Prototype of the gas chromatograph–mass spectrometer to investigate volatile species in the lunar soil for the Luna-Resurs mission

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ABSTRACT
In preparation for the Russian Luna-Resurs mission we combined our compact time-of-flight mass spectrometer (TOF-MS) with a chemical pre-separation of the species by gas chromatography (GC). Coupled measurements with both instruments were successfully performed with the prototype of the mass spectrometer and a flight-like gas chromatograph. The system was tested with two test gas mixtures, a mixture of hydrocarbons and a mixture of noble gases. Due to its capability to record mass spectra over the full mass range at once with high sensitivity and a dynamic range of up to $10^{10}$ within 1 s, the TOF-MS system is a valuable extension of the GC analytical system. Based on the measurements with calibration gases performed with the combined GC–MS prototype and under assumption of mean characteristics for the Moon’s regolith, the detection limit for volatile species in a soil sample is estimated to $2 \cdot 10^{-10}$ by mass for hydrocarbons and $2 \cdot 10^{-9}$ by mass for noble gases.

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1. Introduction
To investigate the lunar poles in detail, the Russian Space Agency, Roskosmos, will launch the two spacecraft Luna-Glob and Luna-Resurs landing on these poles in 2015 and 2019, respectively. Since all lunar soil samples available on Earth originate from a restricted area at the lunar near side, close to the lunar equator, the investigation of the lunar poles is of high interest for extending our understanding of the Moon’s composition and history.

The detection and analysis of the volatile species in the lunar soil sample will be performed by a Gas Analytic Package (GAP), which includes three main instruments: the Thermal Analyser (TA-L), the Gas Chromatograph (GC-L), and the Neutral Gas Mass Spectrometer (NGMS). The GAP experiment works together with the robotic arm of the Luna-Resurs lander that delivers selected portions of regolith to the inlets of the TA-L instrument. Mechanics of the TA-L loads the sample into the Pyrolytic Cell (PC) and seals it for analysis. The PC provides programmed heating of the sample up to 1000 °C and all evolved gases are transferred by helium carrier gas via capillaries to the GC-L for chromatographic measurements. The exit of the GC-L is connected with NGMS through capillary tubing and the by GC-L separated gases are transferred by the carrier gas to the gas inlet of the mass spectrometer for further analysis.

As a part of this experiment our Neutral Gas Mass Spectrometer (NGMS) was selected to detect and analyse the volatile species in the lunar soil. The NGMS is a time-of-flight type mass spectrometer (TOF-MS) with a grid-less ion mirror (reflection) integrated in the ion path to enhance mass spectrometric performance (Scherer et al., 2006). A time-of-flight (TOF) mass spectrometer was chosen because of three significant advantages over sector magnetic instrument and quadrupole mass filters (e.g., see Young (2002)): (1) mass identification and mass resolution depend on time measurements that are technically easy to make, robust, and very accurate; (2) mass resolution does not depend on beam apertures (as in magnetic instruments), yielding intrinsically higher transmission and sensitivity; and (3) mass spectra can be collected rapidly (10,000 spectra per second in our case) with nearly 100% duty cycle because of the use of a storage ion source (Abplanalp et al., 2010). This leads to higher sensitivity than in magnetic and quadrupole instruments where mass spectra result from scanning an instrument parameter.

The ions are generated from the neutral gas by electron impact ionisation. The ion optical design of NGMS is based on the P-BACE...
instrument (Abplanalp et al., 2009). The ion optics of the prototype is integrated in a 325 mm long cylindrical tube with an inner diameter of 72.1 mm. In the detector micro-channel plates (MCP) are used to register the incoming ions. The instrument has a mass range of 1–1000 amu/q and a mass resolution of up to $m/\Delta m = 1100$. With a dynamic range of $10^6$ it is possible to measure species with a partial pressure down to $1 \cdot 10^{-16}$ mbar. A detailed description of the instrument currently under development for the lunar missions can be found in Wurz et al. (2012).

With NGMS, as a TOF-MS, complete mass spectra are recorded at once without the need for scanning over the desired mass range. The high cadence of recorded mass spectra allows the accumulation of mass spectra with a large dynamic range of up to $10^6$ within 1 s integration time. These qualities together with the high sensitivity are needed for the analysis of the gases eluted from a gas chromatographic (GC) column, which provides a pre-separation of the sample.

An additional feature of NGMS is that it can be operated as a standalone instrument for sampling the tenuous lunar exosphere (Wurz et al., 2007).

2. Experimental

2.1. GC–MS setup

The prototype of NGMS is the refurbished mass spectrometer of the P-BACE mission. On the one hand this mass spectrometer was modified to allow an interface with a gas chromatographic column. For this reason a capillary feed through was implemented directly behind the ion source, where a flow splitter reduces the amount of gas from the GC to a suitable pressure for the mass spectrometer. On the other hand some elements of the ion optics were replaced with prototype parts of the flight design for NGMS. To get significant measurements with the GC–MS prototype, spare units of the gas chromatographic instrument from the Phobos-Grunt mission were used. Two GC modules of identical build were available, the first one assembled with an MXT-5 (30 m × 0.25 mm × 0.25 μm, Restek) chromatographic column for separation of hydrocarbons and the second one with a Carbobond (30 m × 0.25 mm × 0.25 μm, Varian/Agilent) column for noble gas separation. Both modules are equipped with a Thermal Conductivity Detector (TCD), which allows, at first order, to check the flow of eluted gases into NGMS, and it also gives the opportunity to operate the GC in a stand-alone mode. Such GC modules have been developed in the frame of the MXT-5 column, using a relatively large sample with a 250 μl sample volume in the loop is then injected by the use of a standard sample valve (SV) in load position, gas chromatographic column (GC) with TCD reference cell (TCD RC) and measurement cell (TCD MC), frit-filters (FF), pressure gauge (PG), and NGMS inlet.

Fig. 1. Scheme of the laboratory GC–MS setup with sample gas (sample), helium carrier gas (He), sample valve (SV) in load position, gas chromatographic column (GC) with TCD reference cell (TCD RC) and measurement cell (TCD MC), frit-filters (FF), pressure gauge (PG), and NGMS inlet.

A sample loop with a defined volume is filled with the calibrated gas mixture to create a sample. In this study the two sample loop volumes of 250 μl and 25 μl were used. The entire sample volume in the loop is then injected by the use of a standard sample valve (cf. Fig. 1). The injected sample is carried by helium as the carrier gas first through the TCD reference cell, then through the gas chromatographic column and finally through the TCD measurement cell with a flow rate of approximately 2.3 ml/min. In this 30 m of capillary column the gas mixture in the sample is separated based on the selectivity of the stationary phase for the different species. At the GC outlet the gas is guided through a short piece of standard 1/32” stainless steel capillary at room temperature to the gas inlet interfacing the GC with the NGMS. Since only a fraction of the GC gas can be introduced into the mass spectrometer due to pressure reasons, this gas inlet consists of a flow splitter with an approximately 30 μm hole in diameter. Safe operation of NGMS (mainly given by the upper pressure limit of the MCPs in the detector) is guaranteed by the pressure reduction of the flow splitter while introducing as much as possible of the GC gas (approximately 1%) in the NGMS to be as sensitive as possible. The rest of the GC gas leaves the GC–MS instrument through the exhaust, which is simulated in the prototype by a roughing vacuum pump. The delicate hole in the gas inlet is protected against dust by two frit-filters, placed at the entrance and exit of the capillary tubing.

At least a pressure of a few 100 mbar is needed in the capillary to provide good flow conditions for sample transportation until the sample reaches the gas inlet of NGMS. Since the capillary outlet is connected to vacuum, a pressure gauge is installed in the capillary line for monitoring and characterising this pressure in the prototype setup (see Fig. 1).

The described setup is a prototype of the flight instrument, designed to be easily reconfigurable to try-out many different technical solutions. This flexibility limits the ability to control the background for which reason the instrument is suffering from increased electric noise and a mass spectrum is rich of peaks originated by the residual gas background.

2.2. Measurements

2.2.1. GC–MS analysis of hydrocarbons

The very first measurements with the lunar GC–MS prototype were performed with the chromatographic module equipped with the MXT-5 column, using a relatively large sample with a 250 μl sample loop. The first calibration gas mixture was made from a mixture, in pure helium, of CO₂ and a variety of hydrocarbons (n-butane, n-pentane, n-hexane, benzene and toluene), at 1000 ppm concentration per volume each. This mixture of hydrocarbons has been selected to test the system because they are easy to identify and light hydrocarbons are also what is expected to be measured on the Moon among other gases in case the source of lunar polar volatiles comes from meteoritic impacts (Gerasimov, 2002) or volcanic eruptions. The presence of some simple organic molecules (CH₄, C₂H₆, and CH₃OH) in the plume of ejected material from the impact at the South Polar Region was detected by LCROSS during the Centaur rocket impact into Cabeus crater (Colaprete et al., 2010). After sample injection into the GC column NGMS recorded one mass spectrum after the other, each with 1 s integration time at 10 kHz extraction frequency. This means each spectrum is a histogram of 10,000 single mass spectrometric measurements.

Fig. 2 shows TCD data over the full retention time span, where the hydrocarbons are well separated. The start of the chromatogram is given by the negative peak. At this time the sample passes the TCD reference cell and the actual measurement cell is filled with almost pure helium, causing a negative peak. The feature immediately after the injection appears due to pressure disturbances introduced during operation of the sampling valve. A water peak is also indicated in this chromatogram, but its
identification was only possible with the help of the mass spectrometer, as will be explained below.

The mass spectrometric analysis of the GC peaks with NGMS is shown in Fig. 3. Due to the fragmentation, caused by electron impact ionisation, several peaks of one compound are available in each mass spectrum. By selecting and summing up the most favourable fragments of each compound, the signal-to-noise ratio can be maximised as it is done in Fig. 3. The summing includes for CO₂ only the parent molecule at mass 44; for water again only the parent molecule at mass 18; for butane the parent molecule at mass 58; for pentane the parent molecule on mass 72; for hexane the fragments on the masses 56 and 57; for benzene the fragments on the masses 74, 77, 78 and 79; and for toluene the fragments on the masses 63, 91 and 92. Using the same composite signal gives

![Fig. 2. TCD data of a GC–MS analysis of the hydrocarbon mixture sample. The mass spectrometric analysis of this sample is shown in Fig. 3.](image)

![Fig. 3. Mass spectrometric analysis of the peaks at GC output by NGMS. Immediately after the CO₂ peak (top left panel) appears a small peak. Since butane has a fragment on the same mass line as the CO₂ parent peak, this small peak appears in the CO₂ panel due to the small difference in retention time.](image)

<table>
<thead>
<tr>
<th>Event</th>
<th>c [ppm]</th>
<th>t_{ret} [s]</th>
<th>S/N (NGMS)</th>
<th>S/N (TCD)</th>
<th>FWHM (NGMS) [s]</th>
<th>FWHM (TCD) [s]</th>
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<td>317</td>
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<td>99</td>
<td>32</td>
<td>36</td>
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<td>102</td>
<td>863</td>
<td>51</td>
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<td>1.5</td>
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<td>1037</td>
<td>45</td>
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<td>1.5</td>
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<td>138</td>
<td>2494</td>
<td>34</td>
<td>1.1</td>
<td>1.7</td>
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<tr>
<td>Benzene</td>
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<td>174</td>
<td>818</td>
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<td>1.9</td>
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<tr>
<td>Toluene</td>
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<td>297</td>
<td>518</td>
<td>9</td>
<td>2.8</td>
<td>2.8</td>
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the signal-to-noise values in Table 1 for each GC peak visible on TCD (Fig. 2). Here the fragments of each compound were used to increase the signal-to-noise values. In case of unknown samples all these peaks at an identical retention time will also be used to identify the measured species, since the fragmentation pattern is characteristic to the molecular structure.

According to the retention times determined from these measurements, Table 1 shows clearly that with a helium flow of 2.3 ml/min and a column temperature of only 40 °C a good separation of all the hydrocarbon compounds can be achieved and that the GC–MS analysis with the available organic compounds needs approximately 5 min. Since the TCD and the gas inlet of NGMS are close together, the difference in retention time is less than a second and therefore the retention times in Table 1 are valid for both the TCD and the NGMS. The results also show the value of having NGMS analysing the columns output, since the achievable signal-to-noise ratio of a GC peak measured with NGMS is a few decades better than with TCD alone. The low signal-to-noise ratios of the TCD can be explained with the electronic noise which is present because of the prototype status of the system. It is known that a TCD can provide signals of clearly better quality. Unfortunately, also the performance of NGMS is affected by the electronic noise of the prototype environment. Of course, for the flight system the electronics grounding scheme will be optimised and EMC shielding will be applied where necessary.

Due to its higher affinity with the column coating toluene spends the longest time in the column and gets therefore a broader peak shape with smaller amplitude. This leads to a much lower signal-to-noise ratio for TCD and NGMS, but for the latter, the signal-to-noise ratio remains high and the peak is easy to analyse. The analysis of toluene could be improved using a temperature ramp for the gas chromatographic column instead of the fixed temperature ramp for the GC, and EMC shielding will be applied where necessary.

The big advantage of GC–MS coupling is that also species can be detected on a mass line where relatively high background is present in the mass spectra. An evidence for this is the water peak in Fig. 2. Initially it was unknown to which compound the corresponding peak in TCD data was belonging, but the analysis of the mass spectra recorded with NGMS showed clear features of the water GC peak. While no measurements are performed the output of the sample loop is at air pressure and therefore also exposed to the water in the air. Even if the unheated sample loop is purged before injecting the volume of the sample loop to the column it is very likely that the calibration gas mixture is contaminated by a small portion of water.

The concentration for water was estimated by comparing the known GC peak of CO₂ to the unknown water peak. Calculating the area of both the H₂O⁺ and CO₂⁺ peaks and taking into account the electron impact ionisation cross sections of both molecules (Itikawa, 2002; Rao et al., 1995) leads to the estimated water concentration in Table 1.

The values for the peak width in time (full width at half-maximum) presented in Table 1 show that the short transfer line between the GC output and the gas inlet to NGMS does not affect the peak shape much for most of the peaks, except for water, where the peak shape is much broader than measured with TCD. Water is one of the most polar compounds; thus it is possible that some adsorption occurred in the transfer line. Generally it seems that peaks are slightly broader measured with NGMS, but this might be also because mass spectra were accumulated for 1 s each: more data points per GC peak would help to detect the peak shape better. As shown in the next section, this can be done easily with NGMS.

Different measurements with this column showed that even with a reduction by a factor of ten each of sample loop volume (25 μl instead of 250 μl) and calibration gas mixture concentration...
NGMS is able to detect unambiguous signals of all the hydrocarbon species. As an example, Fig. 4 shows the response of butane in the mass spectrum recorded with NGMS of such a sample. At these small levels for each substance the parent peak, as well as fragments, are still visible. Measuring the signal of the parent peak of each substance is important to identify a substance and is also included in the derivation of the signal-to-noise ratios (Table 2).

To calculate the detection limit for the prototype of the GC–MS system we can derive from the signal-to-noise ratio that detection at 10 ppm is possible with NGMS (see Fig. 4). Further assuming a mean molar mass of 22 g mol\(^{-1}\) and a density of 2 g cm\(^{-3}\) for Moon’s regolith (Wurz et al., 2007; Fernandes and Wurz, 2012) and an oven...
volume of 500 mm$^3$ for the Lunar missions, a detection limit for hydrocarbons of about $2 \cdot 10^{-10}$ by mass can be calculated for the GC–MS prototype setup. For comparison, for the SAM instrument on the Curiosity rover a sensitivity for organic compounds of $6 \cdot 10^{-10}$ by mass has been quoted (Mahaffy et al., 2012).

### 2.2.2. GC–MS analysis of noble gases

The first measurements with the Carbobond GC module were done with a 250 µl sample loop and a noble gas mixture containing each 1000 ppm of neon, krypton and xenon in pure helium. Typically NGMS recorded continuous mass spectra of the GC gas with 1 s integration time and 10 kHz extraction frequency (see Figs. 5 and 6).

As done in the previous section with the fragments of the hydrocarbons due to electron impact ionisation, the signal-to-noise ratio can also be increased for the noble gases by using the signal of different isotopes. The left panel in Fig. 5 shows the temporal evolution of the krypton peak during a GC–MS analysis for all natural isotopes. Summing up the signal of the most favourable isotopes in terms of maximising the signal-to-noise ratio can also lead to the composite signal shown in the right panel in the same figure. Fig. 6 shows the same approach for each component in the sample and in Table 3 the resulting values of these measurements can be found. For the noble gases the summing includes for neon the isotopes of the masses 20 and 22; for water the fragments of the masses 17 and 18; for krypton the isotopes of the masses 83, 84 and 86; and for xenon the isotopes of the masses 129, 131, 132, 134 and 136.

With the same operation parameters as used for the MXT-5 column (40 °C column temperature, 2.3 ml/min helium flow) a separation of all components out of the noble gas mixture is achieved. Again NGMS is able to detect the species with a much better signal-to-noise ratio than the TCD does. As described in the previous section the signal-to-noise values of the TCD, as well as NGMS, are affected by electronic noise and the mass spectral background of the prototype setup.

Similar to the water peak in the previous section, the concentration in Table 3 was estimated by comparing the GC peak of H$_2$O$^+$ with the known Ne$^+$ peaks and taking the electron impact ionisation cross sections of both species (Rejoub et al., 2002; Rao et al., 1995) into account. In this measurement the water peak is a factor 10 less intense than for the analysis in Table 1, barely above the water background in the mass spectrometer.

Conspicuous is the very long retention time of xenon, which leads to a broad GC peak with small amplitude (cf. Fig. 6), similar to toluene in the hydrocarbon analysis (Table 1). Due to this behaviour the signal-to-noise ratio is smaller and the peak is close to the TCD detection limit, despite the relatively large sample volume injected in the column. That xenon shows a different behaviour, compared to the other noble gases, is no surprise, since its adsorptive properties are well known (see Modak and Pagni (1973)).

By increasing the isothermal column temperature from 40 to 80 °C the situation for xenon can be improved: the retention time of xenon is nearly 5 min shorter and the signal-to-noise ratio can be improved due to a narrower and therefore higher GC peak. While xenon needs at the higher column temperature nearly 5 min less time to pass the chromatographic column, the retention times of the remaining noble gases are shortened only by a few seconds.

Depending on the temporal width of a GC peak the typical continuous acquisition of mass spectra with 1 s integration time might not lead to an optimal sampling of the shape of the GC peak. This problem can be avoided by changing the integration time of a single mass spectrum. With NGMS the integration time per spectrum can be adjusted between 100 ms and 1 s. Fig. 7 shows a comparison between two different sampling rates. In both cases the sample was injected under identical conditions with the only difference of the sampling rate of NGMS. The left panel shows the neon peak sampled with 1 s mass spectra while the right panel shows the same peak sampled with 200 ms mass spectra. At our extraction frequency of 10 kHz these integration times correspond to a histogram of 10,000 and 2000 extractions, respectively, per data point. Naturally the signal-to-noise value decreases with shorter integration times. The optimal GC sampling rate will be a trade-off between the correct peak shape measurement and a sufficient signal-to-noise ratio. For an accurate analysis of the abundance at least 10 samples per GC peak are required. Since the GC peaks with a large retention time are

<table>
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<th>Event</th>
<th>c [ppm]</th>
<th>$t_{res}$ [s]</th>
<th>S/N (NGMS)</th>
<th>S/N (TCD)</th>
<th>FWHM (NGMS) [s]</th>
<th>FWHM (TCD) [s]</th>
</tr>
</thead>
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<tr>
<td>Neon</td>
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<td>72</td>
<td>487</td>
<td>12</td>
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</tr>
<tr>
<td>Water</td>
<td>~85</td>
<td>76</td>
<td>6</td>
<td>–</td>
<td>3.3</td>
<td>–</td>
</tr>
<tr>
<td>Krypton</td>
<td>1000</td>
<td>100</td>
<td>1217</td>
<td>24</td>
<td>3.2</td>
<td>3.5</td>
</tr>
<tr>
<td>Xenon</td>
<td>1000</td>
<td>439</td>
<td>593</td>
<td>8</td>
<td>12.1</td>
<td>11.7</td>
</tr>
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</table>

Fig. 7. Comparison between sampling of a GC peak with 1 s (left panel) and 200 ms (right panel) integration time per mass spectrum.
broader than those with a short retention time, there is no single sampling rate, which is ideal over the full retention time span. Therefore the NGMS flight instrument will have the capability to switch the integration time to a larger value as soon as the higher sampling rate is no longer used.

The top panel of the left column in Fig. 8 shows a GC–MS analysis of the neon peak using the 1000 ppm noble gas mixture described above together with a 250 µl sample loop, while the measurement shown in the top panel of the right column uses the same gas mixture, but with a smaller sample loop of 25 µl. Since the diameter of the column is in both cases the same, the larger sample is in the column geometrically longer and needs therefore more time to pass the gas inlet to NGMS. Due to this reason the peaks of the larger sample are broader compared to the same peaks with the smaller sample volume. The slight shift in retention time between the two sample volumes occurs due to an overload of the stationary phase of the gas chromatographic column with the larger sample volume (left column in Fig. 8).

Based on the measurements presented in Fig. 8 it can be derived that the sample concentration could be lowered again by a decade to reach the detection limit of NGMS. Using the same approach as described in the previous section for the hydrocarbons a detection limit for noble gases of about $2 \cdot 10^{-8}$ by mass can be estimated. Since the cross section for electron impact ionisation is about one decade lower for noble gases compared to hydrocarbons (Rejoub et al., 2002; Sueoka et al., 2005), the one decade higher detection limit for noble gases can be explained.

3. Conclusion

Coupled measurements with chemical pre-separation of the gaseous sample by gas chromatography and subsequent analysis
by our time-of-flight type Neutral Gas Mass Spectrometer, NGMS, were performed successfully with a prototype instrument for the Luna-Resurs mission. Both a mixture of hydrocarbons and a noble gas mixture were tested successfully.

While a GC–MS measurement is running, NGMS is able to record continuously mass spectra of the GC output over the full mass range to maximise the possible chemical information from the sample. In GC–MS mode the integration time per mass spectrum and therewith the sampling frequency of the GC output by NGMS can be chosen between 100 ms and 1 s. This gives the possibility to choose a shorter integration time in the beginning of the chromatogram to sample the less retained and therefore narrower GC peaks with a sufficient amount of data points. Due to the high sensitivity and the large dynamic range of the mass spectrometer of up to $10^6$, we can detect volatiles of about $2 \cdot 10^{-10}$ by mass in the soil sample for hydrocarbons and $2 \cdot 10^{-9}$ by mass for noble gases with the prototype setup of the GC–MS instrument.

For comparison, a sensitivity of $6 \cdot 10^{-10}$ by mass for organic compounds has been quoted for the SAM flight instrument on the Curiosity rover (Mahaffy et al., 2012).

Due to the successful measurements with the NGMS prototype the development of the flight design for NGMS was accomplished and currently the manufacturing of flight hardware is in progress to be ready for flying to the Moon.

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References


