Rediscovery of the Elements

Joseph Black–Magnesia and Fixed Air

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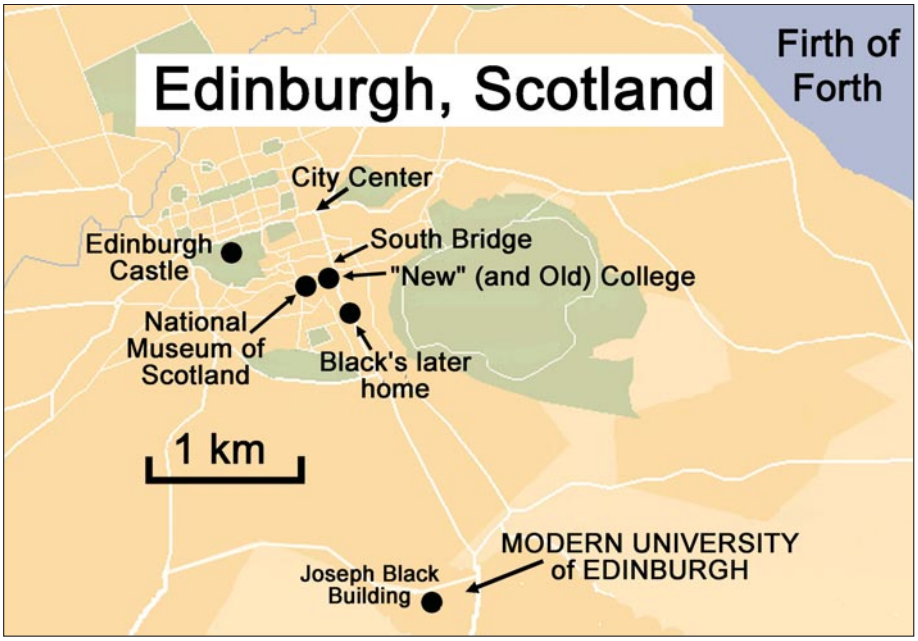

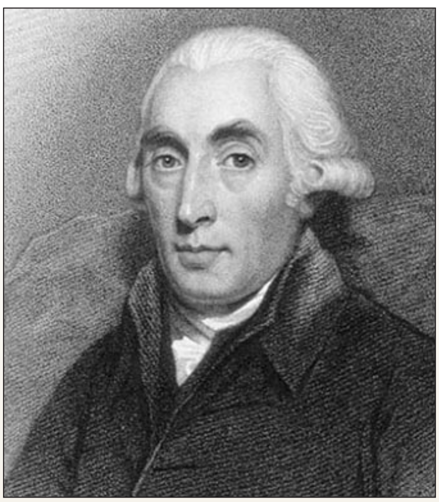

Figure 1. (Left) Engraving of Joseph Black, made in 1800 by James Heath (1757–1834), taken from a ca 1790 portrait by Henry Raeburn (1756–1823). Raeburn was a student of David Martin, whose portrait of young Joseph Black is shown on the front cover.

Figure 2. Modern map of Edinburgh. The chemical discoveries of Black were performed at the Edinburgh "Old College," whose buildings were taken down and replaced by the "New College" during 1827–1831. The Royal Museum of Scotland is located 200 meters west, where exhibits on Black are presented (see Figure 8). The modern campus is 2.7 km south of the "New College." During his last 18 years, Black lived on Nicholson Street, a continuation of South Bridge.

Joseph Black (1728–1799) (Figure 1) is perhaps best known for the discovery and characterization of carbon dioxide (fixed air), made during his research with alkalies and carbonates. Simultaneously, he made the first chemical distinction between calcia (CaO) and magnesia (MgO) and thus could be credited with the discovery of magnesium. This research was performed at the University of Edinburgh, Scotland (Figure 2).[1b]

Black is also known for his pioneering research in latent heat at the University of Glasgow, where he was the first to notice that the temperature of an ice-water mixture does not rise above the freezing point of water until all of the ice has melted (Figure 3).[1d]

Joseph Black's career.[1b,d,2] Joseph Black's family was of Scottish origin. His father was born in Belfast but migrated to Bordeaux, France, where he set up a wine business. The son Joseph returned to the British Isles and studied four years (1746–1750) in Glasgow with William Cullen (1710–1790). Black then matriculated at the University of Edinburgh (1750–1754), where he earned his medical degree with Charles Alston (1683–1760), the Chair of Botany and a specialist of *materia medica* (medicinal drugs). Black then returned to Glasgow in 1756 to become professor of anatomy and lecturer in chemistry, replacing Cullen who had taken a position at the University of Edinburgh; Cullen became one of the distinguished professors at Edinburgh who helped it become one of the leading medical schools in Europe. Then in 1766 Black returned to the University of Edinburgh as professor of chemistry, replacing Cullen who had been promoted to Professor of the Institutes of Medicine. Black remained at Edinburgh the remainder of his life (Figures 4, 5).

Black's scientific reputation was widespread throughout Europe and America, and he was visited frequently by those who sought expertise and guidance from the master (Figures 6, 7). Smithson Tennant (1761–1815; the discoverer of iridium and osmium[3e]) studied with Black in 1781. When the Polish scientist Jedrzej Sniadecki (1768–1838; the discoverer of

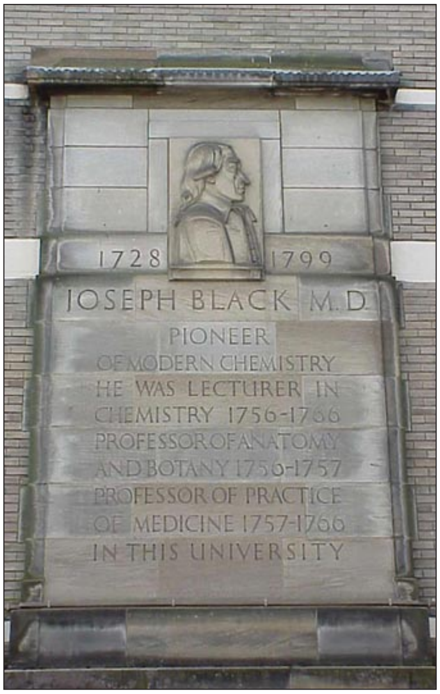

Figure 4. Both Glasgow and Edinburgh claim Joseph Black. This is the Joseph Black Building (Chemistry Building) in Edinburgh (N55° 55.44 W03° 10.58) at the present-day south campus, occupied in 1924.

Figure 3. This plaque resides on the Joseph Black Building (Chemistry Building) of the modern University of Glasgow (N55° 52.32 W04° 17.57). Black's main contribution in Glasgow was his work in latent heat[1d] in 1761. Black preferred to do research in the winter, when ice was available for his calorimetry and latent heat studies. His ideas on the latent heat of steam gave James Watt the inspiration and technology to develop steam power. In his day the campus was on High Street, 3.5 km east of the present campus; the buildings now are completely gone.

"vestium,"[3f] traveled to Western Europe to further his education, he was prevented from visiting Antoine Lavoisier (1743–1794) because of the French Revolution; as an obvious alternative he turned to Black in Edinburgh.[4] Benjamin Rush, a co-signer of the Declaration of Independence, had earned his medical degree at the University of Edinburgh; he returned to Philadelphia in 1769 and presented courses at the College of Philadelphia (today the University of Pennsylvania) based on Joseph Black's very popular lectures.[5]

Caustic and mild alkalies as medicines. As a student at Edinburgh, Joseph Black became intrigued with alkalies, a research interest of his advisor Charles Alston. In the 1700s, alkalies were known to include the groups of vegetable (potash), marine (soda), volatile (ammonia), and calcareous (lime). Each of these alkalies could appear in "mild" (carbonate) and "caustic" (hydroxide) forms.[1b, 6] The compositions of each were unknown, but today we know these as:

Alkali	Mild form	Caustic form
Potash	K_2CO_3	KOH
Soda	Na_2CO_3	NaOH
Ammonia	$(NH_4)_2CO_3$	NH_4OH or NH_3
Calcia	$CaCO_3$	CaO or $Ca(OH)_2$

It was believed that alkalies might be solvents for "urinary calculi" (kidney stones), but the caustic forms were too acrid to be useful medicinal remedies—when caustic alkali was applied to a dog's bladder in an attempt to dissolve the stones, instead they "dissolved the bladder."[7] Hence, the mild forms were prescribed for humans, such as ordinary chalk ($CaCO_3$). It was soon recognized that chalk might also be a useful remedy for stomach maladies because it relieved indigestion. (Today, the antacid $CaCO_3$ is available as "Tums.")

Theory of caustic and mild alkalies. The technology of heating limestone (mild calcia) to produce mortar (quicklime, caustic calcia) has been known for millennia.[8a] It was believed by Medieval chemists[9b] that causticity was induced by a "quantity of pure fire" that had been imparted to the limestone.[1a] By the 1700s a sophisticated theory had been developed by Johann Friedrich Meyer (1705–1765), a German apothecary in Osnabrück (located between Cologne and Hamburg).[1c] Adopting an idea from *terra pinguis*, the "fatty earth" which was the principle of combustibility and the forerunner of "phlogiston,"[9c] Meyer invoked an *acidum pinque* ("fatty acid"), a fiery principle which saturated mild alkalies to produce caustic alkalies. For example,

Chalk ($CaCO_3$) + "acidum pinque" → caustic lime (CaO, slaked lime)

The slippery feeling of a caustic alkali (such as sodium hydroxide, today's lye or "Drano") was explained by the saturation with this oily substance. Shortly afterward, this principle "acidum pinque" was given the more descriptive label of *causticum*.[10]

According to this theory of *causticum*, one could distinguish a mild alkali (such as sodium carbonate or calcium carbonate) from a caustic alkali (such as sodium hydroxide or calcium hydroxide) by adding acid. In this diagnostic test, mild alkalies would effervesce by absorption of causticum, while the caustic alkalies, already saturated with acidum pinque, did not effervesce. This effervescence was understood merely to be, in the parlance of the times, a "symptom of the violent movements caused by mutual saturation of acid and alkali."[10] Today, this effervescence is known to be generation of carbon dioxide and is the standard geologist field test for limestone ($CaCO_3$ + acid → CO_2).

However, there were gravimetric difficulties with this idea: when limestone calcined to form quicklime, it supposedly received causticum from the fire, but it *lost* weight. This loss of weight was known since the ancient Romans—but the significance was not recognized because it was considered to be a simple "loss of water."[8a]

Figure 5. A few sketches survive of the Old College in Edinburgh. This sketch[12b] was made in 1789, and is "Principal Robertson's house" (left) and the "Teviot chambers" (right, used for classrooms and residences). These can be identified as the southernmost buildings of the Old College Quadrangle (see Figure 6).

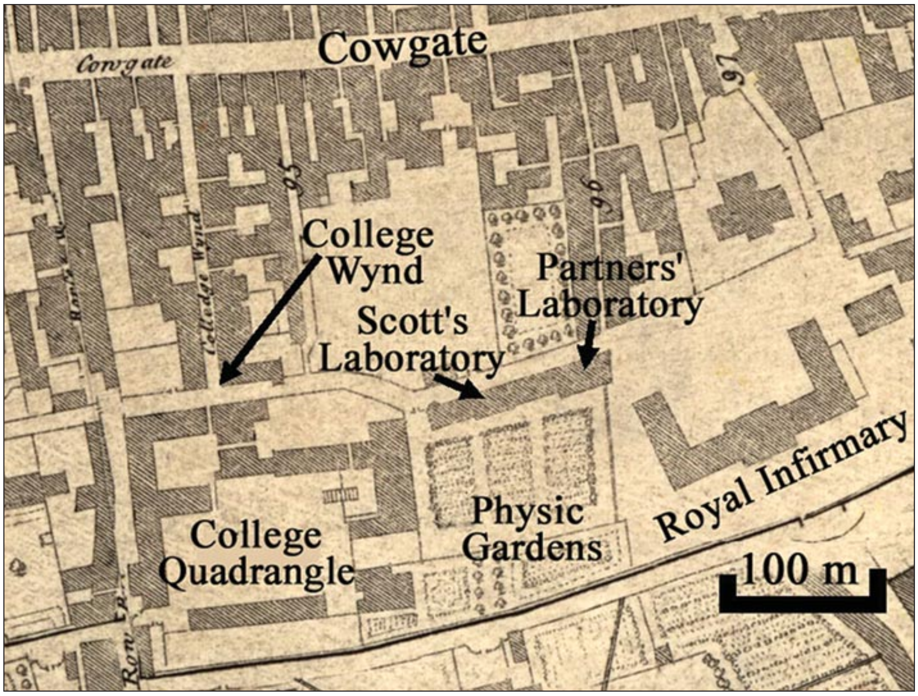

Figure 6. Edgar's 1765 map of the Edinburgh Old College area.[17,20] The College Quadrangle is the main campus of Old College. The "Physic [Medical] Gardens" grew medicinal herbs for patients in the Royal Infirmary. Black probably performed his magnesia research in the Infirmary.[15b,16] College Wynd [wynd = narrow alley] was the main entrance to the campus (see Figure 7). There was no easy access to City Center of Edinburgh; one had to cross Cowgate, essentially a ravine. South Bridge (see Figure 2), the south-north road linking with central Edinburgh, was built in 1788, passing directly through the Physic Gardens.

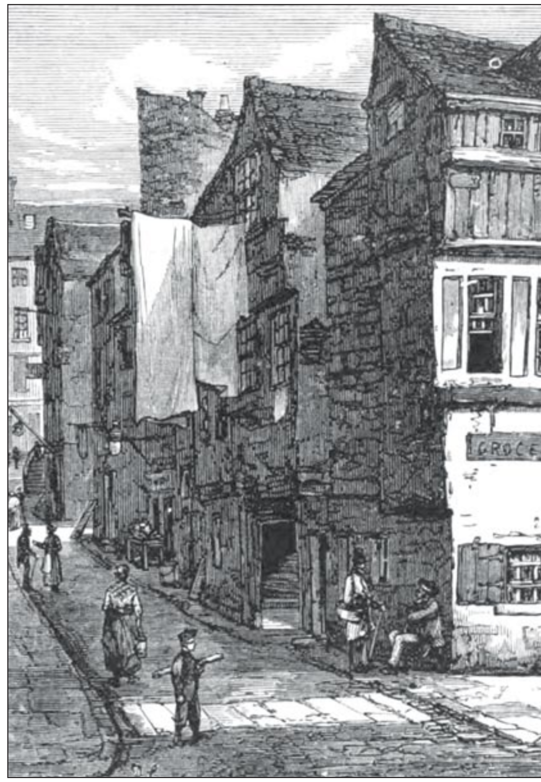

Figure 7. Black's earlier days were spent in College Wynd, a ramshackle district at the north entrance of the Old College. A "wynd" (rhymes with "find") is a narrow alley in Scotland. Nearby was the residence of the Scott family (where Sir Walter Scott was born in 1771),[21a] now marked with a plaque mounted high on a corner building (N55° 56.87 W03° 11.25). (Drawing by William Channing[21a].)

Black's research with carbonates. Black's work, presented in his medical dissertation at the University of Edinburgh,[11] has been described as "a brilliant model, perhaps the first successful model, of a quantitative chemical investigation, as well as a classical exemplar of an experimental science worthy of comparison with Newton's *Opticks*."[2a] Black's early notes[7] from 1751 show that first he had tested the idea that the fire imparted causticum to the chalk ($CaCO_3$) to produce the quicklime (CaO). Since quicklime becomes mild upon exposure to the air, then obviously the quicklime was supposedly losing this causticum to the atmosphere. Perhaps, he thought, one could catch this elusive principle in a bottle.[2b] Black set up an experiment whereby a dish filled with caustic lime (CaO) was allowed to float in water with an inverted glass vessel over it.[2a] Hoping this igneous matter might be collected, instead he observed that the air space above the quicklime, if anything, had been *reduced* in volume, making him suspicious that something had *removed* from the atmosphere. The fact that the quicklime had simultaneously increased in weight while converting to the mild form supported this idea.

Then Black measured the loss of weight of chalk when it was either calcined or treated with acid, and found it to be the same in either case.[1b] Why should two independent phenomena give the same quantitative result? Was there a mysterious air being produced, that was identical, in either calcination or acidification?

Black then reacted calcium oxide with potassium carbonate: "When I precipitated lime by a common alkali (i.e., $CaO + K_2CO_3 \rightarrow CaCO_3$), there is no effervescence: the air quits the alkali for the lime; but it is lime no more, but CCC [chalk]: it now effervesces, which good lime will not."[7] He concluded lime (CaO) contributes nothing to the alkalies; it only removed a peculiar kind of air that prevented their caustic properties from being developed.[7] Black was thus visualizing this fiery principle not as some elusive abstraction, but instead as a chemical entity, which could be passed to or from the atmosphere, or which in solution could silently pass between substances, changing their chemical identities. This was *the* key observation, according to Thomas Thomson (1773–1852), the Scottish chronicler of early 19th century chemical history: "What a multitude of important consequences naturally flowed from this discovery!"[7] (Figure 8).

Chalk and magnesia. Joseph Black then became interested in *magnesia alba* (Figure 9), then sold in Rome as a stomach remedy. Magnesia alba (magnesium carbonate, $MgCO_3$) had been known for several centuries but had been considered to be merely a variety of chalk (calcium carbonate, $CaCO_3$), even by such sages as George Ernst Stahl (1659–1734), the champion of phlogiston.[3b] (Note 1)

For his dissertation topic, why did Joseph Black choose obscure magnesia rather than well-known lime, which would have commanded more attention? A possible reason[2a] was that this mild-mannered scientist wanted to avoid contention between his advisor Alston and Robert Whytt (1714–1766), a "bright luminary in the rising University."[12a] Both Alston and Whytt had performed experiments on the medicinal effects of lime-water and had published on the subject, but there was a dispute of priority. Whytt, a specialist of the nervous system (he discovered the unconscious reflex reaction[2a]), was promoting his own "discovery," oyster-shell lime. Alston criticized Whytt's experimental methods and believed Whytt's oyster shells were no better than ordinary limestone.[2a] Black sidestepped the issue by concentrating on a different medicant.

Figure 8. In the Royal Museum of Scotland on Chambers Street (N55° 56.81 W03° 11.44), dating from 1851, is found this exhibit of Joseph Black, consisting of laboratory glassware, utensils, and bottles. The bottle in the lower left was used by Black to collect carbonic acid.[15d] Another Scottish scientist featured in the museum is Thomas Charles Hope (1766–1844), who fully characterized strontium.[3a]

Figure 9. This 0.44 kg specimen of magnesite (magnesium carbonate) is from Magnesia, Thessaly, Greece, and is the etymological source for magnesia alba *(white magnesia).[8b] Another mineral from the locality,* magnesia nigra *(black magnesia), pyrolusite (manganese dioxide) is the etymological origin of the element manganese.* Photo, elemental collection of the authors.

There was also disagreement between Alston and Whytt regarding the genesis of causticity. Whytt ardently maintained that indeed there *was* a "fiery substance" in caustic lime, while Alston favored some sort of chemical rearrangement. Black thought it would be "presumptuous to settle the quarrel between them"[2a]—but ironically, his research with magnesia did just that.

Black's experiments. For his experiments (Figure 10), Black carefully prepared pure *magnesia alba* ($MgCO_3$) from Epsom salt (Figure 11) and pearl ashes,

$MgSO_4$ (Epsom salt) + K_2CO_3 (pearl ashes) → $MgCO_3$ (magnesia alba) + K_2CO_3.

He separated the $MgCO_3$, dried it, and weighed it. Then he calcined the $MgCO_3$ to produce magnesia (MgO), resulting in a loss of 7/12 of its weight. He surmised the loss of weight was due to the expulsion of the same mysterious air, which he was able to collect and study. He demonstrated that this air, when bubbled into a solution of quicklime (CaO), precipitated a milky precipitate, identified as chalk ($CaCO_3$). Thus, the air could be "fixed," i.e., rendered nonvolatile. (This chemical procedure exists to this day as the classical laboratory test for carbon dioxide.) In subsequent class demonstrations, he fascinated his audiences by showing how the invisible "fixed air" could be literally poured, in the open atmosphere, from a glass vessel onto a candle to extinguish it.

By a series of experiments he showed that:[1b]

magnesia alba ($MgCO_3$) + heat → calcined magnesia (MgO) + fixed air (CO_2)

magnesia alba ($MgCO_3$) + acid (HCl) → magnesia salt ($MgCl_2$) + fixed air (CO_2)

calcined magnesia (MgO) + acid (HCl) → the same magnesia salt ($MgCl_2$)

Black repeated these experiments with lime, and then continued on with the other three alkalies—vegetable, marine, and volatile—and showed that in each case each mild form was simply the caustic form plus fixed air. He developed a general schedule of reactions that applied consistently for all groups. For example, he showed[1b] that reaction of "mild ammonia" with slaked (caustic) lime gives smelly (caustic) ammonia and chalk precipitate:

Mild ammonia ($(NH_4)_2CO_3$) + caustic lime (CaO) → caustic ammonia (NH_3) + chalk ($CaCO_3$)

(solution) + (solution) → (smelly solution!) + (milky precipitate)

Black's full characterizations with magnesium and calcium salts showed the two were definitely different. Thus, Black was the first to establish magnesium as a separate entity, and not just a variation of the calcareous earth.[1b] (Note 2).

Black's research further clarified the nature of the alkaline earth medicants. Previously,

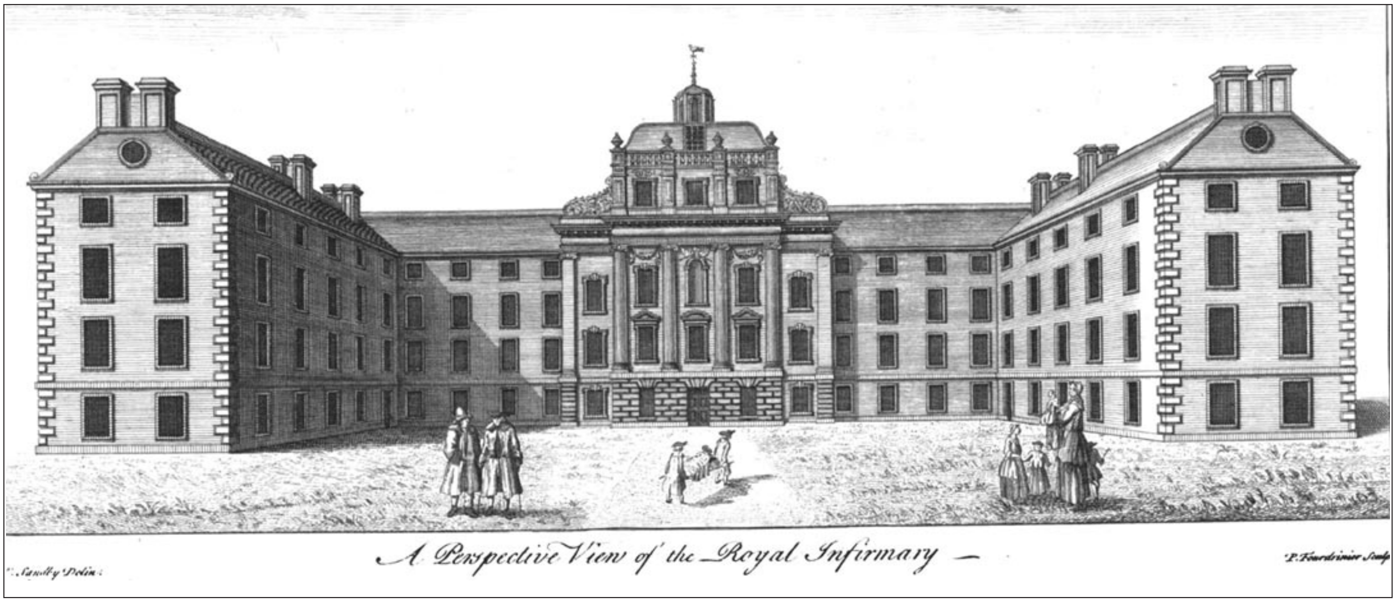

Figure 10. This is the Royal Infirmary, where Black probably performed his carbonate studies for his M.S. dissertation. The Royal Infirmary was the foundation of the Medical Institute at Edinburgh because it allowed medical studies with patients, as well as provided teaching and research facilities. It was built in 1741 and demolished in 1884. (Engraving by Paul Sandby.[21b])

"magnesia alba" or "magnesia" could each refer to either magnesium oxide (MgO) or magnesium carbonate ($MgCO_3$). Likewise, "lime" could mean "quicklime" (CaO) or chalk ($CaCO_3$). No doubt many pharmacological formulations of the 1700s were mixtures. However, after Black's defining research, the descriptions in the pharmacopoeias became unambiguous: CaO was "calcined calx" or "calx usta," MgO was "calcined magnesia" or "magnesia usta" ("usta" is an old Latin word meaning "cremated"); and the mild calcareous alkalies were "carbonas calcis" ($CaCO_3$) and "carbonas magnesiae" ($MgCO_3$).[13] Even today, "magnesia usta" is occasionally used to describe commercial MgO products.

Preamble to Lavoisier. A clear parallel exists between the works of Joseph Black and Antoine Lavoisier.[1b] Lavoisier considered combustion to be the addition of a "principe oxigène,"[1e] a real substance, as opposed to Stahl's of loss of chimeric phlogistron; Black construed calcining as the loss of a gas, instead of the gain of a "fire principle." In both cases, as acclaimed by Antoine Francois Fourcroy (1755–1809), Lavoisier's colleague and advocate, we need not invoke a "principe imaginaire" ("imaginary principle"); instead, we have an "être réel" ("real one")![14a]

In all of these studies, Black depended upon precise gravimetric analysis, perhaps the first to employ the balance totally "in almost every stage" of his chemical investigations.[15c] Fourcroy recognized Black's importance to Lavoisier's New Chemistry three decades later; Fourcroy called him "l'illustre Black, le chef et le Nestor de cette grande révolution chimique" ("the illustrious Black, the chief and the Nestor of the grand chemical revolution").[14b]

Black's laboratory. Where did Joseph Black perform his chemical experiments? This is not known for certain. There are two possibilities[15a,16,17] (see Figure 6): the Medical Institute laboratories ("Scott's and "Partner's") by the Physic Gardens, set up in 1724 to prepare medicines,[17] or the Infirmary where some lectures were held (Figure 10). The more probable choice is the Infirmary, because Black might not have been given access by the operators of the Physic Garden laboratories, owing to envy and internal rivalry.[15b,16] (Because of his successes and reputation, Joseph Black—a fresh M.D. graduate—had actually been proposed as a temporary Chair of Chemistry before Cullen returned to Edinburgh in 1766![12b, 16])

The nature of "fixed air." Black realized that air was a distinct species "dispersed thro' the atmosphere"[2b] and probably was the same *aer malignus* (malignant air) that had often been mentioned under the name of choke-damp, gas sylvestre, spiritus sylvestre, mephitic air, and gas carbonum, among others. He thought this gas was the same as that in the bubbly spas at Pyrmont in Germany[3d] and in caves such as Grotta del Cane[9a,18] (Cave of the Dog) near Naples, Italy (N40º 49.62 E14º 10.32). In Grotta del Cane, a meter blanket of a noxious gas (carbon dioxide) lay low in the cave, harmless to a human being but deadly to a canine at his feet.

Figure 11. Epsom Wells is marked by this monument in the center of a spiral road in Well Way (N51° 19.63 W00° 17.41), located 25 km southwest of London. Epsom Wells, a source of the eponymous salts (magnesium sulfate), was a fashionable spa in the 1600s and 1700s, known for the healthy benefits of its waters (mostly to prevent constipation). In 1851 the well was closed owing to pollution. The building in the background is a nursing home.

(Mark Twain in *Innocents Abroad* reported that he wanted to try the experiment at Grotta del Cane but complained that he "had no dog.")[19] Black also recognized this gas was the same as that produced by fermentation or the burning of charcoal.

Black intended to pursue the "serious study" of "fixed air and similar elastic fluids."[2b] However, a "load of new official duties was laid upon [him]"[2b] and he never resumed this research. Later, Antoine Lavoisier would establish the chemical composition of carbon dioxide (1774),[3b] but in the meantime Joseph Black suspected there might be more to "mephitic air" than simply "fixed air." Shortly after returning to Edinburgh in 1766, he assigned the task of a more comprehensive study of mephitic air to Daniel Rutherford (1749–1819), son of John Rutherford (1695–1779), one of the original founders of the Medical Institute at Edinburgh. Daniel completed his work 17 years after Black's original dissertation; he reported his results in his own dissertation of 1772. In the next issue of *The HEXAGON*, we will see that Daniel discovered a new element in this gas—nitrogen.

Notes

1. Andreas Sigismund Marggraf (1709-1782),[3c] apparently unaware of Black's work, also distinguished magnesia from lime four years later.[9e]

2. A preliminary chemical distinction between magnesia and calcia had been made in 1722 by Friedrich Hoffmann[9d] (1660–1742), the first chair of medicine at Halle, the same university as Stahl's.[3b] A difference was observed in solubility and taste of the sulfates (calcium insoluble, tasteless; magnesium soluble, bitter).

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On behalf of everyone involved in the editorial and production end of *The HEXAGON*, we extend our deepest and most sincere condolences to Professor Jim Marshall, *Beta Eta 1971*, on the loss of Jenny Marshall, *Beta Eta 2003*, his dear and adventuresome life partner, and co-author of the Rediscovery of the Elements series. The artful work of her keen eye, behind the lens of a camera, has graced the cover of *The HEXAGON* for many years.