XXXIX. *Chemical Experiments and Obſervations on Lead Ore.* *By* Richard Watſon, *D. D. F. R. S. in a Letter to Sir* John Pringle, *Bart. P. R. S.*

TO SIR JOHN PRINGLE, BART. P. R. S.

S I R,

Cambridge,
June 13, 1777.

Read July 9, 1778.

THE following experiments and obſervations, it is apprehended, will not be thought unintereſting by perſons verſed in chemiſtry. May I beg the favour of you to communicate them to the Royal Society?

I am, &c.

LEAD ORE, as dug out of the mine, is generally much mixed with ſpar, lime-ſtone, and other ſubſtances, bulk for bulk, heavier than the ore itſelf. It undergoes various dreſſings before it becomes a merchantable commodity, the general tendency of which is to free it, as much as poſſible, from every heterogeneous impurity.

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Suppose that a cubic foot of lead ore, which contained no ſpar or other extraneous matter, would weigh 7800 ounces, and that a cubic foot of ſpar, which contained no lead ore or other foreign ſubſtance, would weigh 2700 ounces, then would a mixture, conſiſting of a cubic foot of pure lead ore and a cubic foot of pure ſpar, weigh 10500 ounces, and one cubic foot of ſuch a mixture would weigh 5250 ounces. It is obvious that, according to the different proportions in which the particular kinds of ſpar and lead ore here aſſumed are ſuppoſed to be mixed together, a cubic foot of the mixture will have different weights, the limits of which are on the one hand 7800, and on the other 2700 ounces; it never can weigh ſo little as 2700 ounces, for then it would conſiſt intirely of ſpar without any lead ore; nor can it ever weigh ſo much as 7800 ounces, for then it would conſiſt intirely of lead ore without any ſpar.

From this view of the matter it is evident, that the purchaſing of lead ore by the meaſure, which is the general though not the univerſal cuſtom in Derbyſhire, is a mode liable to ſome exception; ſince a diſh, containing any definite meaſure, muſt have different weights, according as the ore with which it is filled is more or leſs free from ſpar. And it is ſcarce poſſible, by repeated

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dreſſings, to ſeparate all the ſpar from an ore, or equal portions of it from equal portions of ore.

There is a diverſity, however, in the weights of equal meaſures of lead ore, which probably does not ariſe from ſparry or other heterogeneous accretions, but from the nature of the ore itſelf. I have carefully taken the ſpecific gravities of many of the Derbyſhire lead ores; the weight of a cubic foot of the lighteſt which I met with was 7051 ounces, and the weight of a cubic foot of the heavieſt was 7786 ounces; the difference amounting to between a ninth and a tenth part of the weight of the lighteſt. There are probably other ores of lead which differ more in their ſpecific gravities than theſe here mentioned; but the difference between theſe is ſufficient to ſhew the great uncertainty of purchaſing lead ore by the meaſure, ſince ten diſhes of one ſort of ore may not weigh more than nine diſhes of another ſort, though both the ſorts be equally well dreſſed.

Lead ore is not always of the ſame goodneſs in the ſame mine, nor even in the ſame part of the ſame mine; and, what is more remarkable, the different parts of the ſame lump of ore have different ſpecific gravities. I could not eaſily have believed this, unleſs a variety of experiments had convinced me of the fact.

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They were employed this year at Holywell in ſmelting a lead ore from the Iſle of Man; the ore was rich in ſilver. A lump of this ore, weighing about ten ounces, was broken into ſeveral pieces, and ſuch of the pieces were ſelected as appeared to the eye to be wholly pure. By taking the ſpecific gravities of ſix of theſe pieces I found, that a cubic foot of the lighteſt kind would have weighed 6565 ounces, and a cubic foot of the heavieſt kind would have weighed 7636 ounces. Suppoſing the ſpecific gravity of water to be denoted by 1,000, the mean ſpecific gravity of the ſix different pieces of this ore may be expreſſed by 7.115.

A very pure ſpecimen of teſſellated lead ore, from a mine near Aſhover in Derbyſhire, was broken into ſix pieces, weighing near one ounce each. A cubic foot of the lighteſt of theſe pieces would have weighed 7326 ounces, and a cubic foot of the heavieſt would have weighed 7786 ounces. The mean ſpecific gravity of the ſix pieces was 7.566.

At the ſame mine they frequently meet with ſmall quantities of ſteel-grained lead ore. Six different pieces of the ſame lump of this kind of ore were choſen, each of which appeared quite free from ſpar and every other impurity. A cubic foot of the lighteſt of theſe pieces would have weighed 7188 ounces, and a cubic foot of

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the heavieſt would have weighed 7442 ounces. The mean ſpecific gravity of the ſix pieces was 7.342.

Other lumps of ore, from different mines, were reſpectively broken into different pieces, and ſcarcely any two pieces of the ſame lump were obſerved to agree in their ſpecific gravities. This diverſity in the ſpecific gravities of the ſeveral pieces of the ſame lump of ore may be owing, either to the different proportions in which the conſtituent parts of the ore are combined in the ſeveral pieces; or to the different quantities of extraneous ſubſtances imperceptibly mixed with them, or, which ſeems moſt probable, to a diverſity in the ſize or configuration of theſe pores.

But be the cauſe of the diverſity in the ſpecific gravities of different pieces of the ſame lump of ore what it may, the fact, I believe, is certain, and by no means ſingular; for not to mention the varieties obſervable in the ſpecific gravities of different pieces of roll brimſtone, of corroſive ſublimate, of caſt ſteel, and other factitious ſubſtances, the natural ſpars generally found along with lead ore are ſubject to a ſimilar diverſity, though not perhaps in an equal degree.

A piece of rhomboidal, otherways called refracting or lantern ſpar, was broken into four ſmaller pieces, the ſpecific gravities of which were 2.675, 2.687, 2.715, 2.723; the medium of the four is 2.700. Mr. COTES fixes the ſpecific gravity of Iceland cryſtal at 2.720, and WALLERIUS fixes it at 2.700.

The ſpecific gravities of four pieces of the ſame lump of cubical ſpar were 3.204, 3.218, 3.222, 3.231; the medium of the four is 3.219.

Six ounces of fine teſſellated lead ore were put into a crucible and expoſed, at firſt, to a gentle, afterwards to a ſtrong fire: the ore grew red, and emitted fumes which ſmelled of ſulphur; at length it melted, and the fumes became very copious; they were accompanied with a yellowiſh flame upon the ſurface of the melted ore, and when collected had a whitiſh appearance. The crucible after the ore had continued a full hour in perfect fuſion was taken from the fire, and when it was cold it was broken. The maſs which it contained weighed five ounces and an half; there were no ſcoria obſervable on its ſurface, nor were any particles of metal formed, it was ſtill an ore of lead.

The maſs remaining from the laſt experiment was put into a freſh crucible, and expoſed to a ſtrong melting heat; the fumes which aroſe from it ſeemed to be heavy; they brooded over the ſurface of the melted maſs in undulating flames, which now and then appeared like burning zinc. The lead was now formed, and many

particles

particles of it were ſublimed to at leaſt ſix inches above the ſurface of the liquid in the crucible. After letting the crucible continue two hours in this ſtate, I poured out its contents, and found them conſiſting partly of lead, partly of lead ore, and partly of a very minute portion of browniſh ſcoria.

Theſe experiments prove, that ſome ſubſtance or other is contained in lead ore, which muſt be diſperſed before the ore can be formed into lead; and they ſhew too, that it requires a conſiderable time to effect the diſperſion of this ſubſtance, ſince ſix ounces of ore, though kept three hours or more in complete fuſion, were not wholly brought into the form of lead; and, laſtly, they render it probable, that the fumes, ariſing from melted ore, carry off with them no inconſiderable portion of the lead itſelf. At the great ſmelting houſes in Derbyſhire, they put a ton of ore at a time into the furnace, and work it off in eight hours; the ore might be wholly melted in one hour, but the lead perhaps is not formed in the greateſt poſſible quantity in eight hours.

Some fine teſſelated lead ore from Derbyſhire was pounded into ſmall lumps, each about the ſize of a pea, and carefully picked from ſpar and other impurities. Sixteen ounces of this ore, thus previouſly cleanſed, were diſtilled in an earthen retort; as ſoon at the ore felt the

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fire, the ſtopple of the quilled receiver had a ſtrong ſmell reſembling that of the inflammable air ſeparable from ſome metals by ſolution in acids; ſoon after a ſmall portion of a liquid came over into the receiver; the fire was then raiſed till the retort was of a white heat, when a black matter began to be ſublimed into the neck of the retort; the operation was then diſcontinued. This experiment was undertaken with a view of ſeeing whether ſulphur could be ſeparated from lead ore, as it may be from ſome ſpecies of the pyrites, by diſtillation, and it appears from the iſſue of the experiment that it cannot. What might have been the event of the experiment if it had been conducted with a very gentle heat for a long time, I cannot pretend to ſay. Upon breaking the retort I found, that the ore had been melted during the operation, for there was a conſiſtent cake of ore of the figure of the bottom of the retort; the weight of this cake was fifteen ounces and an half, the weight of the liquid in the receiver, and of the black matter which had been ſublimed, did not together amount to one quarter of an ounce, ſo that a quarter of an ounce or more had been diſperſed, probably in the form of air, or ſome elaſtic fluid. The ore by this proceſs had loſt one thirty-ſecond part of its weight; but I am of opinion, that if the operation had been conducted with a leſs degree of heat and continued

continued for a longer time, the quantity of liquid would have been augmented. The liquid did not efferveſce with either acids or alkalies; nor did it produce any change in the colour of blue paper, though I am certain, from experiment, that one drop of oil of vitriol, though diluted with two ounces of water, would have produced a ſenſible redneſs on the blue paper which I uſed. The liquid, notwithſtanding, had an acid taſte, and a pungent ſmell, reſembling that of the volatile vitriolic acid. The experiment ought to be repeated with a larger quantity of ore, in order to aſcertain the quantity and quality of the liquid, ſeparable therefrom by ſimple diſtillation. The black matter which had been ſublimed into the neck of the retort, was examined with a microſcope, and it appeared to be pure lead ore; hence it is probable, that by a due degree of heat in cloſe veſſels, lead ore might be intirely ſublimed without being decompoſed; for the melted ore which was found at the bottom of the retort, had not any appearance of either ſcoria, or of lead, upon its ſurface. Finding that ſulphur could not be ſeparated from lead ore by diſtilling it without addition, and yet being much diſpoſed to think, that it contained a conſiderable portion of ſulphur, I firſt thought of diſtilling it with charcoal duſt, iron filings, ſand, and other additions; but recollecting that ſulphur might be ſeparated from anti-

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mony by ſolutions in acids, I thought it not improbable, that it might be ſeparated from lead ore by the ſame means, and the ſucceſs of the following experiment abundantly juſtified the conjecture.

Upon ten ounces of lead ore, cleanſed as in the preceding experiment, I poured five ounces of the ſtrongeſt fuming ſpirit of nitre; this ſtrong acid not ſeeming to act upon the ore, I diluted it with five ounces of water; a violent ebullition, accompanied with red fumes, immediately took place; the ſolution of the ore in this menſtruum became manifeſt, and when it was finiſhed, there remained floating upon the ſurface of the menſtruum a cake of fine yellow ſulphur, perfectly reſembling common ſulphur.

I repeated this experiment a great many times, in order to aſcertain the quantity of ſulphur contained in lead ore, and ſeparable therefrom by ſolution in acid of nitre. The reſults of different experiments were ſeldom the ſame: the matter ſeparable from the ore by ſolution, after being repeatedly waſhed in large quantities of hot water, in order to free it from every ſaline admixture, ſometimes amounted to more, ſometimes to leſs than one-third the weight of the ore. This matter may, for the ſake of diſtinction, be called crude ſulphur. Its apparent purity might induce a belief that it contained no hetero-

heterogeneous mixture, yet the following experiments ſhew how much we ſhould be deceived in forming ſuch a conjecture, and how rightly it is denominated crude ſulphur.

From one hundred and twenty parts, by weight, of lead ore, I obtained, by ſolution in acid of nitre, ſubſequent waſhing in hot water, and drying by a gentle fire, forty parts of a ſubſtance which looked like ſulphur: theſe forty parts were put on a red-hot iron, the ſulphur was made manifeſt by a blue flame and pungent ſmell. When the flame went out, there remained upon the iron unconſumed twenty-ſix parts of a greyiſh calx; the weight of the ſulphur which was conſumed muſt therefore have amounted to fourteen parts, or between one eighth and one ninth part of the weight of the ore. It has been obſerved, that the weight of the matter, ſeparable from lead ore by ſolution in acid of nitre, ſometimes exceeded, and ſometimes fell ſhort of, one third part of the weight of the ore; this variety, as far as I have been able to obſerve, does not extend to the quantity of ſulphur contained in a given quantity of ore, but depends upon the quantity of calx remaining after the burning of the ſulphur. Different lead ores will, doubtleſs, contain different quantities of ſulphur; but that the ſulphur contained in the lead ore which I examined conſtitutes between one eighth and one ninth part of the weight of

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the ore, is a conclufion upon which, from a variety of experiments, I am difpofed to rely.

There are annually fmelted in Derbyfhire about ten thoufand tons of lead ore: now if means could be invented (which I think very poffible) of faving the fulphur contained in ten thoufand tons of ore, fuppofing that the ore fhould only yield one tenth of its weight of fulphur, though it unqueftionably contains more, Derbyfhire alone would furnifh annually one thoufand tons of fulphur, the value of which would annually be about fifteen thoufand pounds. I mention this circumftance thus publicly, in hopes that the lead fmelters may be induced to profecute the object. If the fulphur contained in lead ore could be collected, it would not only be a lucrative bufinefs to the fmelters, but a great faving to the nation. We at prefent import the fulphur we ufe, and the confumption of this commodity is exceeding great, in the making of gunpowder, in forming the mixture for covering the bottom and fides of fhips, and in a great variety of arts. The fmelters need not be apprehenfive left the quality of the ore fhould be injured by extracting the fulphur. Eighteen hundred weight of ore, from which the fulphur has been extracted, will certainly yield as much lead as twenty hundred weight of ore, from which the fulphur has not been extracted, and it will probably yield more.

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Arsenic is extracted from a particular ore in Saxony, by roasting the ore in a furnace, which has a long horizontal chimney; the chimney is large, has many windings and angles, that the arsenical vapour which arises from the ore may be the more easily condensed: the arsenic attaches itself like soot to the sides of the chimney, and is from time to time swept out. It is very probable, that by some such contrivance the sulphur contained in lead ore might be collected. The smelters call every thing sulphur which is volatilized during the roasting or fluxing of an ore; but none of those with whom I have conversed had any notion that common sulphur could be separated from lead ore.

The greyish calx which remained upon the iron after the sulphur was consumed, was put upon a piece of lighted charcoal; the heat of the charcoal being quickened by blowing upon it, a great number of globules of lead were formed upon its surface. From hence it appears, that this calx is not an unmetallic earth contained in the ore, which the acid of nitre could not dissolve; but a calx of lead, probably produced by the violent action of the acid, and which, by the addition of phlogiston, may be exhibited in its metallic form. The quantity of this calx depends much upon the action of the acid upon the ore; if that action is violent, the calx is in greater abundance than if it be moderate; and I am not certain whether the experiment might not be ſo managed, that there would be little or no calx remaining; that is, a given quantity of ore might be ſo diſſolved in the acid of nitre, that nothing would remain undiſſolved except the ſulphur. But I have not yet perfectly ſatisfied myſelf as to the conſtituent parts of lead ore. I am certain that it contains lead and ſulphur, a liquid and air: of the exiſtence of the three firſt there can be no doubt, from what has been ſaid, and the air is rendered beautifully apparent by the following experiment.

Let ſome lead ore be reduced into a fine powder, put it into a narrow-bottomed ale glaſs, fill the glaſs three parts with water, drop into the water a portion of the ſtrong acid of nitre, you may judge of the requiſite quantity by ſeeing the ſolution commence, and you will obſerve the ore univerſally covered with bubbles of air, theſe will buoy the ore up in large tufts to the ſurface, and the air will continue to be ſeparated from the ore till the acid becomes ſaturated with the lead. The ſalt ariſing from the union of the nitrous acid to the lead often appears cryſtallized upon the ſurface of the menſtruum in this experiment; and if, when the menſtruum is in that ſtate, a little freſh acid be added, the ſalt inſtantly cryſtallizes and falls down to the bottom of the

glaſs,

glaſs, the acid having abſorbed the water which held it in ſolution. When lead is diſſolved in the manner here mentioned, by a very diluted acid of nitre, there is no appearance of ſulphur upon the ſurface of the menſtruum, there is found at its bottom a black matter which is the ſulphur.

But though lead and ſulphur, a liquid and air, are unqueſtionably conſtituent parts of lead ore, I do not take upon me to ſay, that they are the only conſtituent parts: it is well known, that, during the ſmelting of lead ore, a third part or more of its weight is ſomehow or other loſt, ſince from one and twenty hundred weight of ore they ſeldom obtain above fourteen hundred weight of lead. What is loſt partly conſiſts of a ſcoria which floats upon the ſurface of the lead during the operation of ſmelting, and partly of what is ſublimed up the chimney and diſſipated in the air. The ſcoria, I apprehend, would be very little even from a ton of ore, if the ore was quite free from ſpar: it is the ſpar which is mixed with the ore that conſtitutes the main portion of the ſcoria. I have in my poſſeſſion a ſolid maſs of ſcoria, which accidentally flowed out from a ſmelting furnace, and which in colour and conſiſtency perfectly reſembles grey lime-ſtone; it receives a poliſh as fine as marble, and it might, perhaps, with advantage be caſt into molds for paving ſtones, chimney pieces, and other matters. It arifes from the fpar mixed with the ore, and, by the addition of fufible fpar to the ore during its fufion, its quantity might be increafed at no great expence, in any proportion. That part of the ore which is fublimed and difperfed in the air, confifts partly of the fulphur which is confumed, and partly of lead; this fublimed lead attaches itfelf in part to the fides of the chimney of the fmelting furnace; the reft of it flies up into the air, from whence it falls upon the ground, poifoning the water and herbage upon which it fettles. This fublimed lead might be collected either by making it meet with water, or with the vapour of water during its afcent, or by making it pafs through an horizontal chimney of a fufficient length.

It is not eafy to determine with precifion the quantity of this fublimed lead; a general guefs, however, may throw fome light upon the fubject. They ufually at a fmelting houfe work off three tons, or fixty hundred weight, of lead ore every twenty-four hours; the fulphur contained in fixty hundred weight of ore we will fuppofe to be feven hundred weight, and the lead to be forty hundred weight; the air, liquid, fcoria, and fublimed lead, muft together, upon this fuppofition, amount to thirteen hundred weight; now, admitting three hundred weight of the thirteen to be fublimed lead, it is evident

evident that, could it be collected, there would be an annual ſaving at each ſmelting houſe of above fifty tons, which, ſuppoſing it to be worth four pounds *per* ton, would amount to above two hundred pounds a year. The price and quantity of lead ſublimate here aſſumed are probably both of them below the truth; but my end is anſwered in giving this hint to perſons engaged in the ſmelting buſineſs.

The following experiments, though upon a different ſubject, may not be unacceptable to the lovers of chemiſtry, as I do not remember to have any where met with them. I trouble the Society with a relation of them at this time, that I may not hereafter intrude upon their leiſure.

It is commonly known, that the ſurface of melted lead becomes covered with a pellicle of various colours. I undertook ſome experiments in the courſe of laſt winter, with a view to aſcertain the order in which the colours ſucceeded each other. The lead which lines the boxes in which tea is imported from China happening to be at hand, ſome of it was melted in an iron ladle; but I was much ſurprized to find that its ſurface, though it was preſently covered with a duſky pellicle, did not exhibit any colours. Imagining that the heat was not ſufficiently ſtrong to render the colours viſible, the fire was urged till the ladle became red-hot, the calcined pellicle

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upon the ſurface of the lead was red-hot alſo, but it was ſtill without colour. The ſame parcel of lead was boiled in a crucible for a conſiderable time; during the boiling a copious ſteam was diſcharged, and the ſurface of the lead, as is uſual, became covered with a half vitrified ſcoria. The lead which remained unvitrified was then examined, and it had acquired the property of forming a ſucceſſion of coloured pellicles during the whole time of continuing in a ſtate of fuſion.

Another portion of the ſame kind of lead was expoſed to a ſtrong calcining heat for a long time; the part which remained uncalcined did, at length, acquire the property of exhibiting colours ſufficiently vivid.

Theſe experiments induced me to conclude, that the Chineſe lead was mixed with ſome ſubſtance from which it was neceſſary to free it, either by ſublimation or calcination, before it would exhibit its colours. It would be uſeleſs to mention all the experiments which I made before I diſcovered the heterogeneous ſubſtance with which I ſuppoſed the Chineſe lead was mixed. At laſt I hit upon one which ſeems fully ſufficient to explain the phenomenon. Into a ladle full of melted Derbyſhire lead, which manifeſted a ſucceſſion of the moſt vivid colours, I put a ſmall portion of tin, and obſerved, that as ſoon as the tin was melted, and mixed with the lead, no

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more colours were to be ſeen. I do not know preciſely the ſmalleſt poſſible quantity of tin, which will be ſufficient to deprive a given quantity of lead of its property of forming coloured pellicles, but I have reaſon to believe, that it does not exceed one five thouſandth part of weight of the lead.

Derbyſhire lead, which has loſt its property of exhibiting colours by being mixed with tin, acquires it again, as is mentioned of the Chineſe lead, by being expoſed to a calcining heat for a ſufficient time; the tin it is ſuppoſed being ſeparated from the lead by calcination before all the lead is reduced to a calx.

Some calcined Chineſe lead was reduced to its metallic form by burning ſome tallow over it. The reduced lead gave, when melted, coloured pellicles; the calx of tin, which we ſuppoſe to have been mixed with the calcined lead, not being ſo eaſily reducible as that of lead.

I find that zinc is another metallic ſubſtance which has the ſame property as tin with reſpect to the depriving lead of its power of forming coloured pellicles; but it does not, I think, poſſeſs this power in ſo eminent a degree as tin. I put ſmall portions of biſmuth alſo into melted lead, but the lead ſtill retained its quality of forming colours. I melted together ſome ſilver and lead, but the lead did not thereby loſe its power of forming colours.

 A little

A little tin added to a mixture of lead and bifmuth, or to a mixture of filver and lead, immediately takes away from the refpective mixtures the faculty of forming coloured pellicles.

This quality of tin has hitherto, as far as I know, been unobferved; but every new fact, relative to the actions of bodies one upon another, ought to be recorded. The change produced in lead by the admixture of a fmall portion of tin is much felt by the plumbers, as it makes the metal fo hard and harfh, that it is not without difficulty they can caft it into fheet lead. If their old lead does not work fo willingly, nor exhibit colours fo readily, as new lead, they may refer the difference to the fmall quantity of tin contained in the folder, from which old lead can feldom be thoroughly freed.

With refpect to the order in which the colours fucceed one another upon the furface of melted lead, it feems to be the following one; yellow, purple, blue, yellow, purple, green, pink, green, pink, green. Upon exhibiting the bright furface of melted lead to the air, I have often obferved thefe ten changes to follow one another in a more or lefs rapid fucceffion, according to the degree of heat prevailing in the lead. If the heat is but fmall, the fucceffion ftops before it has gone through all the changes; but with the greateft heat I did not obferve any

further

further variation. All the colours are very vivid, and each ſeems to go through all the ſhades belonging to it before it is changed into the next in order.

The formation of theſe colours may be explained from what has been advanced by Sir ISAAC NEWTON, and illuſtrated by the very ingenious experiments of Mr. DELAVAL, relative to the ſize of the particles conſtituting coloured bodies.