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Technical note: ^{21}Ne in the CoQtz-N quartz standard material

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Abstract. Intercomparison standards produced from mineral samples exposed to the natural cosmic-ray flux are routinely used for interlaboratory comparison and normalization of measurements of the cosmogenic noble gases ^{3}He and ^{21}Ne. This effort is facilitated by availability of multiple standard materials with a wide range of cosmogenic-nuclide concentrations. The 'CoQtz-N' quartz standard, which was originally produced and distributed as an intercomparison standard for cosmogenic ^{10}Be and ^{26}Al, has a relatively low cosmogenic-nuclide concentration compared to other mineral standards used for cosmogenic noble gas analysis, and is therefore potentially useful for assessing linearity of interlaboratory offsets across a wide range of concentrations. This paper reports ^{21}Ne analysis of 13 aliquots of CoQtz-N, interspersed with 14 aliquots of the commonly used CRONUS-A quartz standard, in three analytical sessions on two noble gas mass spectrometer systems. The excess ^{21}Ne concentration in CoQtz-N, normalized to the accepted value of 320 × 10^{6} atoms g^{-1} for CRONUS-A, is 13.87 ± 0.24 × 10^{6} atoms g^{-1}.

1 Intercomparison standards for cosmogenic nuclides produced in quartz

Several cosmic-ray-produced nuclides, most importantly ^{10}Be, ^{14}C, ^{26}Al, and ^{21}Ne, are produced in the common mineral quartz when it is exposed to the cosmic-ray flux at or near the Earth's surface, and concentrations of these nuclides are routinely measured for various applications in geochronology and Earth surface process studies. In an effort to improve accuracy, precision, and reproducibility of such measurements, a number of homogenized quartz standard materials have been produced and distributed among laboratories active in this research area. These materials are naturally occurring samples without any independent knowledge of their irradiation histories, so are not primary or certified concentration standards, but are mainly intended for interlaboratory comparison. Quartz intercomparison standards that have been distributed include the CRONUS-A and CRONUS-N standards, which are described by Jull et al. (2015) and originally intended for interlaboratory comparison of ^{10}Be, ^{14}C, and ^{26}Al measurements, the CREU-1 standard described by Vermeesch et al. (2015) and intended for ^{21}Ne intercomparison measurements, and the CoQtz-N standard described by Binnie et al. (2019) and intended for ^{10}Be and ^{26}Al intercomparison. CRONUS-A has also been used for interlaboratory comparison of ^{21}Ne measurements (Vermeesch et al., 2015; Balco et al., 2019).

Interlaboratory comparison of cosmogenic noble gas measurements by means of these standard materials, both for ^{21}Ne using CREU-1 and CRONUS-A and for ^{3}He using the CRONUS-P pyroxene standard (Schaefer et al., 2016; Blard et al., 2015) has been valuable in revealing systematic offsets among ^{21}Ne and ^{3}He measurements performed at different

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laboratories and on different analytical systems (e.g., Balco et al., 2019; Balter-Kennedy et al., 2023) These differences often exceed nominal absolute concentration uncertainties claimed by the various laboratories. Some data indicating that inter-system/interlaboratory offsets are constant across multiple samples and a wide range of ^{21}Ne/^{3}He concentrations (Balco et al., 2019; Balter-Kennedy et al., 2023) imply that the offsets are most likely the result of undetected inaccuracies in pressure/volume calibrations used to prepare primary gas standards. This, in turn, implies that ^{21}Ne and ^{3}He measurements can be standardized among laboratories by applying constant normalization factors derived from analysis of mineral intercomparison standards. Even if the true concentration of an intercomparison standard is not known from first principles, standardization to a common value reconciles measurements from different laboratories used for production rate calibration and exposure-age determination, thus improving the accuracy and precision of exposure-dating and other surface-process-related applications.

Verifying that interlaboratory offsets are not concentration- or sample-dependent, of course, requires multiple intercomparison standards with a wide range of cosmogenic noble gas concentrations. This paper reports ^{21}Ne measurements on the CoQtz-N standard, which has significantly lower cosmogenic-nuclide concentrations than CRONUS-A and CREU-1, to facilitate its use for interlaboratory comparison for ^{21}Ne as well as ^{10}Be and ^{26}Al as originally intended. The CoQtz-N standard was prepared by Binnie et al. (2019) from a vein quartz boulder exposed in a lag deposit near the locality of Hakos, Namibia, and has ^{10}Be and ^{26}Al concentrations of 2.5×10^{6} and 15.6×10^{6} atoms g^{-1}, respectively. Although the precise location at which the sample was collected was not reported, this locality is near 23.25° S latitude and at approximately 1850 m elevation. Given assumptions of steady-state erosion, this estimated location, and a sample thickness of 15 cm estimated from Figure 1 of Binnie et al. (2019), the ^{10}Be and ^{26}Al concentrations imply an erosion rate near 2.2 m Myr^{-1} and an expected cosmogenic ^{21}Ne concentration near 12×10^{6} atoms g^{-1}. As the CRONUS-A and CREU-1 ^{21}Ne intercomparison standards have extremely high ^{21}Ne concentrations (320 and 348 $\times 10^{6}$ atoms g^{-1}, respectively), CoQtz-N is potentially useful for interlaboratory comparison at less extreme concentrations.

2 Analytical methods

The data reported here consist of measurements of Ne isotope concentrations in the CRONUS-A and CoQtz-N quartz standards, measured in three analytical sessions on two noble gas mass spectrometers and associated extraction systems at the Berkeley Geochronology Center (BGC) and the Lawrence Livermore National Laboratory Noble Gas Lab (LLNL), such that multiple aliquots of both standards were analyzed during the same analytical sessions (Figure 1). Here 'analytical session' means a continuous period of measurements during which the mass spectrometer tuning and extraction and analysis procedure were not changed.

^{21}Ne measurements at BGC were carried out on the BGC "Ohio" mass spectrometer and associated extraction line as described in a number of studies (e.g., Balco et al., 2019; Balter-Kennedy et al., 2020; Bergelin et al., 2022). 75-150 mg of quartz is encapsulated in a crimped segment of Ta tubing, which is placed under vacuum and heated with an 810 nm, 150 W diode laser coupled to a coaxial optical pyrometer in a control loop that allows adjusting the laser power to

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achieve a fixed pyrometer temperature. At BGC, all samples described here were analyzed in two heating steps at 850° C and 1100° C. Gases released during heating are reacted with cold SAES getters and remaining noble gases are frozen to an activated charcoal cold trap at 33 K. Helium is pumped away and the cold trap is warmed to 75 K to release Ne into a MAP-215-50 mass spectrometer originally constructed in 1992 but modernized with updated detectors and counting electronics. All Ne isotopes, as well as other masses necessary for isobar corrections on masses 20 and 22, are measured on a single Channeltron-type electron multiplier operated in ion-counting mode. As this mass spectrometer cannot resolve ^{20}Ne^{+} and ^{40}Ar^{++} on mass 20, correction for ^{40}Ar^{++} is accomplished using a ^{39}Ar spike that allows monitoring of the Ar^{++}/Ar^{+} ratio during analysis, as described in Balco and Shuster (2009). Correction for CO_2^{++} on mass 22, as also described in that reference, is accomplished by obtaining a relationship between Ar and CO_2 charge ratios in offline experiments. No ^{20}NeH contribution on mass 21 can be detected on this instrument under typical operating conditions. Ne isotope signals after isobar correction are further corrected for "cold" blanks measured without any introduction of sample to the extraction line; cold blanks are $<$ 1% of observed signals. The HF/$H_2^{18}O$ interferences are not resolvable on this instrument and are therefore included in the cold blank correction; they are most likely negligible under normal conditions. "Hot" blanks in which empty Ta packets are heated as if they were samples released small amounts of Ne whose isotope composition was indistinguishable from atmosphere. As the aim is eventually to calculate excess ^{21}Ne in relation to atmosphere, a separate hot blank correction is therefore not made. Calibration of Ne isotope signals to absolute amounts is by peak height comparison with an air standard, originally calibrated by pressure/volume measurements using a MKS Baratron pressure transducer, which is analyzed several times daily. By varying the amount of the air standard delivered, the machine response was found to be linear in the pressure range encompassing the samples analyzed here. Corrections for mass discrimination also utilize the air standard.

^{21}Ne measurements at LLNL utilized an effectively identical laser heating system coupled to the LLNL "NG2" extraction line and mass spectrometer system. The sample chamber used for laser heating has a slightly different internal configuration which is less effective at preventing cross-heating of adjacent samples than the BGC chamber. Thus, in the BGC system, samples could be analyzed in two heating steps at 850°and 1100 °C without risk of gas loss from adjacent samples by cross-heating, but the LLNL system required analysis in three heating steps at 450°, 850°, and 1100°. Gases released by heating are reacted with SAES getters, noble gases are trapped on activated charcoal at 33 K, and neon is released into the mass spectrometer at 70 K. The difference in release temperatures between BGC and LLNL procedures is because the two charcoal traps were found to have slightly different noble gas adsorbption characteristics. The LLNL-NG2 mass spectrometer is a Nu Instruments 'Noblesse HR' with five switchable Faraday/multiplier detectors at a spacing nominally intended for multicollection of five Ar isotopes, but also permitting simultaneous measurement of ^{20}Ne, ^{21}Ne, and ^{22}Ne. This instrument is capable of edge resolution of ^{20}Ne^{+} and ^{40}Ar^{++} separately (see Saxton, 2020). Thus, the measurement sequence consists of (i) simultaneous measurement of the combined ^{22}Ne^{+}/CO_2^{++} peak on mass 22, the ^{21}Ne peak on mass 21, and combined ^{20}Ne^{+}/HF on mass 20; (ii) the edge-resolved HF peak flat on the high side of the ^{20}Ne^{+} peak; and (iii) CO_2^{+} on mass 44. A corrected ^{20}Ne signal is obtained by subtracting the HF signal from the combined mass 20 signal; the HF contribution is typically < 0.1% and therefore effectively negligible, but

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is monitored in any case because quartz samples are commonly prepared by HF etching, so a possibility exists that combustion of fluorides could lead to a significant HF interference (however, none was observed for these samples). A corrected ^{22}Ne signal is then obtained using a CO_2^{++}/CO_2^{+} ratio measured offline between analyses; the charge ratio for this instrument during these measurements was found to be in the range 0.0155 - 0.0157. No ^{20}NeH contribution on mass 21 could be observed under these operating conditions, and discussion in Saxton (2020) indicates that it is likely negligible. Corrections for mass discrimination and detector gains utilize an air standard containing a similar amount of Ne as typical samples and analyzed several times daily. Results of cold and hot blank analyses on the LLNL system were equivalent to the results described above for the BGC system and blank corrections were carried out in the same way.

Calibration of Ne isotope signals to absolute abundances on the LLNL system also utilizes the air standard. At the beginning of this work the air standard on the LLNL system was known to contain approximately the same amount of atmosphere as the BGC air standard, but had not been precisely calibrated. For purposes of cosmogenic ^{21}Ne measurements, therefore, a precise calibration was established by analyzing 11 aliquots of the CRONUS-A standard (see Results section below) and assigning the absolute Ne abundance in the standard such that the mean excess ^{21}Ne concentration in all aliquots was equal to the accepted value of 320 $\times$ 10^6 atoms g^{-1} (Vermeesch et al., 2015). Aliquots of air and CRONUS-A of various sizes were then analyzed to verify that the sensitivity was linear over the relevant pressure range. Although the procedure of calibrating a gas standard from a mineral standard that is not a primary or certified standard has not routinely been used in noble gas analysis, it is effectively equivalent to the more common practice of establishing the Ne abundance in the gas standard from first principles pressure/volume measurements and then normalizing subsequent ^{21}Ne measurements to an accepted value for CRONUS-A. In fact, as the results below indicate that excess ^{21}Ne in CRONUS-A can be routinely measured to $\sim$ 1% precision, the 8% overdispersion observed in the intercomparison results of Vermeesch et al. (2015), among systems calibrated against first-principles gas standards, suggests that calibrating a gas standard against CRONUS-A measurements may be more precise, if not more accurate, than calibrating based on first principles.

For all analyses excess ^{21}Ne is then calculated as $N_{21,xs} = N_{21,tot} - N_{20,tot}R_{2120a}$, where $N_{21,xs}$ is the excess ^{21}Ne concentration (atoms g^{-1}), $N_{20,tot}$ and $N_{21,tot}$ are the total measured concentrations of ^{20}Ne and ^{21}Ne (atoms g^{-1}), and R_{2120a} is the atmospheric ^{21}Ne/^{20}Ne ratio (0.002959; see Eberhardt et al., 1965). Excess ^{21}Ne concentrations in all heating steps for each sample are then summed (see further discussion below). Reported uncertainties for both BGC and LLNL measurements include (i) counting uncertainties on all masses, fully propagated through all detector gain, mass discrimination, and isobaric interference corrections, and (ii) an uncertainty in machine sensitivity derived from the reproducibility of the air standards. For both systems the dominant uncertainty is the reproducibility of the ^{21}Ne signal in the air standard, which is approximately 1.5% for the BGC system and 0.8% at LLNL. For CRONUS-A, this results in total calculated uncertainties on the excess ^{21}Ne concentration of approximately 1.8% for BGC measurements and 1% for LLNL measurements. As machine sensitivities are similar on both instruments, the improved uncertainties on the LLNL system are mostly attributable to improved stability and therefore reproducibility of the standards, which in turn is most likely because the electronics on the Nu Noblesse are 30 years younger than those on the MAP-215. The improvement

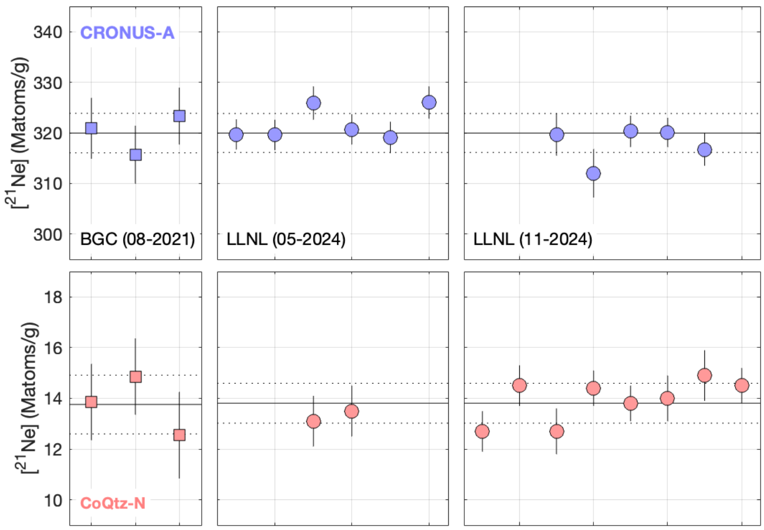

Figure 1. Excess ^{21}Ne concentrations in CRONUS-A and CoQtz-N standards, measured in three analytical sessions at BGC (Aug 2021) and LLNL (Apr 2024, Nov 2024). Data are shown in order of measurement, although multiple heating steps alternated for sets of samples run in the same batch. Error bars are 68% confidence. The solid and dotted black lines indicate means and standard deviations of BGC and LLNL measurements.

in counting statistics due to multicollection and the simplified isobar corrections due to higher instrument resolution make minor contributions.

3 Results and discussion

Three measurements of the excess ^{21}Ne concentration in CRONUS-A at BGC had a mean and standard deviation of $318.7 \pm 3.9 \times 10^6$ atoms g^{-1}, which is indistinguishable from the accepted value of 320, so the BGC measurements were not renormalized (Figure 1; Supplementary Table S1). The three aliquots of CoQtz-N measured at BGC had excess ^{21}Ne concentrations with mean and standard deviation of $13.7 \pm 1.2 \times 10^6$ atoms g^{-1}. Eleven aliquots of CRONUS-A measured at LLNL, as noted, were used for calibration of the air standard, so had excess ^{21}Ne concentrations with mean and standard deviation of $320.0 \pm 3.8 \times 10^6$ atoms g^{-1}. Ten aliquots of CoQtz-N measured at LLNL had mean and standard deviation of $13.81 \pm 0.80 \times 10^6$ atoms g^{-1}. Thus, CoQtz-N measurements at BGC and LLNL agree. The 13 measurements together have a MSWD of 0.80, an error-weighted mean of 13.87×10^6 atoms g^{-1}, and standard error of 0.24×10^6 atoms g^{-1}.

Ne isotope ratios for all heating steps of CoQtz-N are indistinguishable from the cosmogenic-atmospheric mixing line (Figure 2). Low-temperature heating steps are enriched in cosmogenic neon relative to higher-temperature steps, as is

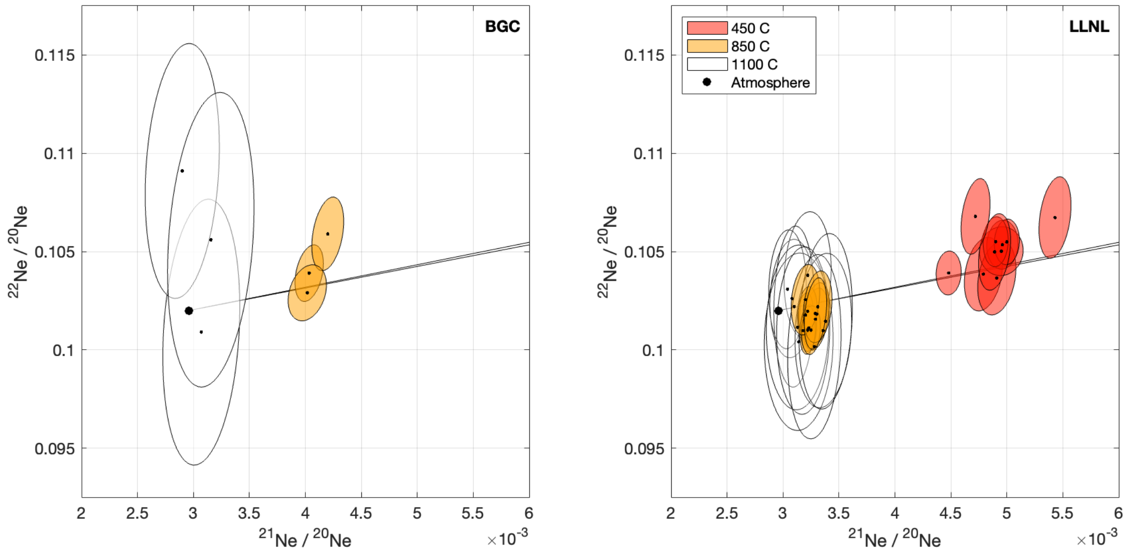

Figure 2. Neon three-isotope plot for step-heating analyses of CoQtz-N at BGC and LLNL. The black dot is the composition of atmospheric neon and the black line is the atmospheric-cosmogenic mixing line. Ellipses are 68% confidence regions.

typical for most quartz samples. High-temperature steps have isotope composition indistinguishable from atmosphere when each analysis is considered individually (Fig. 2), but all except one of the high-temperature steps have ^{21}Ne/^{20}Ne ratios slightly above atmosphere, which would be statistically improbable if in fact only atmospheric Ne was present. Thus, high-temperature steps contain a nonzero amount of excess ^{21}Ne and are included in calculated total excess ^{21}Ne for each sample. Atmospheric Ne concentrations in CoQtz-N quartz, expressed as ^{21}Ne, are 35-50 $\times$ 10^6 atoms g^{-1} (Supplementary Table S1), so excess ^{21}Ne is about 25% of total ^{21}Ne. Although the fact that most of the ^{21}Ne in CoQtz-N is atmospheric limits the precision with which the excess ^{21}Ne concentration can be measured, measurements using a modern mass spectrometer had individual uncertainties in the 5-6% range and an aggregate uncertainty on the full set of measurements near 2%, which is adequate for monitoring linearity of laboratory offsets across a range of ^{21}Ne concentrations.

Although it is not possible to discern from Ne isotope ratios alone whether measured excess ^{21}Ne in CoQtz-N is all cosmogenic or may include a nucleogenic component, comparison with ^{26}Al and ^{10}Be concentrations from Binnie et al. (2019) provides some insight into this. At the estimated sample location discussed above, ^{26}Al and ^{10}Be concentrations lie on the steady erosion line at an erosion rate near 2.2 m Myr^{-1} (Figure 3). The total excess ^{21}Ne concentration, however, implies a lower erosion rate, so ^{26}Al/^{21}Ne and ^{10}Be/^{21}Ne pairs lie below the simple exposure region. This indicates that some portion of the total excess ^{21}Ne concentration is either nucleogenic or inherited from a prior period of exposure. Assuming that 2 $\times$ 10^6 atoms g^{-1} of the total excess ^{21}Ne concentration is either nucleogenic or otherwise

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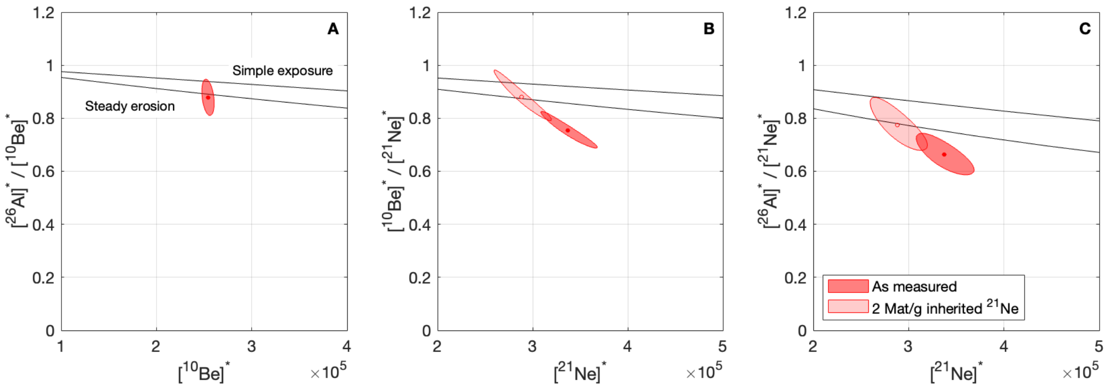

Figure 3. Normalized paired-nuclide plots for ^{10}Be, ^{21}Ne, and ^{26}Al concentrations in CoQtz-N, assuming an estimated sample location and thickness as discussed above in the text. Production rate calculations use the 'St' scaling method and default production rate calibration in version 3 of the online exposure age calculator originally described by Balco et al. (2008) and subsequently updated. The ellipses are 68% confidence regions. If all excess ^{21}Ne is assumed to be cosmogenic (darker ellipses), then ^{21}Ne concentrations show disequilibrium with ^{10}Be and ^{26}Al. Assuming that 2×10^6 atoms g^{-1} of excess ^{21}Ne is nucleogenic or otherwise inherited, and subtracting this from the total (lighter ellipses), results in equilibrium concentrations of all three nuclides.

inherited, and subtracting this from the total, reconciles all three nuclide pairs with steady-state erosion (Fig. 3). Thus, up to approximately 2×10^6 atoms g^{-1} nucleogenic ^{21}Ne may be present in CoQtz-N. However, the presence or absence of nucleogenic ^{21}Ne does not affect the utility of CoQtz-N as an interlaboratory comparison standard for excess ^{21}Ne measurements.

4 Summary

The excess ^{21}Ne concentration in the CoQtz-N standard, normalized to the CRONUS-A standard with a nominal excess ^{21}Ne concentration of 320×10^6 atoms g-1, is $13.87 \pm 0.24 \times 10^6$ atoms g^{-1}. Although the precision of excess ^{21}Ne measurements in CoQtz-N is limited by the higher concentration of included atmospheric ^{21}Ne, analysis of multiple aliquots on a typical modern noble gas mass spectrometer should yield a precise enough measurement to be of value for interlaboratory comparison and evaluation of linearity of interlaboratory offsets. Binnie et al. (2019) reported production of 25 kg of the CoQtz-N standard, which would be sufficient for approximately 200,000 ^{21}Ne measurements. Even if the majority of this material is consumed for ^{10}Be and ^{26}Al analyses, the remainder could, if made available for this purpose, support use as a ^{21}Ne intercomparison standard at a number of noble gas laboratories for some time.

Data availability. All measurements described in the paper are included in the supplementary data.

175 *Competing interests.* Balco is a member of the editorial board of *Geochronology.*

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