Quantitative evolved gas analysis: Winchcombe in comparison with other CM2 meteorites


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Abstract—Two bulk Winchcombe along with six other CM2 meteorite samples were subjected to quantitative evolved gas analysis. The observed release patterns for almost all volatile species demonstrate close similarity for all the samples and especially between those for Winchcombe. This can be considered as a fingerprint for this petrological type of meteorites. We identified several gases including H₂, H₂O, O₂, CO, CO₂, and SO₂ released in different temperature ranges. The sources and mechanisms of their release were also established. Some of the gases, H₂, CO, and CO₂, are released as a result of oxidation of macromolecular organic material from oxygen derived from oxygen-bearing minerals (a part of CO₂ is also released as a result of decomposition of carbonates). The others, O₂ and H₂O, are associated with the phase transformation/decomposition of phyllosilicates and (oxy)hydrates, while a high-temperature release of SO₂ is associated mostly with the decomposition of sulfides and in few cases also with sulfates. A low-temperature release of SO₂ is due to evaporation and oxidation of elemental sulfur from the meteoritic matrix and organic material. The total concentrations of H (mostly represented by H₂O), C, and S, calculated according to calibration of the quadrupole mass spectrometer with reference gases and decomposition of solid samples (CaSO₄⋅2H₂O and NaHCO₃) are in reasonable agreement with those determined by independent methods. Variations in the ratio of the carbon amounts released as CO₂ and CO (C∕C CO₂/C CO) between the samples could be an indicator of their terrestrial weathering.

INTRODUCTION

Evolved gas analysis (EGA) is a widely used technique in a large number of applications in science and industry, from investigations of chemical reactions to food production (see Risoluti & Materazzi, 2018 and references therein). In geo- and cosmochemistry EGA is less common, with a possible exception of the investigation of phase transformations in minerals under heating where thermogravimetric analysis is used in combination with EGA (Małgorzata & Zdzięsaw, 1989). In its pure form, EGA is used to study volatile species released from rocks and minerals during linear heating. The gases are released according to their volatility, location in the mineral structure, or as a result of chemical reactions between coexisting mineral phases in the case of rocks. Thus, EGA gives an outgassing spectrum for each specific gas as a function of temperature, unique for each individual material. The spectra are usually difficult to interpret because they are a superposition of several processes that occur within samples when heated, such as diffusion, decrepitation, phase transformations, and chemical reactions. However, deciphering the processes provides valuable information about the origin and nature of the analyzed samples in terms of their associated volatiles.

Evolved gas analysis has been applied to a number of Apollo samples to determine their volatile budgets.
(Gibson & Moore, 1972a, 1972b, 1973) and continuing through to investigations of Martian rocks by the Curiosity rover (Clark et al., 2021; Ming et al., 2014).

Recently, we developed a quantitative EGA (QEGA) technique and applied it to a selection of meteorites and Apollo lunar samples (Verchovsky, Anand, & Barber, 2020; Verchovsky, Anand, Barber, Sheridan, et al., 2020). The method is based on calibration of a quadrupole mass spectrometer (QMS) with gas flow and makes use of a precise capacitance manometer (MKS baratron®). In addition, calibrations have been performed using chemical compounds decomposing into simple gases when heated, for example NaHCO₃ or CaSO₄·2H₂O.

In the present study we used QEGA to analyze two Winchcombe samples (see the Samples Section) along with several other CM2 samples for comparison.

**ANALYTICAL PROCEDURES**

**Experimental Technique**

Full details of the relevant experimental procedures are given in Verchovsky, Anand, and Barber (2020) and Verchovsky, Anand, Barber, Sheridan, et al. (2020). Briefly, the apparatus consists of a reference system, a precise pressure measurement device, that is, capacitance monometer (MKS baratron®), an extraction furnace and the QMS (Hiden 3F/PIC) used for registration of signals from gas species at different $m/z$. The reference system contains a reservoir with reference gases at 4–5 bar pressure. The reservoir can be filled with pure gases or mixtures of gases with known relative abundances (Table 1). The flow rate of gases from the reservoir is controlled through capillaries (stainless steel, 0.89 mm external diameter) with crimps.

The reference and sample gases were analyzed on the QMS where signal intensities at a number of $m/z$ in the range from 2 to 140 in dynamic mode using peak jumping and ion counting are recorded. The meteorite samples and the reference compounds, NaHCO₃ and CaSO₄·2H₂O, are wrapped in clean Pt foil and heated linearly using a $12^\circC \text{min}^{-1}$ heating rate in the extraction double-wall furnace (with quartz tube inside and ceramic tube outside the space between which is pumped to $10^{-3}$ mbar) from 100 to 1400°C. The evolved gases are continuously passed through the QMS and analyzed in the same way as the reference gases. No carrier gas was used during calibration and sample analyses. Blanks are measured before each sample and the corresponding correction is applied. The blanks seen with a new empty quartz extraction tube are different from those made after sample analyses, mainly in the amount of O₂ released at very high (>1300°C) temperature: oxygen is observed only in the latter cases. This is due to decomposition of SiO₂ vapor originating from the quartz tube at such a high temperature, catalyzed by Pt foil (Verchovsky, Anand, & Barber, 2020). The more Pt is present the higher the amount of oxygen generated. The sample silicates can also contribute to the molecular oxygen budget the same way as suggested for quartz, but to a much lesser extent due to their far smaller mass/surface area compared to the mass/surface area of the quartz tube in the heating zone of the extraction furnace. All the measurements are performed in an automatic computer-controlled mode.

**Calibration**

**Flow Rate Calibration**

Calibration consists of determining the gas flow rate by measuring the pressure of the reference gas in the capacitance monometer using a range of accumulation times and is expressed in cm³ STP per second. To simplify automation of the calibration procedure the reference gas passes through three-parallel capillaries each set for different flow rates. By switching between individual capillaries and their combinations, seven different flow rates of the same gas can be obtained. The results of the calibrations are present in Figure 1.

**QMS Calibration with Reference Gases**

Two gas mixtures prepared by Air Products have been used for the calibration (Table 1). The QMS calibration lines represent dependence of signals (cps) collected at each $m/z$ on flow rate (Figure 2). We fitted the lines using a power law, which gives a better fit than a linear fit, though the difference is generally insignificant. This is important for extrapolation of the fitting lines when calculations are required for the signals outside the range of data points collected during the calibrations. Calibrations with gas mixtures and pure gases give consistent results indicating that the presence of other gases does not significantly affect the calibration results, except when the gases present in the mixtures give signals at the same $m/z$, which is observed, for instance, at $m/z = 28$ where there are contributions from N₂, CO, and CO₂. In this case the second-order lines, from fragments of the gas ions, usually give consistent results. Therefore, it is important to identify all second-order signals for the analyzed gases and their relationships with the main signal that is achieved by analyses of pure gases (Figure 3). The second-order signals are formed as a result of dissociation of the gas molecules into fragment ions in the ion source of the QMS. The relationship between the main and the second-order signals depends on QMS settings, mostly on electron acceleration voltage, and can vary within an order of magnitude.
The calibration lines are used to convert the sample signals in cps into flow rates expressed in cm$^3$ STP per second. In order to find the absolute amount of gas released during linear heating the released curves are integrated over the time of release. Since the number of points collected during linear heating from 100 to 1400°C is large (∼5000) a good determination of the integral is found as a sum of all collected intensities expressed in terms of flow rate (cm$^3$ STP per second) divided by their number and multiplied by the total duration (s) of heating.

**Calibration for H$_2$O and SO$_2$ with NaHCO$_3$ and CaSO$_4$·2H$_2$O**

Decomposition of the pure compounds occurs according to their stochiometric composition:

$$2\text{NaHCO}_3 \rightarrow \text{Na}_2\text{CO}_3 + \text{CO}_2 + \text{H}_2\text{O}$$

$$\text{CaSO}_4 \cdot 2\text{H}_2\text{O} \rightarrow \text{CaO} + 2\text{H}_2\text{O} + \text{SO}_2 + 1/2\text{O}_2.$$ 

H$_2$O is released at low temperatures (100–200°C) from both compounds while SO$_2$ is released at $T > 1000°C$ (Figure 4). The integral of the peaks at $m/z = 17$ (from OH, the secondary signal from water) and $m/z = 64$ over time gives the total number of counts for the amounts of H$_2$O and SO$_2$ respectively, which can be calculated from the weights of the reference sample and the reaction equations. This determines the relationship (conversion factor) between the amounts of gases and the integral number of counts which is then used to find the amounts of H$_2$O and SO$_2$ in the meteorite samples from the corresponding peak integrals. The strong water signal in the QMS (Figure 4b) indicates that despite the pipes between the extraction furnace and QMS being maintained at room temperature, water is only partially condensed. It is assumed that this partial condensation of water occurs the same way during both calibration and sample analyses. Three calibrations made with two aliquots of CaSO$_4$·2H$_2$O and one aliquot of NaHCO$_3$ gave consistent results for the conversion factor: 2.57, 2.45, and 2.67 (in $\times 10^{-14}$ g per count).
In this study we analyzed two fragments (BM.2022, M1-85 and BM.2022, M1-86; 0.419 and 0.507 mg, respectively) of the Winchcombe CM2 meteorite. The full description of the fragments and aliquots (in this paper W1 and W2, respectively) is given by Greenwood et al. (2023) and King et al. (2022), but to summarize: W1 is an example of the highly altered, ~CM2.1 ‘Lithology B’ (Suttle et al., 2023) and W2 is a complex breccia composed of cataclastic matrix and a variety of highly altered clasts, dominated by ~CM2.2, ‘Lithology A’ material (Greenwood et al., 2023; Suttle et al., 2023). To understand whether Winchcombe is a typical CM2 in terms of evolved species composition and release pattern we also analyzed six bulk samples of other CM2

![QMS calibration with different reference gases. The experimental points are fitted with a power law. The calibration lines establish the relationship between QMS signal in cps and flow rate in cm$^3$ STP per second for a certain $m/z$.](image)

**FIGURE 2.** QMS calibration with different reference gases. The experimental points are fitted with a power law. The calibration lines establish the relationship between QMS signal in cps and flow rate in cm$^3$ STP per second for a certain $m/z$. 

**SAMPLES**

In this study we analyzed two fragments (BM.2022, M1-85 and BM.2022, M1-86; 0.419 and 0.507 mg, respectively) of the Winchcombe CM2 meteorite. The full description of the fragments and aliquots (in this paper W1 and W2, respectively) is given by Greenwood et al. (2023) and King et al. (2022), but to summarize: W1 is an example of the highly altered, ~CM2.1 ‘Lithology B’ (Suttle et al., 2023) and W2 is a complex breccia composed of cataclastic matrix and a variety of highly altered clasts, dominated by ~CM2.2, ‘Lithology A’ material (Greenwood et al., 2023; Suttle et al., 2023). To understand whether Winchcombe is a typical CM2 in terms of evolved species composition and release pattern we also analyzed six bulk samples of other CM2
meteorites from the Open University collection: Nogoya, Murray, Cold Bokkeveld, and ALH 88045, 83100, and 82100 with masses 0.811, 0.424, 0.881, 1.163, 0.314, and 0.356 mg, respectively. In addition to QEGA all the CM2 meteorites have been analyzed for C, N, He, Ne, Ar, and Xe by stepped combustion. In the present paper we

FIGURE 3. The relationships between the main and second-order QMS signals for the reference gas mixtures and pure gases. The signals at \( m/z = 14 \) for CO and CO\(_2\) are not formed as a result of fragmentation of the parent molecules but as a result of the formation of double charged ions with mass = 28. The signal at \( m/z = 32 \) for pure SO\(_2\) is formed by \(^{32}\)S\(^+\) ion and not by \(^{16}\)O\(^{16}\)O\(^+\) molecule since the ratio of signals at \( m/z = 32 \) and 34 is \( \sim 12 \) which is much closer to \( ^{32}\)S\(^+/^{34}\)S \((22)\) than to \(^{16}\)O\(^{16}\)O\(^{16}\)O\(^{+}\) \((\sim 250)\). (Color figure can be viewed at wileyonlinelibrary.com)
consider only concentrations of C obtained by stepped combustion to compare them with the QEGA results.

RESULTS

The results of the EGA for W1, W2, and six other CM2s are presented in a series of similar plots (Figures 5–12) showing release patterns of the most important species recorded at \( m/z = 2, 12, 14, 16, 17, 28, 29, 30, 32, 44, \) and 64 with temperature in the same format for all the samples. The total pressure as measured on the ion pump which pumps the evolved gases through QMS is also recorded. Except for some minor differences the pressure variations as well as the release patterns have many similar features for all the samples analyzed. These are listed below:

1. The total pressure has maximum value of up to \( 10^{-6} \) mbar in the temperature range of 600–700°C (Figures 5–12a).
2. The maximum of pressure coincides with maxima of signals at \( m/z = 44 \) or 28 (Figures 5–12a,b,d,e) and also correlate with peaks observed at \( m/z = 2, 12, 14, 16, 29, \) and 30 though the proportions between the peaks varies at different \( m/z \) (Figures 5–12c–g).
3. The closest resemblance is observed for the release patterns at \( m/z = 44 \) and 16 (Figures 5–12b,d) as well as for the signals at \( m/z = 28 \) and 14 (Figures 5–12h).
4. The release pattern at \( m/z = 44 \) is bimodal for most of the samples except for Nogoya and Cold Bokkeveld where it is less pronounced. The low-temperature peak is observed at \( 600 \pm 50°C \) and the high-temperature peak is observed at \( 700 \pm 50°C \), except for Murray where it is observed at \( 800°C \) (Figures 5–12d,e).
5. The maximum signals at \( m/z = 28 \) and 44 are comparable to each other while the signal at \( m/z = 14 \) is smaller than at \( m/z = 12 \), which in turn is smaller than signals at \( m/z = 28 \) and 44 (Figures 5–12b).
6. The signals at \( m/z = 28, 29, \) and 30 are well correlated at their max release in the temperature range 600–1000°C and their relationships are closer to those for combination of C and O isotopes (\( ^{12}C^{16}O, ^{13}C^{16}O, ^{12}C^{18}O \)) in CO rather than for nitrogen isotopes (Figures 5–12g).
7. All the samples show a maximum of signal at \( m/z = 2 \) between 600 and 800°C (Figures 5–12c). In two samples (Figures 7c and 10c) the signals at \( m/z = 2 \) also show an additional low-temperature release peak.
8. The release pattern of the species at \( m/z = 17 \) is smooth with a maximum between 300 and 600°C (Figures 5–12c). The lower temperature release is

![Figure 4](https://example.com/figure4.png)

**FIGURE 4.** Release of SO\(_2\) (a) and water (b) from known aliquots of CaSO\(_4\)·2H\(_2\)O versus time at \( m/z = 64 \) and 17, respectively. Integration of the curves over time gives the total cps for the known amounts of the gases from which the relationship between the total number of counts and the amount of the gases is established.
FIGURE 5. The total pressure variations (a) and release patterns of volatile species at different m/z (b–h) for the Winchcombe (W1) sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 6. The total pressure variations (a) and release patterns of volatile species at different m/z (b–h) for the Winchcombe (W2) sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 7. The total pressure variations (a) and release patterns of volatile species at different m/z (b–h) for the ALH 82100 sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 8. The total pressure variations (a) and release patterns of volatile species at different m/z (b–h) for the ALH 83100 sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 9. The total pressure variations (a) and release patterns of volatile species at different $m/z$ (b–h) for the ALH 88045 sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 10. The total pressure variations (a) and release patterns of volatile species at different m/z (b–h) for the Murray sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 11. The total pressure variations (a) and release patterns of volatile species at different m/z (b–h) for the Nogoya sample. (Color figure can be viewed at wileyonlinelibrary.com)
FIGURE 12. The total pressure variations (a) and release patterns of volatile species at different \( m/z \) (b–h) for the Cold Bokkeveld sample. (Color figure can be viewed at wileyonlinelibrary.com)
observed in the samples where the low-temperature release at m/z = 2 is also observed.

9. Some samples have a strong signal at m/z = 32 at T > 1200°C where it is correlated with the signal at m/z = 16 (Figures 8 and 10–12d). In addition, most of the samples have a low-temperature peak at m/z = 32 in the range 500–600°C (Figures 5–12d). The release pattern of this low-temperature component is very similar to that observed at m/z = 17 (Figures 5–12c).

10. The signal at m/z = 64 is generally observed in two temperature ranges 200–800°C and 1200–1380°C (Figures 5–12d and 13).

11. Apart from many similarities with the above observations both Winchcombe samples have an almost identical release pattern for all the analyzed species, reproducing each other in detail (Figures 5 and 6).

We also note that the results obtained earlier by QEGA for the Murchison (also CM2) sample (Verchovsky, Anand, Barber, Sheridan, et al., 2020) are very similar to those listed above.

**DISCUSSION**

**Identification of the Evolved Gas Species**

When there is no interference between the species with the same m/z, including the secondary order signals, identification of the species is straightforward. Thus, the signal at m/z = 17 corresponds to H2O (assuming constant OH+/H2O+ ratio), at m/z = 44 to CO2 and at m/z = 64 to SO2.

Thermal decomposition of water occurs at 2200°C and can be reduced to 1300–1400°C by Pt catalyst (Jellinek & Kachi, 1984). Therefore, it is possible that part of the molecular hydrogen is the result of recombination of H2O fragments formed during its dissociation in the QMS where there is a hot (~2000°C) filament and ionizing electrons. However, the releases of water and H2 are not at all similar to each other: the release of H2O is quite smooth while the H2 release has many features observed at different temperatures (Figures 5–12c). Therefore, it is difficult to imagine how recombination of H2O molecule fragments could lead to formation of H2 by such a complicated and very specific process without correlation with the release of the main signal at m/z = 17. Moreover, some features of the H2 release coincide with those for CO2 and CO. Thus, if water produces molecular hydrogen its contribution is small and most of the observed H2 comes from other sources (see next section). Since most of the water is released by 1000°C its dissociation in the furnace at 1300–1400°C, if it occurs, makes an insignificant contribution to H2. Water partly turns into OH (at m/z = 17) as evidenced by the nature of its release, which is identical to H2O (at m/z = 18) as can be seen from the experiment with gypsum (Figure 13).

Recombination of H2O fragments also results in the formation of O2, and since the signal at m/z = 32 is much smaller than for H2 in the samples analyzed (Figures 5–12c,d) its influence on the O2 signal can potentially be significant. The results obtained for gypsum (Figure 13), used for calibration for water and SO2 indicate that in the temperature range where most water is released no signal from oxygen is observed, that is, the process of fragmentation of water molecules does not occur in our experiments, at least to the extent that can be detected. Oxygen is only released at high temperature where it is expected to appear together with SO2 as a result of decomposition of CaSO4. Small amounts of H2 released at low and middle T appear to be an impurity in the gypsum. Thus, the results for oxygen agree with those for hydrogen discussed in the previous paragraph.

Some contribution at m/z = 32 is also expected from the second-order signal of SO2 (Figure 3). Since we did not analyze pure SO2 gas at the same time as the meteorite samples and used different QMS settings during the analysis, the relationship between the main and the second-order signals for SO2 may differ slightly from what it would be if pure SO2 were analyzed with the same QMS settings as used for the samples. The high-temperature release of SO2 from Nogoya (Figure 14) can also be used to determine the relationships between the main and secondary signals for SO2, since it does not seem to mix with any other signals and, in particular, oxygen from decomposition of SiO2 of the sample tube. There are at least three second-order signals associated with SO2 that closely and in detail correlate with that at m/z = 64: at m/z = 16, 32, and 34 (the signal at m/z = 48 was not measured). As expected, the ratios between the signals are similar but not exactly the same as determined for pure SO2 (Figure 3). The ratio between signals at m/z = 32 and 34 (~24) indicates that the signal at m/z = 32 is a 32S+ ion and not 16O18O+ ion since the ratio of molecular oxygen with a mass 32 (16O16O) to that with a mass 34 (16O18O) is ~250 and the 32S/34S ratio is 22. This result is also similar to what was determined for pure SO2. The ratio of the signal at m/z = 64 to the signal at m/z = 32 (32S+) is ~10 (for pure SO2 it is ~50). Note that the ratio of SO2 to O2 signals for the gases formed during decomposition of calcium sulfate is about 0.6 (Figure 13), indicating that the release of SO2 from Nogoya shown in Figure 14 is not a result of decomposition of a sulfate (see next section). For the W1 and W2 samples, a significant contribution from SO2 in the signal at m/z = 32 is observed at low-temperature (250–300°C) where a small amount of O2 is released while
the signal at \( m/z = 64 \) is relatively high (Figures 5 and 6d). For the main O\(_2\) release this contribution is almost negligible since the signal at \( m/z = 64 \) at \( m/z = 32 \) is significantly lower than that at \( m/z = 32 \). For ALH 82100 (Figure 7d) most of the signal at \( m/z = 32 \) is from SO\(_2\) since both signals are well correlated and the ratio of the signal at \( m/z = 64 \) to that at \( m/z = 32 \) is \( \sim 10 \). For ALH 83100, ALH 88045 and Murray (Figures 8–10d) most of the signal at \( m/z = 32 \) corresponds to O\(_2\) since the ratio of the signal at \( m/z = 64 \) to the signal at \( m/z = 32 \) is much less than 10. For Nogoya and Cold Bokkeveld (Figures 11 and 12d) there is a small contribution of the second-order signal from SO\(_2\) at \( m/z = 32 \) since the signal of SO\(_2\) is higher than at \( m/z = 32 \), although the ratio between them is less than 10 and the signals are not correlated. Thus, in most samples, the majority of the signal at \( m/z = 32 \) corresponds to O\(_2\).

For \( m/z = 28 \) the situation is more complicated. Apart from the interference between CO and N\(_2\) there is also a contribution at this \( m/z \) from the secondary signal of CO\(_2\). Pure CO\(_2\) gives secondary signals at \( m/z = 16, 28 \) and 12 which are smaller than the main line at \( m/z = 44 \) by factors of 5, 10, and 20, respectively (Figure 3). So, a significant contribution from CO\(_2\) is possible only when the CO\(_2\) signal is significantly higher than that at \( m/z = 28 \). At the same level of the main signals the contribution from CO\(_2\) at \( m/z = 28 \) is only 10%. For our samples a moderate contribution from CO\(_2\) is possible only in the case of Nogoya in the temperature range 200–600°C (Figure 11b) and at the very low temperature (\( \sim 100°C \)) for ALH 82100 (Figure 7b). For all other samples and temperature ranges the contribution from CO\(_2\) at \( m/z = 28 \) is negligible. Thus, the signal at \( m/z = 28 \) is potentially created by CO and N\(_2\). To assess the contribution from each of them the relationship between the signals at \( m/z = 28, 14, \) and 12 for the pure gases has to be considered. Pure N\(_2\) gives a second-order signal at \( m/z = 14 \) that is a factor of 15 times smaller than that at \( m/z = 28 \) (Figure 3). Pure CO gives second-order signals at \( m/z = 14 \) and 12 at factors of 100 and 43 smaller than the main signal correspondingly. Thus, at the same level of the main signals at \( m/z = 28 \) N\(_2\) gives a higher signal at \( m/z = 14 \) than CO gives the signal at \( m/z = 12 \) (Figure 3). In fact, the signal at \( m/z = 14 \) is significantly smaller than that at \( m/z = 12 \) (Figures 5–12b) which means that CO is the only gas creating the signal at \( m/z = 28 \) for all our samples. As mentioned in the previous section, the relationship between signals at \( m/z = 28, 29, \) and 30 (Figures 5–12g) also supports this conclusion.

We also considered whether the signal at \( m/z = 14 \) might be associated with nitrogen. We concluded that it was not, since this signal is well-correlated with that at \( m/z = 28 \) (Figures 5–12h). That, together with the association of the signal at \( m/z = 28 \) with CO, as discussed in the previous paragraph, means that it comes from CO. It also follows from the relationships between signal at \( m/z = 14 \) for pure CO and N\(_2\) (Figure 3). And although the signal at \( m/z = 14 \) cannot be formed directly...
by dissociation of CO; it could be a result of a combination of $^{12}$C$^+$ with H$_2$ to form CH$_2$ ion or due to formation of a double charged CO$^{++}$ ion in the QMS. Although CO dominates the $m/z=28$ signal, it is not responsible for the signal at $m/z=12$ as might be expected. In fact, the signal at this mass is much better correlated with CO than CO (Figures 5–12e,f), that is, the signal at $m/z=12$ comes mainly from CO$_2$. This also follows from the relationships between signals at $m/z=12$ for pure CO$_2$ and CO (Figure 3).

In all meteorite samples an $m/z=14$ peak is observed at 400 ± 50°C (Figures 5–12h). It is not, however, related to the release of N$_2$, as it might seem at first glance. This peak is accompanied by the signal at $m/z=29$, released exactly at the same temperature as that at $m/z=14$ (Figures 5–12g). An important point is that this peak at $m/z=29$ is not correlated with either $m/z=28$ or $m/z=30$. Therefore, the only reasonable explanation for the observation is to suggest that these peaks represent hydrocarbons (CH$_2$ and C$_2$H$_6$) formed as a result of decomposition of a specific organic compound present in all the samples. Similar explanations can be given to the very pronounced peak observed at 200°C for the signal at $m/z=14$ only for ALH 82100 (Figure 7h), since the peak is also accompanied by a pronounced peak at $m/z=30$. Thus, this observation can also be explained by the presence of a specific organic compound in this sample which decomposes to form CH$_2$ and C$_2$H$_6$. Of course, other hydrocarbons can also be released, for example, methane, which could also contribute to $m/z=16$. However, if present, it is obscured by the high signal at $m/z=16$ from CO$_2$ (see Figures 3 and 5–12d). We note here that the amounts of carbon and hydrogen released from the meteorites in the form of hydrocarbon appears to be relatively small; most of which is released in the form of CO$_2$ and CO and molecular hydrogen correspondingly.

A clear signal at $m/z=34$ was detected for all the samples, but it seems not to be associated with H$_2$S since it is well correlated with the release of oxygen and not with release of H$_2$ or sulfur. Figure 15 shows this for Nogoya, but similar correlations are also observed for all other samples where a pronounced broad signal at $m/z=32$ is observed (Figures 5d, 6d, 8d, 9d, 11d, 12d). The relationship between the signals at $m/z=32$ and $m/z=34$ (∼10, Figure 15) suggests that the latter is not due to molecule of $^{16}$O$^{18}$O since for oxygen the ratio of $^{16}$O$^{16}$O/$^{16}$O$^{18}$O is ~250. Thus, a plausible choice for the signal at $m/z=34$ is H$_2$O$_2$. The correlation of the $m/z$ 34 signal, in addition with O$_2$, also with the release of water (Figures 5–12c) confirms that formation of the molecule can indeed occur. It is important to note here that no signal at $m/z=34$ is observed during release of H$_2$O from CaSO$_4$.2H$_2$O reference sample, suggesting that H$_2$O$_2$ is not formed in QMS but rather associated with chemical reactions occurring during heating of the meteorite samples.

**Sources and Mechanisms of Volatile Release from CM2 Meteorites**

**Carbon Monoxide and Dioxide**

Most CO$_2$ is released at a relatively low temperature in contrast to CO, most of which is released at higher temperature (Figures 5–12b). The fact that the low-temperature maximum of CO$_2$ release is observed at 550–650°C suggests a contribution from calcium/magnesium/iron carbonates often present in CM2 meteorites, which decompose to CO$_2$ in this temperature range. This is confirmed by stepped combustion data for the CM2 samples (Figure 16) and for Winchcombe in particular (King et al., 2022). The difference in the observed decomposition temperature may be due to the predominant type of carbonate present in each sample. Formation of the rest of CO and CO likely occurs as a result of a chemical reaction between organic material in the meteorites and oxygen-bearing minerals. Some variations in the release pattern of the species can be associated with the difference in mineralogy and organic material distribution in the samples that can affect efficiency of the organic matter oxidation. Nitrogen,
which is also a relatively abundant element in carbonaceous chondrites, is closely associated with organic macromolecular material. It can be released as a result of oxidation but has a negligible contribution to the signal at $m/z = 28$ compared to that derived from CO and at $m/z = 14$ from CO$_2$ fragments, as discussed above.

**Water**

Water ($m/z = 17$) is released in a wide temperature range with a peak at 300–600°C (Figures 5–12c). According to thermogravimetric analysis (TGA) water from CM meteorites can be released upon heating from different sites and by different mechanisms including release of adsorbed molecular water and as a result of decomposition of (oxy)hydroxides and phyllosilicates, with the latter expected to be its dominating source (Garenne et al., 2014). Our data are generally consistent with these results. However very little, if any, adsorbed water is released below 200°C which is less than expected (Garenne et al., 2014) and this is true for the sample ALH 83100, which was also analyzed by TGA (Garenne et al., 2014). This may be due to loss of the low-temperature water while the samples were under high vacuum prior to EGA. The maximum water release at ~300°C from ALH 82100 and Murray apparently suggests that it comes mostly from (oxy)hydroxides. In all other samples most of the water is released in the temperature range 400–700°C corresponding to decomposition of phyllosilicates. The close relationship between release of water and decomposition of phyllosilicates is established by the correlation between the total hydrogen content (mostly in the form of H$_2$O, see Table 2) and the phyllosilicate fraction in CM2s (Suttle et al., 2021).

**Oxygen**

The correlation between O$_2$ and H$_2$O releases (Figures 5, 6, 8, 9, 11, and 12d,e) suggests a similar mechanism for their source and evolution. Interestingly this is not observed if H$_2$O is released at a much lower temperature (~300°C), that is, from (oxy)hydroxides, as is seen for ALH 82100 and to some extent for Murray (Figures 7 and 10d,e), but only when H$_2$O is released in the 400–700°C temperature range. Thus, it seems that the molecular oxygen that appears along with H$_2$O is also associated with phyllosilicate decomposition but not with
<table>
<thead>
<tr>
<th>Sample</th>
<th>H\textsubscript{2}, wt% ×10\textsuperscript{−2} (H/C, at)</th>
<th>Total H (wt%)</th>
<th>H\textsubscript{2}O (wt%)</th>
<th>Total C (wt%) (CO\textsubscript{2}/CO)</th>
<th>Total S (sulfate S) (wt%)</th>
<th>Elemental S (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Meas. DBIM</td>
<td>Meas. DBIM</td>
<td>Meas. DBIM</td>
<td>Meas. DBIM</td>
<td>Meas. DBIM</td>
<td>Meas. DBIM</td>
</tr>
<tr>
<td>W1</td>
<td>4.9 ± 0.3 (0.54 ± 0.1)\textsuperscript{1}</td>
<td>1.7 ± 0.3</td>
<td>15 ± 2</td>
<td>1.4 ± 0.2 (0.67)\textsuperscript{2}</td>
<td>1.9 ± 0.1</td>
<td>0.05 ± 0.08</td>
</tr>
<tr>
<td></td>
<td>8.1 ± 1 (0.66 ± 0.1)</td>
<td>1.9 ± 0.3</td>
<td>17 ± 3</td>
<td>1.5 ± 0.2 (0.71)</td>
<td>2.1 ± 0.1</td>
<td>0.11 ± 0.02</td>
</tr>
<tr>
<td>Nogoya</td>
<td>4.2 ± 0.5 (0.31 ± 0.1)</td>
<td>1.3 ± 0.3</td>
<td>12 ± 1.7</td>
<td>1.6 ± 0.2 (2.4)</td>
<td>1.6 ± 0.3</td>
<td>0.54 ± 0.07</td>
</tr>
<tr>
<td></td>
<td>1.0\textsuperscript{3}</td>
<td>1–1.4\textsuperscript{4}</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Murray</td>
<td>7.1 ± 0.9 (0.30 ± 0.1)</td>
<td>1.3 ± 0.2</td>
<td>11.3 ± 1.7</td>
<td>2.8 ± 0.4 (1.56)</td>
<td>2.3 ± 0.2</td>
<td>0.09 ± 0.01</td>
</tr>
<tr>
<td></td>
<td>(0.68)\textsuperscript{5}</td>
<td>0.6–1.0\textsuperscript{6}</td>
<td>6.7\textsuperscript{a}</td>
<td>2.1 ± 0.3</td>
<td></td>
<td>1.77\textsuperscript{c}</td>
</tr>
<tr>
<td></td>
<td>1.9\textsuperscript{m}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.06\textsuperscript{c}</td>
</tr>
<tr>
<td>Cold Bokkeveld</td>
<td>6.1 ± 0.8 (0.32 ± 0.1)</td>
<td>1.6 ± 0.3</td>
<td>13.4 ± 1.8</td>
<td>2.3 ± 0.3 (1.9)</td>
<td>1.8 ± 0.2</td>
<td>0.39 ± 0.05</td>
</tr>
<tr>
<td></td>
<td>(0.58)\textsuperscript{6}</td>
<td>0.6–1.2\textsuperscript{b}</td>
<td>9.1\textsuperscript{a}</td>
<td>3.4 ± 0.4</td>
<td>(1.2 ± 0.3)\textsuperscript{i}</td>
<td>0.57\textsuperscript{c}</td>
</tr>
<tr>
<td></td>
<td>1.4\textsuperscript{m}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ALH 83100</td>
<td>11 ± 1.5 (0.53 ± 0.1)</td>
<td>2.3 ± 0.3</td>
<td>19 ± 3</td>
<td>2.6 ± 0.4 (2.75)</td>
<td>0.95 ± 0.1</td>
<td>0.21 ± 0.03</td>
</tr>
<tr>
<td></td>
<td>(0.65)\textsuperscript{6}</td>
<td>1.46\textsuperscript{d}</td>
<td></td>
<td>3.5 ± 0.4</td>
<td>(1.0 ± 0.3)\textsuperscript{i}</td>
<td></td>
</tr>
<tr>
<td>ALH 82100</td>
<td>3.5 ± 0.4 (0.42 ± 0.1)</td>
<td>0.5 ± 0.07</td>
<td>4.2 ± 0.5</td>
<td>1.63 ± 0.2 (5.6)</td>
<td>1.3 ± 0.1</td>
<td>0.35 ± 0.05</td>
</tr>
<tr>
<td>ALH 88045</td>
<td>5.4 ± 0.6 (0.86 ± 0.1)</td>
<td>1.0 ± 0.2</td>
<td>8.8 ± 1.2</td>
<td>0.75 ± 0.11 (4.3)</td>
<td>0.87 ± 0.06</td>
<td>0.13 ± 0.02</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Eiler and Kitchen (2004).
\textsuperscript{b}Kerridge (1985), Vacher et al. (2020).
\textsuperscript{c}Kaplan and Hulston (1966).
\textsuperscript{d}Alexander et al. (2010, 2013).
\textsuperscript{e}Burgess et al. (1991).
\textsuperscript{f}Dreibus et al. (1995).
\textsuperscript{g}Alexander et al. (2007).
\textsuperscript{h}Rubin et al. (2007).
\textsuperscript{i}Determined by independent methods.
\textsuperscript{j}H/C atomic ratio.
\textsuperscript{k}CO\textsubscript{2}/CO ratio.
\textsuperscript{l}Sulfate-associated S concentration.
\textsuperscript{m}Recalculated from vol\% from Howard et al. (2015).
decomposition of (oxy)hydroxides and the second-order signal from SO\textsubscript{2} as discussed in the previous section. The release of O\textsubscript{2} at very high temperature (>1300°C) is due to an artifact and not related to the meteorite samples, as was mentioned in the Experimental Technique Section.

**Hydrogen**

A broad H\textsubscript{2} release within a 200–1200°C interval suggests that it mostly comes from decomposition/oxidation of the organic macromolecular material, since this is the temperature range where most carbon is released in the form of CO and CO\textsubscript{2} (release of CO\textsubscript{2} from carbonates is seen on the broad total CO\textsubscript{2} release as an additional narrow peak at 600–700°C; Figures 5–12b,e). In more detail this can be seen in Figure 17. For W1, W2, Nogoya, and Cold Bokkeveld most of the local peaks of H\textsubscript{2} are observed at the same temperature as for CO while for ALH83100 and ALH88045 some of them correlate with the release of CO\textsubscript{2}, instead, and for ALH 82100 and Murray, with the release of water. The correlation between H\textsubscript{2}, CO, and CO\textsubscript{2} peaks is pointing to the same release mechanism for these species, which appears to be destruction (oxidation) of the macromolecular material. At the same time the release patterns are generally not correlated with each other, that is, the proportions between the local peaks differ significantly, which can be due to variations in the H/C ratios in different types of organic matter degraded/oxidized at different temperatures. This is confirmed by H/C ratios (Table 2) deduced from the total concentrations of C and H, released as H\textsubscript{2} (see Concentration of the Volatile Species Released During EGA Section) that are in reasonable agreement (except for Murray and Cold Bokkeveld, see below) with the H/C ratios determined in insoluble organic matter (IOM) obtained by dissolution of bulk CM2 meteorites in CsF-HF-HCl (Alexander et al., 2007). The separate low-temperature peak of H\textsubscript{2} observed for ALH 82100 and Murray present at the same temperature where the water associated with (oxy)hydroxides is released (Figure 17) indicates that this hydrogen may also be associated with decomposition of these minerals. The low yield of oxygen observed in the temperature range (Figures 7d and 10d) indicates that thermal decomposition of water does not play a significant role for the release of hydrogen (see Identification of the Evolved Gas Species Section).

**Sulfur Dioxide**

Release of SO\textsubscript{2} at high (>1100°C) temperature shows some differences between the meteorite samples (Figure 18). In some samples (W1, W2, Nogoya, and ALH 88045) it has a single peak with maximum at \(T > 1350^\circ\text{C}\) and is accompanied with a little molecular oxygen including that coming from decomposition of quartz. For the other (ALH 82100, 83100, Cold Bokkeveld, and Murray) samples it is shifted to the low-temperature side, bimodal (except for Murray) and accompanied with large amounts of O\textsubscript{2} associated with decomposition of quartz reaction tube (see the Experimental Technique Section). However, in general there is no correlation between release of this O\textsubscript{2} and SO\textsubscript{2}, that is, oxygen is not directly related to the release of SO\textsubscript{2}.

The relatively low-temperature SO\textsubscript{2} release (at \(T < 900^\circ\text{C}\)) has a significantly smaller signal at \(m/z = 64\) than that observed at high temperature and shows highly variable release patterns and temperature ranges for different meteorites. For Nogoya, Cold Bokkeveld, and ALH 88045 SO\textsubscript{2} is evolved between 500 and 900°C, for all other samples it is from 200 to 600°C, and all samples exhibit a series of peaks at different temperatures (Figures 5–12d).

All these observations can be explained in terms of chemical forms of sulfur present in CM2 meteorites. It is known (Airieau et al., 2005; Bates et al., 2020; Burgess et al., 1991; Gao & Thiemens, 1993; Howard et al., 2011, 2015; Kaplan & Hulston, 1966; Lee, 1993; Monster et al., 1965; Rubin et al., 2007) that in carbonaceous chondrites, and in CM2s in particular, there are three principal chemical forms of sulfur: sulfides (mainly troilite, pyrrhotite, and pentlandite), elemental S and sulfates (gypsum, bloedite, or epsomite). The elemental S is present in both the meteoritic matrix and in the organic material. Sulfide reduced form of sulfur is generally dominated, though in some studies of CM meteorites (Burgess et al., 1991; Gao & Thiemens, 1993; Kaplan & Hulston, 1966) the oxidized (sulfate) sulfur was reported to be the most abundant its form. As it is highly volatile, elemental S is probably released first upon heating compared to its other forms. Since it is detected in the form of SO\textsubscript{2} there must be sufficient oxygen (in different forms) present to oxidize sulfur. Indeed, release of carbon as CO\textsubscript{2} and CO in this temperature range confirms this since these gases are produced from organic or elemental carbon. Elemental sulfur from the organic matter (e.g., sulfonic acid) seems to be released first, followed by release from the matrix when it starts to degrade at higher temperature. This is confirmed by correlations between peaks of SO\textsubscript{2}, CO\textsubscript{2}, and H\textsubscript{2} observed for the releases at 200–300°C. Oxidation of this S and C appears to occur from oxygen present in the organic matter. Release of SO\textsubscript{2} in the 400–800°C temperature range is not clearly correlated with any other species and appears to result from oxidation of the elemental sulfur in the matrix. The complex character of SO\textsubscript{2} release in this temperature interval, with several individual peaks, can be explained by occurrence of the elemental sulfur in different energetic positions in the matrix. However, a contribution of SO\textsubscript{2} from some sulfides cannot be ruled out.

The high-temperature release of SO\textsubscript{2} is evidently related to sulfide and/or sulfate decomposition which
FIGURE 17. Correlation of H₂ releases with CO, CO₂, and H₂O for the studied meteorites. For W1, W2, Nogoya, and Cold Bokkeveld most of the local peaks of H₂ are observed at the same temperature as for CO while for ALH83100 and ALH88045 some of them correlate with the release CO₂, and for ALH 82100 and Murray, with the release of water. (Color figure can be viewed at wileyonlinelibrary.com)
occurs at 1100–1300°C depending on its chemical forms and the presence of catalysts. As was mentioned in the Identification of the Evolved Gas Species Section, decomposition of pure gypsum gives O₂ along with SO₂ at \( T \geq 1300°C \). In the samples with the low O₂ signal from decomposition of quartz and particularly for Nogoya, the release curves for \( m/z = 32 \) and 16 follow exactly that for \( m/z = 64 \) and therefore can be the second-order signals from SO₂ (see the Identification of the Evolved Gas Species Section for more detail discussion about this). (Color figure can be viewed at wileyonlinelibrary.com)

FIGURE 18. The high-temperature release of SO₂ along with the signal at \( m/z = 16 \) and 32. The samples corresponding to the upper row plots show weak signals of O₂ from decomposition of quartz, while the samples associated with the bottom row plots display strong such O₂ signals at \( T > 1300°C \). In the samples with the low O₂ signal from decomposition of quartz and particularly for Nogoya, the release curves for \( m/z = 32 \) and 16 follow exactly that for \( m/z = 64 \) and therefore can be the second-order signals from SO₂ (see the Identification of the Evolved Gas Species Section for more detail discussion about this). (Color figure can be viewed at wileyonlinelibrary.com)
Concentration of the Volatile SpeciesReleased During EGA

The concentrations of the analyzed gases in Winchcombe and other CM2 samples are presented in Table 2 along with those determined by other independent methods for the same or similar meteorites.

Carbon

The total carbon content has been calculated as a sum of CO and CO₂ releases, taking into account the contribution of the second-order signal at m/z = 28 from that at m/z = 44. The correction is based on the results of pure CO₂ measurement (Figure 3) from which the ratio between the main and the second-order signals at m/z = 44 and 28 can be found. The carbon emanates from a mixture of an oxidized meteoritic organic component with δ¹³C of −25 to −10‰ (Sephton, 2014) plus gas from the decomposition of carbonates. CM2 chondrites are known to contain carbonates with δ¹³C up to ∼+70‰ (Grady et al., 1988). The contribution of carbonate to the total carbon content can be assessed from the stepped combustion results (Figure 16) where it is seen as a release of CO₂ at 500–700°C and a shift in δ¹³C to higher values. This was most clearly revealed for Winchcombe when analyzed with a high-temperature resolution.

The relationship between CO₂ and CO is significantly variable (0.67–5.6) in different meteorites (Table 2). In Winchcombe it is the lowest, while in the Antarctic meteorites it is much higher with the highest value in ALH 82100. The non-Antarctic meteorites are all observed falls, hence unweathered as well as Winchcombe. In contrast, the Antarctic meteorites are finds, and known to be affected to various extents by terrestrial weathering, especially the formation of carbonates with δ¹³C ∼ 0 ± 10‰ (Grady et al., 1991). The study found that CM2 chondrites from Antarctica could have as much as twice the carbonate content as non-Antarctic meteorites. This observation suggests that the CO₂/CO variations could result from the presence of terrestrial weathering products, specifically the decomposition of Antarctic carbonates to CO₂.

There is good agreement between the concentrations of carbon obtained by QEGA and by stepped combustion (Table 2), except for ALH 83100, where the former gives 2.6 times higher value than the latter. It might be a result of the sample heterogeneity given that a very small aliquot (0.3 mg) was used for QEGA. With the possible exception of Antarctic meteorites, carbonate decomposition does not significantly contribute to the variations in the H/C ratios calculated for total carbon and hydrogen (as H₂) contents, discussed in the next paragraph.

Hydrogen

According to our results Winchcombe and other CM2 meteorites contain hydrogen associated with organic material and water from phyllosilicates. Hydrogen from the former source accounts only few percent of its total amount by weight. Most of the hydrogen is related to water. The water, as well as the total hydrogen contents, reasonably agree with the results obtained by independent methods (Table 2). Since we believe that hydrogen released as H₂ and the total carbon (except for that related to carbonate) are associated with meteoritic organic matter, the calculated H/C ratio for our samples should be comparable with that of the organic matter. The IOM in CM meteorites has this ratio within 0.59–0.71 (Alexander et al., 2007). For most of our samples the H/C ratios are within or close to this range. Contribution from carbonate or other carbon phases not related to IOM can reduce the ratio to a certain extent but probably not by a factor of 2 as it is observed for Nogoya, Murray, and Cold Bokkeveld. The reason for the discrepancy is not entirely clear but may be related to the formation of hydrocarbons, in which form a part of H₂ is released. This may also be due to the oxidation of part of the molecular hydrogen during the release process. In this case the H/C ratio will be affected much more than the H₂O content due to the high H₂O/H₂ ratio, such that, even accounting for the entire discrepancy in H/C ratio reported here compared to previously published ratios by other techniques would result in changes to the reported H₂O abundance of ∼0.3 wt%, well within the reported uncertainties.

Sulfur

In general, the total sulfur concentrations calculated within the range known for CM2 meteorites (Table 2). Most of it is associated with sulfides. Sulfates are identified in three of the seven samples. The concentration of sulfur related to sulfate in the meteorites can be estimated as 1.1, 1.0, and 1.2 wt%, respectively (Table 2). These concentrations are somewhat higher than those associated with gypsum (0.2–0.6 wt% of S) found in few other CM meteorites (Bates et al., 2020; Howard et al., 2015). Sulfates could be products of aqueous alteration on the CM2 parent bodies, although, according to some studies (Losiak & Velbel, 2011; Velbel & Palmer, 2011) a significant part of the sulfur may be due to terrestrial contamination. However, the isotopic composition of oxygen in sulfates from CM and CI carbonaceous chondrites (Airieau et al., 2005) is compatible with extra-terrestrial origin of the minerals. The contribution from the elemental sulfur accounts for from 4% to 32% of its total amount, which is also in the range determined by the independent methods.
CONCLUSIONS

1. The results for Winchcombe and other CM2 meteorites analyzed in the present study by QEGA show many common features in the release pattern for almost all volatile species. The release pattern can be considered as a fingerprint for CM2 meteorites. Lunar soils also show characteristic release but again all soil samples are very similar (Verchovsky, Anand, & Barber, 2020). Crucially, although CM2 meteorites and lunar soils release the same volatiles, the release patterns are distinct. A comparison with other types of meteorites such as CI, CV, and CR is required to confirm this conclusion.

2. Analysis of the observed release patterns of volatile species at different m/z obtained by QEGA allows the identification of common gases and establishment of their origin and the processes involved in their evolution during linear heating. Carbon (as CO and CO$_2$) and hydrogen (as H$_2$) are mostly released as a result of oxidation of organic material. A part of CO$_2$ is also released as a result of decomposition of carbonates. Most water and molecular oxygen are released in association with decomposition of phyllosilicates. In some cases, water is predominantly released from (oxy)hydrates where it is accompanied by release of H$_2$. Most sulfur is released as a result of decomposition of sulfides at high temperature as SO$_2$. A small fraction of the total sulfur is released at relatively low temperature by evaporation of elemental sulfur from the meteoritic matrix and organic material which is instantly oxidized and appears as SO$_2$. In three of the studied meteorites sulfatic sulfur was also identified.

3. The total concentrations of hydrogen, carbon, and sulfur found in the meteorites analyzed by QEGA are in reasonable agreement with those determined by independent methods.

4. As the least affected by terrestrial contamination, Winchcombe shows some distinct features from other CM2s and in particular for the CO$_2$/CO ratio which could be a sensitive indicator of such contamination.

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Data Availability Statement—The data that support the findings of this study are available from the corresponding author upon reasonable request.

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REFERENCES


