What Do Molecular Markers Tell Us About the Sources of Organic Carbon?

MARAMA 2007 Regional Haze Science Meeting
Monica Mazurek, Civil & Environmental Engineering Department
Timonium, MD
July 10-11, 2007
Geochemical studies of sources and fate of carbonaceous PM

Hills of Westwood, circa early 1980’s

UCLA, Los Angeles, CA
GCMS analysis organic PM

GCMS Plot West LA August

- Composite 5-6 filters (24-hr), 6 day cycle
- 3.8 μg/m³ (elutable organic compounds)
### Molecular markers in ambient PM...

#### TOTAL

<table>
<thead>
<tr>
<th><strong>Alkanes</strong></th>
<th><strong>PAHs</strong></th>
<th><strong>Other</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>n-pentacosane</td>
<td>benzo[b]fluoranthene</td>
<td>hopanes</td>
</tr>
<tr>
<td>n-hexacosane</td>
<td>benzo[k]fluoranthene</td>
<td>steranes</td>
</tr>
<tr>
<td>n-heptacosane</td>
<td>benzo[e]pyrene</td>
<td>nonanal</td>
</tr>
<tr>
<td>n-octacosane</td>
<td>indeno[1,2,3-cd]pyrene</td>
<td>diterpenoids</td>
</tr>
<tr>
<td>n-nonacosane</td>
<td>indeno[1,2,3-cd]fluoranthene</td>
<td>sterols</td>
</tr>
<tr>
<td>n-triacontane</td>
<td>retene</td>
<td>cholesterol</td>
</tr>
<tr>
<td>n-hentriacontane</td>
<td>coronene</td>
<td>7H-benz[de]anthracen-7-one</td>
</tr>
<tr>
<td>n-dotriacontane</td>
<td></td>
<td>benz[a]anthracene-7,12-dione</td>
</tr>
<tr>
<td>anteiso-triacontane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>iso-hentriacontane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>anteiso-hentriacontane</td>
<td></td>
<td></td>
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<tr>
<td>iso-dotriacontane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>anteiso-dotriacontane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>iso-tritriacontane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>phytane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>pristane</td>
<td></td>
<td></td>
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</tbody>
</table>

#### Acids

- 21 n-alkanoic acids (with \( C_{10} \) to \( C_{30} \))
- 10 aliphatic dicarboxylic acids
- 1 aromatic polycarboxylic acid

<table>
<thead>
<tr>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>cis-9-n-octadecenoic acid</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Petroleum biomarkers

- Characterization crude oils
- Geochemical evidence oil field evaluation
- Distribution patterns homologs large organic
- Sources.....
Fossil bacteria and microbiota became today’s petroleum
Hopane detection in PM complex organic mixtures

Queens Winter 2002 Composite

Total Ion Current

Int. Std

17α(H),21β(H)-Hopane @46.73 min

Mass Spectrum

Quantitation ion: m/z 191
Molecular weight: m/z 412
$N$-Alkane carbon number and source provenance

Surface waxes from vegetation vs. gasoline, diesel fuels

Distribution of hydrocarbons versus carbon number

Mazurek and Simoneit, 1984
Carbon Preference Index (CPI) and source provenance for vegetation

\[ \text{Carbon Preference Index} = \frac{\sum \text{odd homologs}}{\sum \text{even homologs}} \]

**N-Alkanes & N-Alkanones**

\[ \text{Carbon Preference Index} = \frac{\sum \text{odd homologs}}{\sum \text{even homologs}} \]

**N-Fatty Acids & N-Alkanols**

\[ \text{Carbon Preference Index} = \frac{\sum \text{even homologs}}{\sum \text{odd homologs}} \]
Carbon Preference Index (CPI) & Carbon Number Distribution

CPI > 3 ➔ Significant contribution of recent biological material

CPI ~ 1 ➔ Significant contribution degraded material and fossil fuel compounds

Homolog Range and Carbon Number Maximum

<table>
<thead>
<tr>
<th>Source</th>
<th>Range</th>
<th>CMax</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bacterial, microbial</td>
<td>C15-C19</td>
<td>C16, C18</td>
</tr>
<tr>
<td>Petroleum hydrocarbons</td>
<td>C12-C35</td>
<td>C23-C24</td>
</tr>
<tr>
<td>Plant waxes</td>
<td>C22-C36</td>
<td>C27, C29</td>
</tr>
</tbody>
</table>

Mazurek and Simoneit, 1984
Molecular markers used for source apportionment of ambient PM

Simoneit et al. 1980, JAPCA 30, 387-390

Contamination of the Lake Tahoe Air Basin by High Molecular Weight Petroleum Residues

Bernd R. T. Simoneit, M. A. Mazurek
Institute of Geophysics and Planetary Physics
University of California, Los Angeles

T. A. Cahill
Air Quality Group, Crocker Nuclear Laboratory
University of California, Davis

The atmosphere of the Lake Tahoe air basin is contaminated with high molecular weight (> C12) petroleum hydrocarbons. Aerosol samples were collected by high-volume filtration and the solvent-soluble organic matter was analyzed. The relative concentrations of petroleum residues found were as follows: winter > summer and day > night. This contamination is primarily due to the poorer combustion of diesel and home heating fuels at that altitude and during periods of colder climate.
N-Alkane homolog distribution C20-C36

Figure 2. Distribution diagrams for n-alkanes (height of dotted lines indicate isoprenoids) as determined from GC/MS data for the Lake Tahoe aerosols. (a) Sample 1, Sugarpine Point State Park, night, summer; (b) sample 2, Sugarpine Point State Park, day, summer; (c) sample 6, Battle Creek Meadow Ranch; (d) sample 9, grass vegetation; (e) sample 3, Sugarpine Point State Park, winter; (f) sample 5, Sierra Ski Ranch, summer.

Simoneit et al., JAPCA, 30, 1980, 387-390
Source of organic PM traced to Southern California petroleum products

Lake Tahoe winter PM hopanes & steranes

Southern California Bight sediment hopanes & steranes

Figure 3. Relative distribution histogram for extended diterpanes and triterpanes (based on the m/z 191 mass chromatograms of the GC/MS data) for an example of Lake Tahoe aerosol and a sediment from the Southern California Bight which is contaminated by petroleum seepage. (a) Sample 3, Sugarpine Point State Park, winter; (b) Sample 193, seabed Santa Barbara Basin (reference 5). The R and S diastereomers are also indicated and C_{28} hopane I is 17α(H),18α(H),21β(H)-28,30-bisnorhopane.
Receptor modeling and CMB-MM

Professor Glen Cass
Chemical species mass balance

- Accounts for various forms of organic carbon
- Multiple analytical measurements
- Monitor variations in fine aerosol composition from bulk C to molecular organic C

Cass Group, eg., Rogge et al., Atmos. Env. 1993
Urban sources of fine carbonaceous particles

Los Angeles, CA fine particle emissions inventory

- Oil-fired boiler
- Autos (non- and catalyst-equipped)
- Heavy-duty diesel trucks
- Home heating furnace
- Home fireplace
- Roofing tar pots
- Road dust
- Tire dust
- Brake dust
- Cigarette smoke
- Meat cooking operations
- Vegetation (cultivated, native)

Mathematical Models Accounting for Individual Emission Contributions from Discrete Urban Sources
Fine particle diesel emissions

- Complex mixture distribution over GC FID elution time
- Homolog distributions characteristic of source n-alkanes: \( C_{\text{max}} \)
  - Ratio odd/even n-alkanes
- Histogram distribution facilitates incorporation into source apportionment model
PAH emissions profiles gasoline vs. diesel

Units = μg/km

Rogge et al., *ES&T* 1993
Urban vs. rural ....
Primary vs. secondary organics


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Downtown Los Angeles – Primary Urban Aerosol

Rubidoux Receptor Site (~ 70 miles downwind) – Secondary “Urban” Aerosol
Estimating source contribution

**Total C (EC+OC) : Organic Matter (1.4 X OC) : Elutable Organic Matter : Molecular Tracer**

Mass emission ratios

\[
\frac{\text{hopane concentration} \, \text{ng/m}^3}{\text{OC concentration} \, \mu\text{g/m}^3} = \text{ratio of source emission rates}
\]

Rogge et al., 1993 vehicle exhaust = 2.7X10^{-3}

Relative Source Contribution to Modeled OC Compounds

Ambient Concentrations

Diesel vehicles = 60%  Gasoline Vehicles = 30%
Paved road dust = 10%

Schauer et al., 1996
Relative source contribution to modeled OC compounds ambient concentrations

Mass balance at Downtown Los Angeles, 1982, Schauer et al., 1996
Urban sources of PM2.5 mass

- Chemical Mass Balance – Molecular Markers (CMB-MM)
- Requires detailed chemical analysis
- Mathematical modeling tool
- Linear combinations of source types
- Emissions inventories for major sources PM$_{2.5}$

“Source Apportionment of Airborne Particulate Matter Using Organic Compounds as Tracers”  

AE 30, 1996  


- Engineering approach receives 2001 Haagen-Smit Award
- Basis for current EPA Supersites PM2.5 molecular source apportionment program
- Employed currently Rutgers & NJ, NY, CT Organic PM2.5 & air sampling
Dominant molecular tracers of emission sources for particulate carbon....

~ 500 markers

Adapted from Simoneit, 2002; Oros and Simoneit, 2000

<table>
<thead>
<tr>
<th>Compound or compound class</th>
<th>Major source</th>
<th>Emission processa</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-Alkanes, C_{15-20} (odd/even)</td>
<td>Microbial</td>
<td>Direct/resuspension</td>
</tr>
<tr>
<td>C_{20-37} (odd/even)</td>
<td>Plant waxes</td>
<td>Direct/biomass burning</td>
</tr>
<tr>
<td>C_{15-37} (CPI=1)</td>
<td>Urban</td>
<td>Vehicle exhaust</td>
</tr>
<tr>
<td>n-Alkenes, C_{15-C_{37}}</td>
<td>Biomass/coal</td>
<td>Burning</td>
</tr>
<tr>
<td>n-Alkanones, C_{15-C_{35}}</td>
<td>Biomass/coal</td>
<td>Biodegradation/burning</td>
</tr>
<tr>
<td>n-Alkanals, C_{15-C_{35}}</td>
<td>Biomass/coal</td>
<td>Biodegradation/burning</td>
</tr>
<tr>
<td>n-Alkanoic acids, C_{15-C_{37}}</td>
<td>Microbial/biomass</td>
<td>Direct/resuspension/burning</td>
</tr>
<tr>
<td>C_{20-C_{26}}</td>
<td>Seed oils</td>
<td>Cooking/direct</td>
</tr>
<tr>
<td>C_{20-C_{26}}</td>
<td>Higher plants</td>
<td>Direct/burning</td>
</tr>
<tr>
<td>n-Alkanoic acid salts, C_{15-C_{20}}</td>
<td>Marine biomass</td>
<td>Sea slick resuspension</td>
</tr>
<tr>
<td>n-Alkanols, C_{14-C_{36}}</td>
<td>Biomass</td>
<td>Direct</td>
</tr>
<tr>
<td>n-Alkynitriles, C_{24-C_{30}}</td>
<td>Biomass/coal</td>
<td>Burning</td>
</tr>
<tr>
<td>Alkanedioic acids, C_{6-C_{28}}</td>
<td>Various</td>
<td>Photo-oxidation, combustion</td>
</tr>
<tr>
<td>Hydroxyacids, C_{2-C_{10}}</td>
<td>Various</td>
<td>Photo-oxidation</td>
</tr>
<tr>
<td>Oxoacids, C_{3-C_{10}}</td>
<td>Various</td>
<td>Photo-oxidation</td>
</tr>
<tr>
<td>Wax esters</td>
<td>Plant waxes</td>
<td>Biomass burning/direct</td>
</tr>
<tr>
<td>Triterpenyl alkanoates</td>
<td>Tropical vegetation</td>
<td>Biomass burning</td>
</tr>
<tr>
<td>Tiacylglycerides</td>
<td>Flora/fauna</td>
<td>Biomass burning/cooking</td>
</tr>
<tr>
<td>Methoxyphenols</td>
<td>Biomass with lignins</td>
<td>Burning</td>
</tr>
<tr>
<td>Levoglucosan (mannosan, galactosan)</td>
<td>Biomass with cellulose</td>
<td>Burning</td>
</tr>
<tr>
<td>Cholesterol</td>
<td>Urban/algae</td>
<td>Cooking/direct</td>
</tr>
<tr>
<td>Phytosterols</td>
<td>Higher plants</td>
<td>Burning/direct</td>
</tr>
<tr>
<td>Triterpenoids</td>
<td>Higher plants</td>
<td>Burning/direct</td>
</tr>
<tr>
<td>Diterpenoids (resin acids)</td>
<td>Higher plants (gymnosperms)</td>
<td>Burning/direct</td>
</tr>
<tr>
<td>Hopanes/steranes</td>
<td>Petroleum, coal</td>
<td>Urban (vehicular exhaust, motor oils, low temperature coal combustion)</td>
</tr>
<tr>
<td>Unresolved complex mixture (UCM)</td>
<td>Petroleum, coal</td>
<td>Urban (vehicular exhaust, motor oils, low temperature coal combustion)</td>
</tr>
<tr>
<td>Alkylpicenes/alkylchrysenes</td>
<td>Coal</td>
<td>Urban (burning/heating)</td>
</tr>
<tr>
<td>Polynuclear aromatic hydrocarbons (PAHs)</td>
<td>Ubiquitous</td>
<td>All pyrogenic processes</td>
</tr>
</tbody>
</table>
Sources of fine particles in NYC

Molecular markers as source indicators
fossil and modern carbon
Speciation of Organics for Apportionment of PM2.5 in the NY City Area (SOAP) \textbf{SOAP 2002-2003}

**Goals**

- Sources of fine carbonaceous particles
- Ambient concentrations TC, EC, OC
- Ambient concentrations molecular markers

\textbf{NY, NJ CT Fine Particulate Matter Study}

**Contributors:** Steve McDow, Min Li, Lee Alter, John Graham, Dirk Felton, Thomas McKenna, Charles Pietarinen, Alan Leston, Steve Bailey

Toll Plaza 13, NJ Turnpike
PM-2.5 Collection

SOAP 2002-2003 network field program

Queen’s College NY Supersite
Elizabeth, NJ
Chester, NJ
Westport, CT

Completed full annual cycle May 2002-2003 using Speciation Trends Network Schedule

400 successful ambient filters
Fine Particle Collection

Tisch 2 or 4 Channel Sampler

Quartz fiber filter collection substrate, 102mm

24 hr, 113 lpm

Sampling, transport, sample handling, and analytical procedures for ppt (10^{-12}) level organics
EC, OC and TC filter mass loadings (μg/cm²) SOAP network archived filters (~470 filters)

- EC, OC Archive 25%
- GCMS markers 50%
- LCMS Polar 25%

NYC Fine Particle Filters
# SOAP fine particle composites

## Identical days, 6-10 filters per composite

<table>
<thead>
<tr>
<th>Season</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>Early summer ‘02</td>
<td>Eliz, Qns, Chs</td>
</tr>
<tr>
<td>Summer ’02</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Early fall, ’02</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Fall, ’02</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Fall, ’02 precision</td>
<td>Eliz, Qns, Wpt, Chs(2)</td>
</tr>
<tr>
<td>Early winter, ’02-’03</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Winter, ’03</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Early spring, ’03</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Spring, ’03</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
<tr>
<td>Late spring, ’03</td>
<td>Eliz, Qns, Wpt, Chs</td>
</tr>
</tbody>
</table>
Analytical protocol GCMS markers

1. Spike Internal Standards
2. Five-point calibration standards
3. Soxhlet Extraction 250 ml organic solvent
4. Extract Condensation to 1.0 ml
5. Methylation agent

- Extract I 500 ul Derivatized
- Extract II 500 ul Derivatized w/BSTFA

- Extract III 500 ul Derivatized w/BSTFA
- GC/MS Analysis

- Extract I: Organic acids
- Extract II: Neutral compounds
- Extract III: Polar compounds
Molecular level instrumentation to detect & measure molecular markers

GCMS quadrupole – nonpolar compounds

LCMS ion trap – polar compounds
NYC area molecular markers

**Acids**

21 *n*-alkanoic acids
(with C_{10} to C_{30}) CPI noted above bar

10 aliphatic dicarboxylic acids
(C_{3} to C_{10})

1 aromatic polycarboxylic acid
cis-9-n-octadecenoic acid

**N-Alkanoic Acids (C10-C30) and Diacids (C3-C9)**

Composite 1: **Winter** Dec02, Jan03, Feb03 (6 filters)
Composite 2: **Spring** Mar03, Apr03 (10 filters)
N-Alkanoic acid distribution SOAP ’02-'03
$\mathcal{A}$-Alkanoic acid distribution other studies

Downtown L.A. (Annual Ave.)

Atlanta (Annual Ave.)

Philadelphia (Annual Ave.)

Houston (Summer)
SOAP network restaurant distribution

- **Fast Food Restaurant Locations**
  - > 110
  - 91 - 110
  - 71 - 90
  - 51 - 70
  - 31 - 50
  - 11 - 30
  - 3 - 10
  - SOAP Sampling Site

- **Asian + Chinese Restaurant Locations**
  - > 200
  - 151 - 200
  - 101 - 150
  - 71 - 100
  - 41 - 70
  - 11 - 40
  - 2 - 10
  - SOAP Sampling Site

- **Indian Restaurant Locations**
  - > 30
  - 21 - 30
  - 11 - 20
  - 0 - 10
  - SOAP Sampling Site

- **Mexican Restaurant Locations**
  - > 30
  - 21 - 30
  - 11 - 20
  - 0 - 10
  - SOAP Sampling Site
Investigate source(s) of extremely high CPIs for C12 to C30 $n$-alkanoic acids present in SOAP ambient fine particle
### Fats and oils screened in NYC area fine particle samples

<table>
<thead>
<tr>
<th>Cmpd Name</th>
<th>Molecular Weight</th>
<th>Formula</th>
<th>CAS Number</th>
<th>Cmpd Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-Hexanoic acid (Caproic, C6:0)</td>
<td>116</td>
<td>C6H12O2</td>
<td>142-62-1</td>
<td>6:0</td>
</tr>
<tr>
<td>n-Octanoic acid (= Caprylic acid, C8:0)</td>
<td>144</td>
<td>C8H16O2</td>
<td>124-07-2</td>
<td>8:0</td>
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<tr>
<td>n-Decanoic acid (= Capric acid, C10:0)</td>
<td>172</td>
<td>C10H20O2</td>
<td>334-48-5</td>
<td>10:0</td>
</tr>
<tr>
<td>n-Dodecanoic acid (= Lauric acid, C12:0)</td>
<td>200</td>
<td>C12H24O2</td>
<td>143-07-7</td>
<td>12:0</td>
</tr>
<tr>
<td>n-Tetradecanoic acid (= Myristic acid, C14:0)</td>
<td>228</td>
<td>C14H28O2</td>
<td>544-63-8</td>
<td>14:0</td>
</tr>
<tr>
<td>n-Hexadecanoic acid (= Palmitic acid, C16:0)</td>
<td>256</td>
<td>C16H32O2</td>
<td>57-10-3</td>
<td>16:0</td>
</tr>
<tr>
<td>n-Octadecanoic acid (= Stearic acid, C18:0)</td>
<td>298</td>
<td>C18H36O2</td>
<td>57-11-4</td>
<td>18:0</td>
</tr>
<tr>
<td>9-Octadecenoic acid (9Z) (= Oleic, C18:1)</td>
<td>296</td>
<td>C18H34O2</td>
<td>112-80-1</td>
<td>18:1</td>
</tr>
<tr>
<td>9,12-Octadecadienoic acid (= Linoleic, C18:2)</td>
<td>294</td>
<td>C18H32O2</td>
<td>60-33-3</td>
<td>18:2</td>
</tr>
<tr>
<td>9,12,15-Octadecatrienoic acid, (9Z,12Z,15Z) (= Linolenic, C18:3)</td>
<td>292</td>
<td>C18H30O2</td>
<td>463-40-1</td>
<td>18:3</td>
</tr>
<tr>
<td>n-Eicosanoic acid (= Arachidic acid, C20:0)</td>
<td>312</td>
<td>C20H40O2</td>
<td>506-30-9</td>
<td>20:0</td>
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<tr>
<td>Eicosenoic acid (C20:1)</td>
<td>310</td>
<td>C20H38O2</td>
<td>28933-89-3</td>
<td>20:1</td>
</tr>
<tr>
<td>n-Docosanoic acid (= Behenic acid, C22:0)</td>
<td>340</td>
<td>C22H44O2</td>
<td>112-85-6</td>
<td>22:0</td>
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<tr>
<td>n-Tetracosanoic acid (= Lignoceric acid, C24:0)</td>
<td>368</td>
<td>C24H48O2</td>
<td>557-59-5</td>
<td>24:0</td>
</tr>
</tbody>
</table>
Oil fatty acid distributions
Solid & commercial fats fatty acid distributions
Commercial cooking oil

- Fryolator oil
- Cottonseed/canola blend w/TBHQ & methyl silicone
- New vs. used
  - 50 hours operation at 350 °F
- 100 gallons/week
  - 14 gallons/unit
  - 3 meals/day
N-Alkanoic acid profiles and sources in SOAP fine PM
Updated emission profiles for light-duty diesel and gasoline-powered vehicles

Mass balance relationships for vehicle markers (hopanes and steranes) to EC and OC mass emission rates

NYS DEC Mobile Sources & Technology Development – Dr. Shida Tang
SOAP Motor vehicle marker to EC seasonal distribution

**Hopanes/EC (10^3)**

**Hopanes/OC (10^3)**
Organic complex mixtures diesel & gasoline powered vehicles
What do molecular markers tell us about the sources of organic carbon?

**Forensic tools for carbonaceous PM source contribution**

...MORE SOURCES AND PROFILES NEEDED

As homologous series distribution patterns

- Carbon number distribution, range & maximum
- Carbon Preference Index (odd to even; even to odd)
- Ratio resolved versus unresolved components

As individual compounds and within compound groups
  (PAHs, hopanes, steranes, fossil isoprenoids, simple sugars & carbohydrates)

**Essential input to Chemical Mass Balance-Molecular Marker Receptor Models**

**Field studies conducted over long-term produce insights and critical updates to emission inventories**
Acknowledgements

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Research Faculty:
Dr. Min Li

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Majad Ullah Mike Mankbadi
Joe Narcisco Gaurang Patel
Joe Savoly Aleksey Shinder
Andrew Muller Mohammed Burkari
Jesse Hansen
End of Presentation

Thank you

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