sugars	Sugar Alcohols	Sugar Acids	Dicarboxylic Sugar Acids		Deoxy Sugar	Acids	
3C	$\begin{array}{c} CH_2OH\\ I\\ C=0\\ I\\ CH_2OH\\ \end{array}$ Dihydroxyacetone	CH ₂ OH HCOH CH ₂ OH Glycerol 160 nmol/g (100%)	$\begin{array}{c} & \text{CO}_2\text{H} \\ \text{I} & \text{C} & -\text{OH} \\ \text{I} & \\ & \text{CH}_2\text{OH} \\ \end{array}$ Glyceric acid 80 nmol/g					
4C		$\begin{array}{c} \mathrm{CH_{2}OH}\\ \mathrm{H-C-OH}\\ \mathrm{H-C-OH}\\ \mathrm{H-C-OH}\\ \mathrm{CH_{2}OH}\\ \mathrm{Erythritol\ \&\ Threitol}\\ (1\%) \end{array}$	$\begin{array}{c} \mathrm{CO_{2}H} \\ \\ \mathrm{H-C-OH} \\ \\ \mathrm{H-C-OH} \\ \\ \mathrm{CH_{2}OH} \\ \end{array}$ Erythronic & Threonic acid (4nmol/g)	$\begin{array}{c} \mathrm{CO_{2}H}\\ \mathrm{I}\\ \mathrm{H}-\mathrm{C}-\mathrm{OH}\\ \mathrm{I}\\ \mathrm{HO}-\mathrm{C}-\mathrm{H}\\ \mathrm{I}\\ \mathrm{CO_{2}H}\\ \mathrm{Tartaric}\ \&\\ \mathrm{Mesotartaric\ acid}\end{array}$	CO ₂ H H ₃ C — C — OH CH ₂ OH 2-Methyl glyceric acid	$\begin{array}{c} & \text{CO}_2\text{H} \\ \text{H} - \overset{\text{I}}{\text{C}} - \text{OH} \\ \text{H} - \overset{\text{I}}{\text{C}} - \text{H} \\ \text{H} - \overset{\text{I}}{\text{C}} - \text{H} \\ \text{H} - \overset{\text{I}}{\text{C}} + \overset{\text{I}}{\text{CH}_2\text{OH}} \\ \end{array}$	$\begin{array}{c} & \text{CO}_2\text{H} \\ \text{H}-\text{C}-\text{OH} \\ \text{I} \\ \text{H}-\text{C}-\text{OH} \\ \text{I} \\ \text{CH}_3 \end{array}$ 2, 3 Dihydroxy butyric acid (& diastereomer)	$\begin{array}{c} \text{CO}_2\text{H}\\ \text{I}\\ \text{H}-\text{C}-\text{H}\\ \text{I}\\ \text{H}-\text{C}-\text{OH}\\ \text{I}\\ \text{CH}_2\text{OH}\\ 3,4 \text{ Dihydroxy}\\ \text{butyric acid} \end{array}$
5C		$\begin{array}{c} CH_2OH\\ \\ H-C-OH\\ H-C-OH\\ H-C-OH\\ \\ H-C-OH\\ \\ CH_2OH\\ \end{array}$ Ribitol & Isomers	$\begin{array}{c} \text{CO}_{2}\text{H}\\ \text{I}\\ \text{H}-\text{C}-\text{OH}\\ \text{I}\\ \text{H}-\text{C}-\text{OH}\\ \text{I}\\ \text{H}-\text{C}-\text{OH}\\ \text{I}\\ \text{CH}_{2}\text{OH}\\ \end{array}$ Ribonic acid & Isomers	$\begin{array}{c} & & CO_2H \\ & \\ H-C-OH \\ HO-C-H \\ H-C-OH \\ \\ H-C-OH \\ \\ CO_2H \end{array}$	$\begin{array}{c} & CO_2H \\ I \\ H-C-H \\ H-C-OH \\ I \\ H-C-OH \\ I \\ CH_2OH \end{array}$			
6C	*	$\begin{array}{c} CH_2OH \\ H - C - OH \\ H O - C - H \\ H O - C - H \\ H - C - OH \\ H - C - OH \\ H - C - OH \\ C H_2OH \\ \end{array}$ Glucitol & Isomers	$\begin{array}{c} \text{CO}_{2}\text{H}\\ \\ \text{H}-\text{C}-\text{OH}\\ \\ \text{HO}-\text{C}-\text{H}\\ \\ \text{H}-\text{C}-\text{OH}\\ \\ \text{H}-\text{C}-\text{OH}\\ \\ \text{CH}_{2}\text{OH}\\ \end{array}$ Gluconic acid & lsomers	$\begin{array}{c} & CO_2H \\ I \\ H - C - OH \\ HO - C - H \\ H - C - OH \\ I \\ H - C - OH \\ C - OH \\ CO_2H \\ \hline \\ Glucaric acid \\ \& Isomers \end{array}$	$\begin{array}{c} & CO_2H \\ \\ H-C-H \\ H-C-OH \\ \\ H-C-OH \\ \\ H-C-OH \\ \\ C-OH \\ \\ CH_2OH \end{array}$	$\begin{array}{c} & & & CO_2H \\ & & & & I \\ H - C - OH \\ H - C - H \\ H - C - OH \\ H - C - OH \\ H - C - OH \\ CH_2OH \end{array}$ 3-Deoxyhexonic aci	d	

Racemic amino acids from the ultraviolet photolysis of interstellar ice analogues

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 † NASA-Ames Research Center, Mail Stop 245-6, Moffett Field, California 94035-1000, USA most volatile species (that is, H₂, He, Ne) condense onto grains, coating them with a thin layer of ice⁹. This ice is composed primarily of amorphous H₂O, but usually also contains a variety of other simple molecules^{9,10}, such as CO₂, CO, CH₃OH, and NH₃. Laboratory studies¹¹ and astronomical observations^{10,12} indicate that radiation processing of such ices can create complex organic compounds¹³. Many of the organic molecules that are present in carbonaceous chondrites (primitive carbon-rich meteorites) and comet and asteroid dust are thought to come, at least in part, from the ice and complex compounds constructed in the interstellar medium (ISM).

Perhaps the most convincing molecular evidence for the interstellar heritage of meteoritic molecules is their high deuterium (D) enrichment^{3,14}. At the low temperatures in dense molecular clouds deuterium fractionation is efficient and elevated D/H ratios are seen

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THE PHOTOCHEMISTRY OF PYRIMIDINE IN REALISTIC ASTROPHYSICAL ICES AND THE PRODUCTION OF NUCLEOBASES

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ABSTRACT

Nucleobases, together with deoxyribose/ribose and phosphoric acid, are the building blocks of DNA and RNA for all known life. The presence of nucleobase-like compounds in carbonaceous chondrites delivered to the Earth raises the question of an extraterrestrial origin for the molecules that triggered life on our planet. Whether these molecules are formed in interstellar/protostellar environments, in small parent bodies in the solar system, or both, is currently unclear. Recent experiments show that the UV irradiation of pyrimidine ($C_4H_4N_2$) in H₂O-rich ice mixtures that contain NH₃, CH₃OH, or CH₄ leads to the formation of the pyrimidine-based nucleobases uracil, cytosine, and thymine. In this work, we discuss the low-temperature UV irradiation of pyrimidine in realistic astrophysical ice mixtures containing H₂O, CH₃OH, and NH₃, with or without CH₄, to search for the production of nucleobases and other prebiotic compounds. These experiments show the presence of uracil, urea, glycerol, hexamethylenetetramine, small amino acids, and small carboxylic acids in all samples. Cytosine was only found in one sample produced from ices irradiated with a higher UV dose, while thymine was not found in any sample, even after irradiation with a higher UV dose. Results are discussed to evaluate the role of the photochemistry of pyrimidine in the inventory of organic molecules detected in meteorites and their astrophysical/astrobiological implications.

Key words: astrobiology – astrochemistry – ISM: molecules – methods: laboratory: molecular – molecular processes – ultraviolet: ISM

Overall Scheme

Experiments

Irradiation of ices at 12 K

- 1) $H_2O: CH_3OH in 2:1 + UV$
- 2) $H_2O: {}^{13}CH_3OH in 2:1 + UV$
- 3) Blank + UV
- 4) $H_20 + UV$
- 5) H₂O: ¹³CH₃OH in 2:1

Analysis/Detection

GC-MS methods

1) **(+)-butanol/TFAA** (identification and separation of enantiomers)

2) **BSTFA** (identification of sugar derivatives containing 3 or more derivatizable groups)

3) **MTBSTFA** (identification of smaller deoxysugar derivatives that contain only two OH groups

Products

Sugars, Deoxysugars, Deoxysugar alcohols, Deoxysugar acids

Irradiation of ices at low temperature.

- > Gas mixtures were prepared in a glass line (background pressure: ~10⁻⁶ mbar) by mixing the vapors of H_2O and either CH_3OH or ¹³ CH_3OH in a glass bulb (volume: 2.09 L).
- Ratios between mixture components were determined by their partial pressure (±0.05 mbar).
- Typical residues were produced from H₂O:CH₃OH (2:1) and H₂O:¹³CH₃OH (2:1) ice mixtures in which totals of 1.23 mmol of H₂O and 0.61 mmol of CH₃OH or ¹³CH₃OH were co-deposited at rates of about 1.7 µmol min⁻¹, while being simultaneously irradiated for 17–19 h with photon doses of 0.35–0.39 photons molecule⁻¹.
- > The UV source used was a microwave-powered H_2 -discharge UV lamp.
- After simultaneous deposition and irradiation, samples were warmed under static vacuum to room temperature at about 0.75 K min⁻¹, at which time each residue covering the substrate was taken with 200 μL of H2O and transferred into a clean, prebaked (500 °C) vial for further analysis.

Analysis of laboratory residues at room temperature:

1) Each H2O-dissolved residue was divided into smaller aliquots and analyzed with three different GC-MS methods.

2) Before any derivatization, each aliquot was dried in a desiccator under vacuum for 1–2 h.

3) The three derivatizations methods used in this work are:

(a) (+)-butanol/TFAA (identification and separation of enantiomers),

(b) BSTFA (identification of sugar derivatives containing 3 or more derivatizable groups),

(c) MTBSTFA (identification of smaller deoxysugar derivatives that contain only two OH groups).

Table 1 Deoxysugar derivatives identified in the ice photolysis residues (regular and ¹³ C-labeled)						
Compounds ^a	Formulas	R _t (min) ^b	Abundances in residues ^c (pmol)	Detected in meteorites?		
Deoxysugars						
2-Deoxyribose	$C_5H_{10}O_4$	61.2, 61.4	217-3855	Undetermined ^k		
2-Deoxyxylose ^d	$C_5H_{10}O_4$	57.0, 57.3	373-3636 ^e	Undetermined ^k		
Deoxysugar alcohols						
1,2-Propanediol ^f	$C_3H_8O_2$	9.9	<u>≥</u> 8-375	Yes ^{l,m}		
1,3-Propanediol ^{f,g}	$C_3H_8O_2$	36.9	≥19-27	No		
2-Methyl-1,3-propanediol ^{g,h}	$C_4H_{10}O_2$	38.7	≤1038-3354 ^h	No		
2-(Hydroxymethyl)-1,3-propanediol	$C_4H_{10}O_3$	30.9	n.d.	Yes ^I		
1,2,3-Butanetriol	$C_4H_{10}O_3$	14.5	6-39	No		
1,2,4-Butanetriol	$C_4H_{10}O_3$	32.2	35-50	Yes ^I		
Deoxysugar acids						
3,4-Dihydroxybutyric acid ^{i,j}	$C_4H_8O_4$	16.5	_	Yes ⁿ		
Sugars						
Ribose	$C_{5}H_{10}O_{5}$	64.7, 65.0	237-2467	No		

n.d., Not detected

^aCompounds were detected using the (+)-2-butanol/TFAA derivatization method, unless otherwise stated

^bRetention times (R_t) correspond to average values in the GC-MS chromatograms of the residues, or to standard chromatograms if compounds were not detected in the residues. Chiral compounds whose enantiomers are separated have two retention times

^cAbundances for chiral compounds correspond to the sum for both enantiomers

^dTentatively identified by comparison of its mass spectrum with that of the 2-deoxyribose standard (see Fig. 1 and Supplementary Fig. 3)

eAbundances estimated based on the GC-MS detector response for the 2-deoxyribose standard

Volatile compounds that may have been partially lost during the warm-up phase and/or the sample preparation. Abundances given thus correspond to lower limits

^gDetected in samples derivatized with the MTBSTFA method

^hElutes with another unidentified compound with similar mass fragments, so abundances given are upper limits

ⁱTentatively identified by comparison with the NIST mass spectrometry library in samples derivatized with the BSTFA method

^jMay be present in its dimer form

^kThe presence of these compounds in meteorites is uncertain, and further analyses are required

Detected in Murchison (Fig. 4)

^mDetected in GRA 06100 (Fig. 4)

ⁿDetected in Murchison and Murray⁴



Fig. 1 Identification of 2-deoxyribose in ice photolysis residues. **a** Single-ion monitoring (SIM) chromatograms of residues produced from the UV irradiation of H₂O:CH₃OH (2:1) (12 C sample, *m*/*z* = 195 Da) and H₂O: 13 CH₃OH (2:1) (13 C sample, 200 Da) ice mixtures after derivatization with (+)-2-butanol/TFAA. The peaks around 57.0 and 57.3 min in both chromatograms are tentatively assigned to 2-deoxyxylose (see Supplementary Fig. 2). Intensities are offset for clarity. **b** From top to bottom, mass spectra of the peaks assigned to 2-deoxy-L-ribose in the regular residue, the ¹³C-labeled residue, and a standard of 2-deoxyribose. The molecular structure of 2-deoxyribose is shown without derivatization. Assignments of the fragments in the mass spectra can be found in Supplementary Table 1



Supplementary Fig. 5 Determination of the level of contaminant 2-deoxyribose and ribose in the residues. **a**, From top to bottom, SIM chromatograms (m/z = 195 Da) of a residue produced from an H₂O:CH₃OH (2:1) ice mixture, a piece of Al foil with no deposited ices that was exposed to UV irradiation, a pure H₂O ice (no methanol) deposited and exposed to UV irradiation, and an H₂O:¹³CH₃OH (2:1) ice mixture deposited but UV irradiated, showing the peaks for the L and D enantiomers of 2-deoxyribose. **b**, From top to bottom, SIM chromatograms (278 Da) of the same residue and the same three control samples, showing the peaks for the L and D enantiomers of ribose. All chromatograms are plotted using the same intensity scale, with intensities offset for clarity. Chromatograms of control samples show some contaminant 2-deoxyribose in the control samples, but no contaminant ribose. Abundances of 2-deoxyribose in Tables 1 and 2 were corrected by subtracting the contribution of the contaminant 2-deoxyribose in the control sample in which an H₂O:¹³CH₃OH (2:1) ice mixture was deposited but not UV irradiated.

Deoxysugar alcohols



Fig. 2 Identification of the 4C deoxysugar alcohols 2-methyl-1,3-propanediol, 1,2,3-butanetriol, and 1,2,4-butanetriol in ice photolysis residues. **a** SIM chromatograms of residues produced from the UV irradiation of H₂O:CH₃OH (2:1) (¹²C sample, m/z = 261 Da) and H₂O:¹³CH₃OH (2:1) (¹³C sample, 264 Da) ice mixtures after derivatization with MTBSTFA. Intensities are offset for clarity. **b** From top to bottom, mass spectra of the peaks assigned to 2-methyl-1,3-propanediol in the regular residue, the ¹³C-labeled residue, and a standard of 2-methyl-1,3-propanediol. The difference in relative intensities for the peaks at 133 and 261 Da suggests a coelution with an unidentified compound with similar mass fragments. **c** SIM chromatograms of the same regular (141 Da) and ¹³C-labeled (143 Da) residues after derivatization with (+)-2-butanol/TFAA. Intensities are offset for clarity. **d** From top to bottom, mass spectra of the peaks assigned to 1,2,3-butanetriol in the regular residue, the ¹³C-labeled residue, and a standard of 1,2,3-butanetriol. **e** SIM chromatograms

Identification deoxysugar alcohols in meteorites



Fig. 4 Identification of three deoxysugar alcohols in meteorites. **a** Single-ion chromatogram (SIC) of a sample from GRA 06100 (m/z = 141 Da) derivatized with (+)-2-butanol/TFAA. **b** Mass spectrum of the peak assigned to 1,2-propanediol, compared with the mass spectrum of a standard of 1,2-propanediol. **c** SIC of a sample from Murchison (167 Da) derivatized with (+)-2-butanol/TFAA. **d** Mass spectrum of the peak assigned to 2-(hydroxymethyl)-1,3-propanediol, compared with the mass spectrum of a standard of 2-(hydroxymethyl)-1,3-propanediol. **e** SIC of the same Murchison sample (153 Da). **f** Mass

Compounds	Underivatized Formulas (Masses in Da)	Derivatized Masses (Da) (' ² C)	Derivatized Masses (Da) ('°C)	Structures of Molecular lons and Fragments
1,2-Propanediol ^{a,†}	C ₃ H ₈ O ₂ (76)	268*	(271) [†]	$M^{+}[2 \times TFA]$
		155	(158) [†]	M* – (TFA–O)
		141	(143) [†]	M^+ – (TFA–O–CH ₂)
1,3-Propanediol ^{b,‡}	C ₃ H ₈ O ₂ (76)	304*	(307)‡	M^+ [2 × t-BDMS]
		247	(250) [‡]	M ⁺ – (t -butyl)
		219	(221)‡	M ⁺ - (t-butyl) - (CO)
2-Methyl-1,3-propanediol ^b	C ₄ H ₁₀ O ₂ (90)	318*	322*	$M^{+}[2 \times t - BDMS]$
		261	264	$M^{+} - (t - butyl)$
2-(Hydroxymethyl)-1,3-propanediol ^{a,†}	C ₄ H ₁₀ O ₃ (106)	394*	(398) [†]	M* [3 × TFA]
		281	(285) [†]	M* – (TFA–O)
		167	(171) [†]	M ⁺ - (TFA-0) - (TFA-0H)
		166	(170) [†]	M* – 2 (TFA–OH)
,2,3-Butanetriol ^a	C ₄ H ₁₀ O ₃ (106)	394*	398*	M^{+} [3 × TFA]
		281	285	M* – (TFA–O)
		253	255	M ⁺ – (TFA–O–CH–CH ₃)
		167	171	M* - (TFA-0) - (TFA-0H)
		141	143	TFA-O-CH-CH ₃
1,2,4-Butanetriol ^a	C ₄ H ₁₀ O ₃ (106)	394*	398*	M^* [3 × TFA]
		281	285	M* – (TFA–O)
		267	270	M^+ – (TFA–O–CH ₂)
		167	171	M ⁺ – (TFA–O) – (TFA–OH)
		166	170	M* – 2 (TFA–OH)
		153	156	M ⁺ - (TFA-O-CH ₂) - (TFA-OH)
2-Deoxyribose ^a , 2-deoxyxylose ^a	C ₅ H ₁₀ O ₄ (134)	382*	387*	M [*] [butyl + 2 × TFA]
		309	314	M ⁺ – (butyl–O)
		195	200	M ⁺ - (butyl-O) - (TFA-OH)
		81	86	M ⁺ - (butyl-O) - 2 (TFA-OH)
Ribose ^a	C ₅ H ₁₀ O ₅ (150)	494*	499*	M [*] [butyl + 3 × TFA]
		421	426	M ⁺ – (butyl–O)
		278	282	M* - (butyl-O) - (TFA-OH) - (CHO)
		193	198	M ⁺ - (butyl-O) - 2 (TFA-OH)

a Derivatized with (+)-2-butanol/TFAA; b Derivatized with MTBSTFA

[†] Only found in meteorites (Fig. 4), so there is no ¹³C-labelled mass spectrum; [‡] Only detected in regular (¹²C) residues (Supplementary Fig. 1)

M^{}, molecular ion (not observed); butyl, (+)-butyl group (CH₁CH₃)CH₂CH₃, from derivatization; 57 Da); TFA, trifluoroacetyl group (CF₃-C=O, from derivatization; 97 Da); *t*-BDMS, *tert*-butyldimethylsylil group (Si(CH₃)₂C(CH₃)₃, from derivatization; adds 114 Da after loss of a hydrogen); *t*-butyl, *tert*-butyl group (C(CH₃)₃, from derivatization; 57 Da); CO, carbon and oxygen attached to a *t*-butyl group (28 Da for ¹²C, 29 Da for ¹³C); CHO, aldehyde group (29 Da for ¹²C, 30 Da for ¹³C) The molecular structures of the derivatization tags are shown in Supplementary Fig. 1

- The formation mechanisms for deoxysugar derivatives under experimental conditions simulating ice photochemistry in cold astrophysical environments are difficult to determine, due to the stochastic nature of the chemical processes taking place in the ice matrix.
- ➤ Indeed, the energy of the incident UV photons is sufficient to break chemical bonds and ionize species, resulting in the release of H atoms together with OH, CH₃, CH₂OH, and CH₃O radicals and ions from the photolysis of H₂O and CH₃OH.
- In addition, a formose-type mechanism is expected to yield sugars as the majority products, including branched compounds, but no deoxysugar derivatives.

- All the residues produced in this work contained a wide variety of sugars, sugar alcohols, and sugar acids.
- In addition to these common sugar derivatives, they identified several deoxysugars, deoxysugar alcohols, and deoxysugar acids.
- This is the first definitive identification of a deoxysugar in laboratory ice photolysis residues.
- Decreasing order of abundance-
- sugar alcohols > sugars > sugar acids
- In particular, the presence of both sugar and deoxysugar derivatives in laboratory residues and meteorites compounds favours ice photochemistry over a formose-type reaction mechanism for their formation.
- However, the formation mechanism and meteoritic distribution of these compounds need to be studied in more detail.