

LAB REPORT INVESTIGATING THE OXIDATION REACTION OF MAGNESIUM

INCLUDING MINI REPORT TO DISCUSS IR SPECTRUM ANALYSIS METHODS

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PURPOSE OF EXPERIMENT

THEORY

Elemental magnesium forms 'metallic bonding' with its own atoms. As it is in group 2 it has 2 valence electrons that become delocalized and enter a 'sea' of electrons that exists within an array of the positive 2+ magnesium nuclei [1] seen in Figure 1.

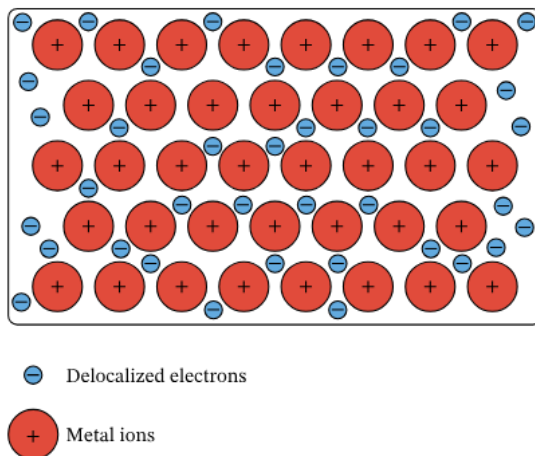


FIGURE 1 - ELEMENTAL MAGNESIUM ATOMS FORMING IONIC METALLIC BONDING [2]

Oxygen belongs to group 6 and thus requires gaining 2 electrons to fill the valence shell. At standard room temperature and pressure (STP), oxygen atoms form covalent double bonds by 'sharing' 2 valence electrons with another oxygen atom as the second atom does the same, seen in Figure 2.

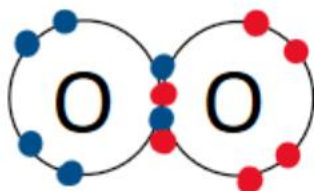


FIGURE 2 - OXYGEN ATOMS FORMING COVALENT DOUBLE BONDS

The reaction of magnesium and oxygen requires activation energy to 'break' the metallic and covalent bonds described above, enabling ions to be available for reaction. The activation energy in this experiment is supplied by external heating of the material by a Bunsen burner. As magnesium seeks to 'lose' 2 electrons and oxygen gain them to form full valence shells, ionic bonding as seen in Figure 2 occurs.



FIGURE 2 - LEWIS STRUCTURE FORMULA OF MAGNESIUM OXIDATION IONIC BONDING

These ionic bonds have less energy than the former metallic and covalent bonds, as such, energy is released from the formation due to the law of conservation of energy; characterizing the reaction as exothermic. The change in energy of this reaction involving activation energy and heat output is shown in Figure 3.

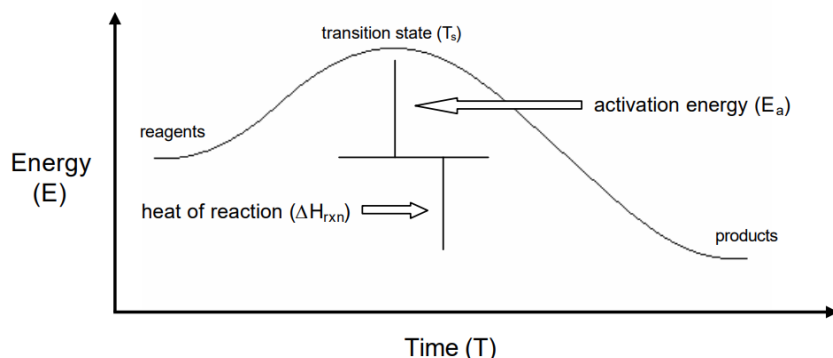
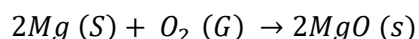


FIGURE 3 - ENERGY-TIME GRAPH OF AN EXOTHERMIC REACTION [3]

The law of definite proportion is shown in Figure 2, meaning that 1 Magnesium atom reacts with 1 Oxygen atom in a 1:1 ratio to form magnesium oxide. As discussed, Oxygen forms diatomic O_2 molecules at STP. Following stoichiometry, which is underpinned by the conservation of mass and energy to determine that the proportion of elements in the reactants must be the same as in the products [4]. As we have 2 oxygen atoms, to achieve the 1:1 ratio of magnesium oxide we must have 2 magnesium atoms available for the reaction and produce 2 moles of magnesium oxide.



EQUATION 1 - BALANCED EQUATION OF MAGNESIUM OXIDE FORMATION

MOLES

Moles are a unit of measurement used to provide a macroscopic view of the number of atoms in a particular amount of a substance. It is defined by the number of atoms/molecules as there are in 12g of ^{12}C (Carbon-12 isotope) [5]. This number is called the Avogadro's constant and has a value of $N_A = 6.02214076 \times 10^{23}$ [6]. The number of particles within a substance can therefore be calculated by $N = n \times N_A$, where N is the number of particles and n is the number of moles of the substance.

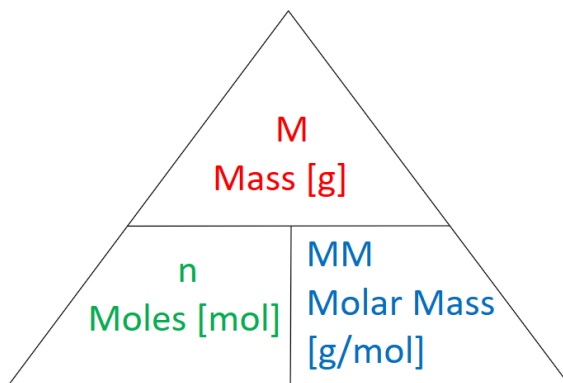


FIGURE 4 - MULTIPLICATION DIVISION TRIANGLE DESCRIBING RELATIONSHIP BETWEEN MASS, MOLES AND MOLAR MASS

Figure 4 shows the relationship between mass of a substance in grams and number of moles. Molar mass can be found on the periodic table as the atomic number and is calculated by adding the number of protons within an atom and the number of neutrons, $A = Z + n$ or *Mass number = no. of protons + no. of neutrons*. N.B. the number of neutrons can vary between isotopes of atoms and therefore so can the atomic number and molar mass. The unit of molar mass is grams per mole [g/mol].

As such the number of moles of a substance can be found with the following equation:

$$n [\text{mol}] = \frac{\text{Mass [g]}}{\text{Molar Mass [g/mol]}}$$

EQUATION 2 - CALCULATION OF NUMBER OF MOLES WITHIN A MASS OF SUBSTANCE

APPLICATIONS IN OME & BEYOND

All explosives release energy by the oxidation of a fuel. Conventional explosives consist of a self-contained molecule of a fuel and oxidizer, the oxidizer typically being a nitrate group NO_3 [7]. As the oxygen is integral to the molecule, in contrast with the burning of magnesium in atmosphere, conventional explosives can react much more rapidly due to the density of available fuel and oxygen.

Stoichiometry is important in understanding the oxygen balance of conventional explosives. A zero or positive oxygen balance means that all of the carbon and hydrogen within the molecule will result in products of carbon dioxide and water [8]. This means the detonation is more efficient (complete) and therefore powerful compared to an explosive with a negative oxygen balance. An additional benefit of a non-negative oxygen balance is that no carbon monoxide, a toxic gas, is produced.

The brilliant white light produced when magnesium is burnt is vital to many illumination pyrotechnic compositions. The high burning temperature is also desirable in certain applications such as rocket motor ignitors [9].

PROCEDURE

HAZARDS AND PPE

Magnesium is a flammable solid. When in hot water it reacts to form an alkali solution of magnesium hydroxide and gives off hydrogen gas which is extremely flammable [2].

The other hazard of this experiment is handling the hot crucibles. This is avoided by allowing the crucibles sufficient time to cool down but tongs will be available if the items are required to be moved before this point.

These hazards are mitigated by standard laboratory PPE (Lab coat, protective eyewear and disposable gloves) but most importantly, good laboratory practices and awareness.

EQUIPMENT

- Heatproof mat, tripod and clay triangle
- Crucible with lid and tongs
- Magnesium chips
- Balance accurate to 1×10^{-4} kg

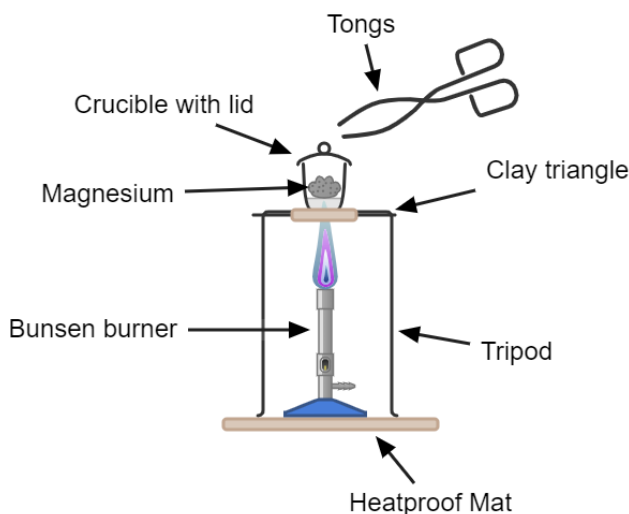


FIGURE 5 - EXPERIMENT EQUIPMENT SETUP

METHOD

1. Ensure you have the correct PPE on (Laboratory coat, gloves and goggles).
2. Set up the heat-proof mat, Bunsen burner, tripod and clay triangle as shown in Figure 5.
3. Weigh the crucible and lid empty – write this value down.
 - a. Add 1~1.5g of magnesium to the crucible and record the new mass including the lid. You
4. Put the lid on crucible off-centre to allow for air to enter without allowing the magnesium oxide to escape.
5. Use the tongs to place the crucible on the clay triangle.
6. Light the Bunsen burner and then open the air collar on the chimney to increase available oxygen for combustion and achieve a hotter blue flame.
7. Heat the crucible for 10 minute to allow sufficient time for all of the magnesium to oxidize.
 - a. Turn off the Bunsen burner and allow time for the crucible to cool down.
8. When cooled, weigh the crucible and lid containing the magnesium oxide – write this value down.

Note: for this experiment the crucible was heated in a furnace at 600°C for 30 minutes. The auto-ignition temperature of magnesium is 473°C [10].

RESULTS

Mass of crucible and lid [g]	72.4
Mass of crucible, lid and magnesium [g]	73.4
Mass of magnesium before heating [g]	$73.4 - 72.4 = 1.0$
Mass of crucible, lid and product after heating [g]	73.9
Mass of product after heating [g]	$73.9 - 72.4 = 1.5$
Mass of oxygen reacted with magnesium [g]	$1.5 - 1.0 = 0.5$
Moles of oxygen [mol]	$\frac{0.5[g]}{16[\frac{g}{mol}]} = 0.03125$
Moles of magnesium [mol]	$\frac{1.0}{24} = 0.041\bar{6}$
Mole ratio of Mg to O	$\frac{0.041\bar{6}}{0.03125} = 1.3 \therefore \text{mol ratio is } \approx 1:1$
Determined formula of the oxide	$Mg^{2+}O^{2-}$
Percent by mass of Mg in the oxide	$\frac{24}{24 + 16} \times 100 = 60\%$
Percent by mass of O in the oxide	$\frac{16}{24 + 16} \times 100 = 40\%$
Balanced chemical equation	$2Mg + O_2 \rightarrow 2MgO$

TABLE 1 - EXPERIMENT DATA AND RESULTS FROM APPENDIX A – EXPERIMENT OBSERVATIONS



FIGURE 6 - MgO REACTION PRODUCT

DISCUSSION

In Table 1, the Mole ratio of Mg to O is 1.3:1. As not all of the magnesium's surface area is exposed to oxygen it may not have fully oxidized, leading to this slight surplus in moles of Mg:O. This surplus may be reduced by a higher heating temperature as the time of heating (30 minutes) should be sufficient.

In Figure 6 a greenish yellow by-product can be observed, this is magnesium nitride Mg_3N_2 [11] and contributes to some of the error in the oxidation of magnesium. 78% of earth's atmosphere is comprised of Nitrogen (N_2) [12]. As nitrogen atoms require 3 electrons to complete their valence shell, triple covalent bonds are formed between nitrogen atoms. These triple bonds are much stronger than O_2 's double bonds, with an enthalpy of formation of 945.33kJ/mol compared to 498.34kJ/mol [13]. Consequently, despite the abundance of nitrogen in the atmosphere, far less of it reacts with the magnesium to form Mg_3N_2 .

$$\text{moles Mg} - \text{moles O} \times \text{molar mass of } Mg_3N_2 = \frac{1.0}{24} - \frac{0.5}{16} \times ((24 \times 3) + (14 \times 2)) = 1.0416g$$

EQUATION 3 - THEORETICAL CALCULATION OF MASS OF Mg_3N_2 PRODUCED FROM UNREACTED Mg

The theoretical mass of Mg_3N_2 as a by-product from Equation 3 does not align with the total mass of products as recorded in Table 1, as such the main source of error was unreacted Mg.

CONCLUSION

This report has discussed an experiment in which magnesium was heated in a crucible to form magnesium oxide. The theory behind the bonding of the component elements and products has been discussed as well as the use of moles in a macroscopic view of the quantities of a substance.

The theoretical mole ratio of Mg to O is 1:1, whereas, the experiment resulted in a surplus of Mg to O in a ratio of 1.3:1. This surplus was the result of not all the magnesium atoms being subjected to heating and oxygen due to the surface area of the magnesium pellets exposed. A secondary error of the production of magnesium nitride was discussed, but ultimately did not contribute significantly to this error.

This experiment may produce results closer to the theoretical equation with higher heating temperature. Although, more magnesium nitride may be formed due to the increase in energy available for breaking nitrogen triple bonds.

BIBLIOGRAPHY

- [1] J. Clark, "Metallic bonding," ChemGuide, 2019. [Online]. Available: <https://www.chemguide.co.uk/atoms/bonding/metallic.html#:~:text=Metallic%20bonding%20in%20magnesium&text=Magnesium%20has%20the%20outer%20electronic,ions%22%20and%20%22sea%22..> [Accessed 24 07 2023].
- [2] J. Clark, "Reactions of Group 2 Elements with Water," LibreTexts Chemistry, [Online]. Available: [https://chem.libretexts.org/Bookshelves/Inorganic_Chemistry/Supplemental_Modules_and_Websites_\(Inorganic_Chemistry\)/Descriptive_Chemistry/Elements_Organized_by_Block/1_s-Block_Elements/Group__2_Elements%3A_The_Alkaline_Earth_Metals/1Group_2%3A_Chemical_Re.](https://chem.libretexts.org/Bookshelves/Inorganic_Chemistry/Supplemental_Modules_and_Websites_(Inorganic_Chemistry)/Descriptive_Chemistry/Elements_Organized_by_Block/1_s-Block_Elements/Group__2_Elements%3A_The_Alkaline_Earth_Metals/1Group_2%3A_Chemical_Re.) [Accessed 23 07 2023].
- [3] I. Guch, 24 Lessons that rocked the world, Alexandria, VA: Cavalcade Publishing, 2004.
- [4] Encyclopaedia Britannica, "Stoichiometry," 2023.
- [5] P. Becker, "Determination of the Avogadro constant - A contribution to the new definition of the mass unit kilogram," *The European Physics Journal*, 2009.
- [6] P. J. B. A. P. O. R. B. G. Richard J. C. Brown, "Redefinition of the Mole in the Revised International System of Units and the Ongoing Importance of Metrology for Accurate Chemical Measurements," *Analytical Chemistry*, vol. 93, no. 36, pp. 12147-12494, 2021.
- [7] J. L. Maienschein, "Research Topics in Explosives - A look at Explosives Behaviours," Lawrence Livermore National Laboratory, Seattle, 2013.
- [8] . J. M. S. Dr. Chunlin He, "Potassium 4,5-Bis(dinitromethyl)furoxanate: A Green Primary Explosive with a Positive Oxygen Balance," *Journal of the German Chemical Society*, 2016.
- [9] L. d. B. e. al, "Qualification of Magnesium/Teflon/Viton pyrotechnic composition used in rocket motors ignition system," Insitute of Aeronautics and Space, Sao Jose, Brazil, 2016.
- [10] Inchem, "Magnesium Powder".
- [11] ChemEurope, "Encyclopedia - Magnesium Nitride," [Online]. Available: https://www.chemeuropa.com/en/encyclopedia/Magnesium_nitride.html. [Accessed 26 07 2023].
- [12] NASA, "The Composition of Planetary Atmospheres," [Online]. Available: https://www.nasa.gov/sites/default/files/files/YOSS_Act_4.pdf. [Accessed 26 07 2023].
- [13] A. Z. -. UCSB, "Bond Dissociation Energies," [Online]. Available: <https://labs.chem.ucsb.edu/zakarian/armen/11---bonddissociationenergy.pdf>. [Accessed 26 07 2023].
- [14] Nagwa, "Lesson Explainer: Metallic Bonding," [Online]. Available: <https://www.nagwa.com/en/explainers/843104808380/>. [Accessed 24 07 2023].

APPENDICES

APPENDIX A – EXPERIMENT OBSERVATIONS

DATA SHEET to RECORD OBSERVATIONS, CALCULATIONS and RESULTS

date 07/07/23

STUDENT'S NAME: THOMAS VOLPATO

PARTNER'S NAME: MARCUS BAKER - WELLS

Mass of crucible and cover	72.4 g
Mass of crucible, cover and magnesium	73.4 g
Mass of magnesium before heating	$73.4 - 72.4$ $= 1.0 \text{ g}$
Mass of crucible, cover and product after heating	~~73.4~~ 73.9 g ~~70.0~~
Mass of product after heating	$73.9 - 72.4$ ~~$73.4 - 72.4$~~ $= 1.5 \text{ g}$
Mass of oxygen which reacted with magnesium	$1.5 \text{ g} - 1.0$ $= 0.5 \text{ g}$
Moles of oxygen	$= \frac{0.5 \text{ g}}{16 \text{ g/mol}} = 0.03125 \text{ mol}$
Moles of magnesium	$= \frac{1.0}{24} = 0.0416 \text{ mol}$

30 min @ 600°C

$2\text{Mg} + \text{O}_2 \rightarrow \text{Mg}^{2+}\text{O}^{2-}$

5

Mole ratio of Mg to O	$\frac{0.0416}{0.03125} = 1.3 \approx 1:1^*$
Determined formula of the oxide	$Mg^{2+} O^{2-}$
Percent by mass of Mg in the oxide	$\frac{24}{24+16} \times 100 = 60\%$
Percent by mass of O in the oxide	$\frac{16}{40} \times 100 = 40\%$
Balanced chemical equation	$2Mg + O_2 \rightarrow 2MgO$

To calculate moles of magnesium and oxygen, use the correct molar masses as a conversion factor . Refer to lecture notes for the details

SHOW ALL YOUR CALCULATIONS and REMEMBER THE UNITS !!!

* 1.3:1 Mg to O ratio due to incomplete oxidation of the pellets.
The inner core was not exposed to enough heat and/or O_2 to heat and form MgO .

Recommended sources:

[Empirical Formula of Magnesium Oxide by Experiment Chemistry Tutorial](#)

[Empirical Formula of Magnesium Oxide](#) and videos within.

MINI REPORT – EVALUATION OF SPECTRAL ANALYSIS METHODS: FT-IR AND RAMAN

INTRODUCTION

Fourier Transform Infrared (FT-IR) and RAMAN Spectroscopy are both confirmatory test methods used in analytical chemistry. They are quantitative and will identify amounts of substances as opposed to presumptive tests that will only indicate the presence of a substance such as flame tests [1]. The disadvantage of this spectroscopy equipment is that both are expensive to purchase and operate, some devices require complex sample preparation and additionally they require standardized laboratory conditions and calibration.

FT-IR

FT-IR operates by exposing a sample to Infrared radiation from a laser. The bonds in this sample will absorb some of this energy corresponding to the size and vibration of the bonds between the atoms. This discrepancy is logged by a detector. As no two compounds will have the same combination of atoms or more importantly, inter-atomic bond sizes; the component bonds of the sample can be characterized and identified by this absorption spectra. Additionally the size of absorption spectra peaks can determine the quantity of a substance [2].

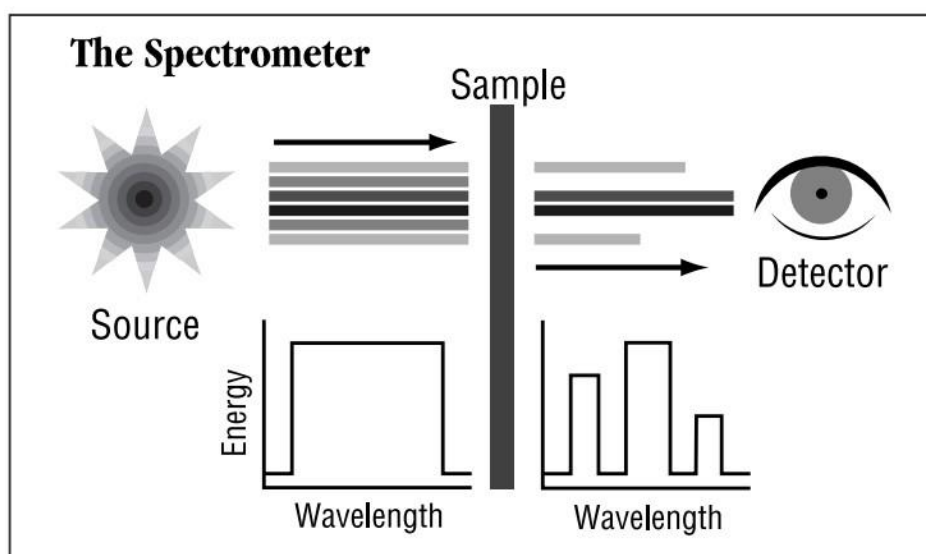


Table 1 - Simple graphic depicting a spectrometer [2]

RAMAN

This method of spectroscopy uses a single wavelength laser. When these photons transfer their energy to a molecules bond and excites it to a higher energy level, most of this energy will be emitted as a photon (scattered) elastically when it returns to its original ground state. $0.1 \times 10^{-6}\%$ of these interactions result in the bond returning to a different energy level and thus scattering the emitted photon to a different frequency [3]. This discrepancy is similar for different atom to atom bonds but unique to individual molecules. The main advantage of RAMAN is that little sample preparation is required and it can be more portable, although it is more expensive.

MOLECULE IDENTIFICATION

Between the 4000-1500 cm^{-1} wavelengths the functional groups such as hydroxide (OH) within the molecule can be identified. This region is useful for identifying what the molecule is comprised of but cannot specifically identify compounds. Less than 1500 cm^{-1} wavelengths are known as the 'fingerprint region'. These are complex to analyze but can specifically identify compounds under analysis due to their unique values [4]. Reference material for bond wavelengths and fingerprint regions are widely available and many spectrum analysis software integrate libraries of this data for quick analysis of samples.

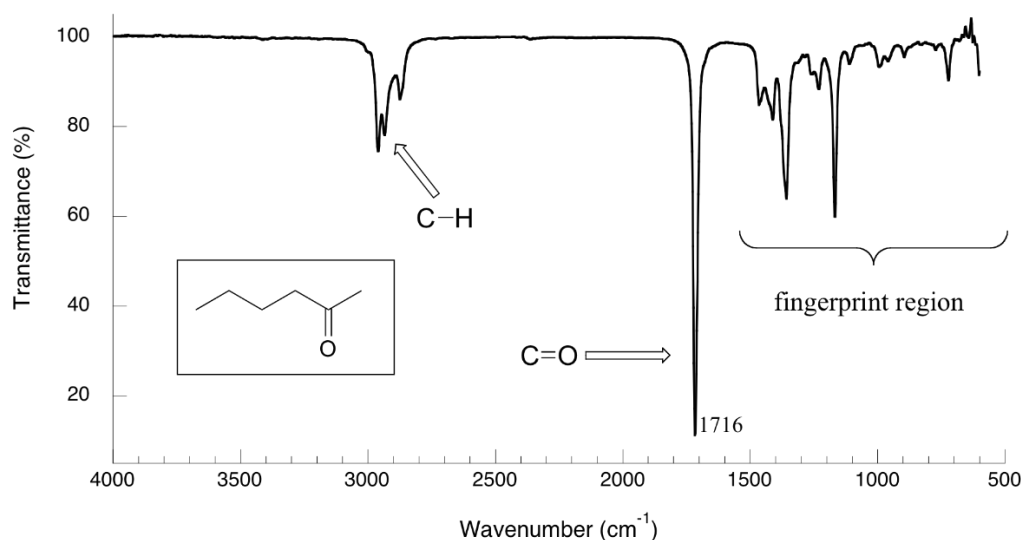


Figure 1 - IR spectrum of Hexanal [4]

REFERENCES

- [1] S. R. Alyson Grine, "Presumptive and Confirmatory Forensic Tests," [Online]. Available: https://www.sog.unc.edu/sites/default/files/course_materials/Presumptive%20and%20Confirmatory%20Forensic%20Tests.pdf. [Accessed 25 07 2023].
- [2] Thermo Nicolet Corporation, "Introduction to Fourier Transform Infrared Spectrometry," ThermoNicolet, Madison WI, 2002.
- [3] Traces Centre, "Understanding Raman Spectroscopy," 2021.
- [4] University of Texas, "Infrared Spectroscopy (IR)," [Online]. Available: https://www.utdallas.edu/~scortes/ochem/OChem_Lab1/recit_notes/ir_presentation.pdf. [Accessed 26 07 2023].
- [5] LibreTexts Chemistry, "Interpreting Infrared Spectra," [Online]. Available: https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Organic_Chemistry_%28Morsch_et_al.%29/12%3A_Structure_Determination_-_Mass_Spectrometry_and_Infrared_Spectroscopy/12.07%3A_Interpreting_Infrared_Spectra. [Accessed 26 07 2023].