Main text:
Mechanochemical reactions[1] have developed from a laboratory curiosity to a viable alternative to conventional chemistry in bulk solvents. Mechanochemistry enables room temperature reactions without bulk solvents,[2] access to modified or previously unknown reactivity,[3] and routes to molecules and materials that are otherwise difficult or even impossible to access.[4] Ball milling, twin screw extrusion,[5] and accelerated aging[6] have been applied successfully for the synthesis of a number of popular Metal Organic Frameworks (MOFs),[7] including carboxylate-based MOF-74[8] and IRMOF materials,[9] zeolitic imidazolate frameworks (ZIFs),[10] and zirconium-based UiO- and NU-systems.[11] Importantly, mechanochemistry permits simple, room-temperature and solvent-free assembly of MOFs from reagents that, although preferred for industrial synthesis, are not usable by conventional solution chemistry due to poor solubility: metal oxides, carbonates or other basic salts.[12-14]

In spite of the rapid growth in the application of mechanochemistry in chemical and materials synthesis, the underlying reaction mechanisms and kinetics remain poorly understood, and it was not until 2013 that in situ techniques for monitoring structural and chemical transformations in a ball mill were reported, using powder X-ray diffraction (PXRD) and/or Raman spectroscopy.[15] However, other simpler methods exist for monitoring mechanochemical reactions, provided direct acquisition of structural data is not required. In the context of inorganic solids, it was shown that kinetics of mechanochemical reactions involving a gaseous reactant or product may be followed by monitoring the pressure and temperature of the gas in a reaction vessel of constant volume. For example, Avvakumov was reported to have followed the reaction of BaCO$_3$ and WO$_3$ to form BaWO$_4$ and CO$_2$ gas by monitoring the change in pressure of evolved CO$_2$.[16] Despite these early kinetic experiments and the ever increasing body of work on mechanochemistry of gaseous reactants and products,[17] as well as the interest that mechanochemistry of MOFs has garnered,[18] the kinetics of MOF-forming mechanochemical reactions have not yet been studied using gaseous reactants or products.

Here we demonstrate how the use of a metal carbonate[19] precursor can enable monitoring of the clean, effective synthesis of the ZIF topology zinc imidazolate,[20] as well as the popular sodalite (SOD) topology of zinc 2-methylimidazolate[21] (ZIF-8/MAF-4, sold as Basolite Z1200 or Porolite Z8, Figures 1a-c). Monitoring of reaction progress through changes of pressure inside a reaction vessel due to release of CO$_2$ allowed the discovery and prevention of a feedback mechanism that creates a complex metal carbonate if excess imidazole is not used.
Reactions were conducted in a Retsch PM400 planetary mill, using PM GrindControl™ vessels equipped for real-time measurement and recording of pressure and temperature inside the vessel. To establish the chemical significance of pressure measurements, we first investigated a simple model reaction of solid CaCO$_3$ with MoO$_3$ which, upon milling in equimolar amounts, is expected to yield CaMoO$_4$ and one equivalent of CO$_2$ gas (Figure 1d-e). In a typical experiment, 5.9 g of MoO$_3$ (0.04 mol) and 4.1 g of CaCO$_3$ (0.04 mol) were milled at a frequency of 350 rpm. Reaction conditions (milling time and media) were varied to achieve a broad range of yields (SI 2.2). During milling, pressure in the reaction vessel increased in a monotonic fashion, aside from a small pressure drop at the end of the milling time associated with a rapid temperature drop. Reaction conversions were calculated both from the final pressure in the reaction vessel and from subsequent thermogravimetric analysis (TGA) of the milled samples, giving values in excellent agreement (Figure 1e).

Having established that monitoring the increase of gas pressure during milling can be used to quantify the extent of reaction, we explored the neat grinding (NG) mechanochemical reaction of imidazole (HIm) with basic zinc carbonate, [ZnCO$_3$][Zn(OH)$_2$] (ZnCarb) in a Zn:Im ratio of 1:2. Pressure inside the vessel rose slowly during the first 5 min of milling, after which it rapidly increased, reaching a maximum within ca. 12 min (Figure 2a). A significantly faster reaction was observed if the
reaction was done by liquid-assisted grinding (LAG,\textsuperscript{[22]} milling in the presence of a liquid additive) or by ion- and liquid-assisted grinding (ILAG, milling in the presence of a liquid additive as well as a catalytic amount of a salt\textsuperscript{[23]}). Specifically, the addition of 3 mL of ethanol (EtOH) to 13.5 g of the reaction mixture led to a rapid increase of pressure immediately upon the onset of milling, reaching a maximum in ca. 10 min (Figure 2a). Similar observations were made for ILAG, conducted with addition of 3 mL of EtOH and 400 mg (10 mol% with respect to zinc) of NH\textsubscript{4}NO\textsubscript{3} as the salt additive (Figure 2a).

![Figure 2. a) Time-dependent reaction vessel pressure profiles of the mechanochemical reactions of basic zinc carbonate, [ZnCO\textsubscript{3}][Zn(OH)\textsubscript{2}] with imidazole under NG (solid), LAG (ethanol, dashed), and ILAG (ethanol, NH\textsubscript{4}NO\textsubscript{3}, dotted) milling, and b) experimental PXRD patterns of the reagents and products of milling syntheses of zni-Zn(Im\textsubscript{2}) from basic zinc carbonate and imidazole, and the calculated PXRD pattern of zni-Zn(Im\textsubscript{2}) (CSD code IMIDZB03).]

The LAG and ILAG reactions led to significantly higher final reaction pressures compared to neat milling, indicating conversions of 87\% for LAG and ILAG and 64\% for neat milling, after accounting for the vapour pressure of EtOH and solubility of CO\textsubscript{2} in EtOH (SI-2.1). However, analysis of the reaction products by powder X-ray diffraction (PXRD) immediately after milling revealed that the zni-topology framework zni-Zn(Im\textsubscript{2}) was the only crystalline component (Figure 1b), indicating full conversion. Moreover, thermogravimetric analysis (TGA) of the milling products, conducted after washing with ethanol in order to remove any unreacted HIm, provided an excellent match for pure Zn(Im\textsubscript{2}), without any residual zinc carbonate reactant. The TGA and PXRD results therefore strongly indicate that all three approaches result in quantitative conversion of the reactants to zni-Zn(Im\textsubscript{2}), in contrast to reaction vessel pressure measurements. As a potential explanation for the discrepancy between pressures measured for neat milling and for reactions in the presence of a liquid phase, we consider that some of the CO\textsubscript{2} gas might be retained in the pores\textsuperscript{[24]} of zni-Zn(Im\textsubscript{2}) formed by neat grinding. In the case of LAG or ILAG reactions, the pores of zni-Zn(Im\textsubscript{2}) might be occupied by the liquid additive, reducing such entrapment of CO\textsubscript{2}.

Next, we investigated reaction vessel pressure changes for the reaction of basic zinc carbonate and 2-methylimidazole (HMeIm\textsubscript{2}), expected to yield the popular sodalite (SOD) topological form of Zn(MeIm\textsubscript{2}) (ZIF-8). The reaction vessel pressure profile was very different from that observed with
HIm. Milling the two reactants in a 1:2 stoichiometric ratio of \( \text{ZnCarb} \) and \( \text{HMeIm} \) (15 g scale), led to a monotonic increase in vessel pressure over the first 10 min milling, followed by a slow decrease (Figure 3a). The appearance of such a temporary peak in reaction vessel pressure was observed in all repeated experiments. PXRD analysis of the reaction products after 15 min milling revealed only Bragg reflections of ZIF-8. However, after 1 hr milling, PXRD analysis of the products revealed additional peaks, consistent with a complex metal carbonate (1) of unknown structure, previously reported\(^{25}\) to form upon exposure of ZIF-8 to liquid water or moist \( \text{CO}_2 \). Close examination of the \(^1\text{H}\rightarrow^{13}\text{C}\) CP-MAS SSNMR spectrum of the product after 15 min milling shows signals of 1, indicating that it is present in the mixture, though undetected by PXRD (Figure S20). Similar behavior was observed with LAG, conducted by adding 3 mL of EtOH to 15 g of reaction mixture. In this case, the reaction vessel pressure also exhibited an early maximum, and slowly diminished over time. Analysis by PXRD again revealed the formation of ZIF-8, along with low-intensity reflections consistent with 1.

These observations suggest that the mechanochemical reaction of basic zinc carbonate and \( \text{HMeIm} \) proceeds in two stages, first yielding ZIF-8 and producing an increase in the reaction vessel pressure due to \( \text{CO}_2 \) evolution. Further milling, however, leads to absorption of the nascent \( \text{CO}_2 \) gas by ZIF-8 and further reaction between ZIF-8 and the \( \text{H}_2\text{O} \) and \( \text{CO}_2 \) byproducts, which yields the carbonate phase 1, accompanied by a decrease in the reaction vessel pressure. This proposed reaction pathway was also verified by analogous experiments in a shaker mill, the smaller scale of which enabled more thorough sampling of the reaction mixture. Analysis of PXRD patterns of the LAG reaction mixture after 5 min and 10 min milling revealed that ZIF-8 was the only reaction product, while Bragg reflections of 1 were clearly visible after 30 min (Figure S21). Such reaction behavior was not greatly affected by the choice of milling liquid (water, methanol or isopropanol), although it seems that the use of bulkier milling liquids leads to less carbonate byproduct (Figure S22).

In order to substantiate that a carbonate phase is indeed formed on reaction of ZIF-8 and moist \( \text{CO}_2 \), crystal structure determination of 1 was carried out directly from PXRD data\(^{26}\) recorded on a sample prepared (independently) by exposing a suspension of ZIF-8 in water to a stream of \( \text{CO}_2 \) gas. The PXRD data for 1 were indexed using the ITO code\(^{27}\) in the program CRYSFIRE,\(^{28}\) giving a unit cell with orthorhombic metric symmetry \((a = 10.54 \text{ Å}, b = 12.06 \text{ Å}, c = 4.70 \text{ Å}; V = 596.5 \text{ Å}^3)\). The space group was assigned as \( \text{Pbc}_{2}\)\(_1\), with two formula units \( \text{Zn}(\text{MeIm})_2\text{CO}_3 \) in the unit cell \((Z = 2)\). Profile fitting using the Le Bail method in the GSAS program\(^{29}\) gave a good quality of fit \((R_{wp} = 3.99\%, R_p = 2.81\%; \text{ Figure S25})\), and the refined unit cell parameters were used in the subsequent structure solution calculation, which was carried out using the direct-space genetic algorithm technique in the program EAGER.\(^{30}\) The best structure solution was then used as the starting model for Rietveld refinement, carried out using the GSAS program,\(^{29}\) giving a good final Rietveld fit \((R_{wp} = 4.97\%, R_p = 3.37\%; \text{ Figure 3c})\) to the PXRD data, very close to the quality of the Le Bail fit, with the following refined parameters: \(a = 10.5295(3) \text{ Å}, b = 12.0539(4) \text{ Å}, c = 4.6928(1) \text{ Å}; V = 595.62(4) \text{ Å}^3\).

In the structure of 1, each zinc cation is bonded tetrahedrally to two 2-methylimidazolate and two \( \text{CO}_2 \)-anions. The 2-methylimidazolate anions bridge the zinc cations into one-dimensional ribbons, which are then connected into sheets by carbonate anions (Figure 3d). The carbonate anions display two modes of coordination: one of the oxygen atoms is dicoordinated and connects zinc atoms within one 2D sheet, while the other two oxygen atoms are monocoordinated to two neighbouring zinc atoms from a second 2D sheet (Figure 3e). In this way, the carbonate serves both as a building block for the 2D sheets and as the perpendicular strut stacking the sheets together into a 3D framework.

The manometric observation of the reaction between basic zinc carbonate and \( \text{HMeIm} \) thus revealed a parasitic process that converts the target ZIF-8 into a complex carbonate 1. The composition of 1 suggests that the process could involve a reaction in which 2-methylimidazolate anions are replaced by carbonates:

\[
2 \text{Zn(MeIm)}_2 + \text{H}_2\text{O} + \text{CO}_2 \rightarrow \text{Zn(MeIm)}_2::\text{ZnCO}_3 + 2 \text{HMeIm} \quad \text{(Eq 1)}
\]
Figure 3. a) Time-dependent reaction vessel pressure profiles and b) PXRD patterns for the different syntheses of SOD-Zn(HMelm): from basic zinc carbonate and HMelm. Top to bottom: NG reactions; Zn:HMelm = 1:2 (15 min and 1 hr), Zn:HMelm = 1:3 (1 hr), ethanol LAG reactions; Zn:HMelm = 1:2 (15 min and 1 hr). Stars indicate peaks of 1 in the reaction mixtures. Structure determination of complex carbonate 1 from PXRD data: c) the final fit obtained in the Rietveld refinement, and views of the structure of 1 along d) the c-axis and e) the a-axis.
The proposed equation suggests that formation of 1 may be avoided by using excess HMeIm in the reaction mixture, both as a pore-filling agent to minimize access of carbonate anions to zinc centers, and taking advantage of Le Chatelier’s principle to push the equilibrium towards ZIF-8. Indeed, manometric monitoring of the neat milling reaction of basic zinc carbonate with three equivalents of HMeIm per zinc revealed a continuous increase in reaction vessel pressure, which plateaued after ca. 50 min milling (Figures 3a, S13). PXRD analysis of the sample after 1 hr milling revealed the presence of ZIF-8 as the only reaction product, with no observable Bragg reflections due to 1 (Figure S12). After washing with EtOH, thermogravimetric analysis of the ZIF-8 product gave a residue of 36% (Figure S18), which is consistent with the ZnO residue expected for pure ZIF-8 (36.4%). Furthermore, unlike the Zn:HMeIm = 1:2 reaction product, the $^1$H→$^{13}$C CP-MAS SSNMR spectrum of the Zn:HMeIm = 1:3 reaction product after 15 min milling showed only ZIF-8 peaks, and no signals due to 1 (Figure S20). Similarly, PXRD analysis of analogous LAG reactions with excess HMeIm in a shaker mill showed no evidence for the presence of 1 even after 30 min milling with methanol, ethanol or isopropanol as the liquid additive (Figure S23). Interestingly, none of the milling products from the Zn:HMeIm = 1:3 preparations showed evidence of HMeIm in the PXRD pattern, in line with the hypothesis that extra ligand is encapsulated in the pores of ZIF-8.

We have therefore shown that using excess HMeIm in the mechanochemical reaction of basic zinc carbonate and HMeIm facilitates the formation of large quantities of pure ZIF-8 at room temperature under mild conditions. However, the fact that washing with solvent is still required as part of the purification process detracts somewhat from the environmental benefits of using mechanochemistry, so a fully solvent-free method for synthesizing and purifying MOFs would be desirable. To tackle this problem, we prepared ZIF-8 mechanochemically from zinc carbonate at a 90 g scale and purified it without using solvents at any stage for the first time. Specifically, the product resulting from an NG reaction between basic zinc carbonate and HMeIm (Zn:HMeIm = 1:3) carried out in a planetary mill was purified by sublimation (200 °C under vacuum for 5 hr) of the included HMeIm without any detrimental effect on the ZIF-8, as corroborated by PXRD, TGA and N$_2$ sorption isotherms (Figures S25-S27). In addition, this route yielded a MOF with 1785 m$^2$/g BET surface area, comparable to the commercial analogue (1758 m$^2$/g). The sublimed HMeIm was collected as colourless crystals and was found to be pure by solution-state $^1$H NMR spectroscopy, allowing the possibility to reuse this material as the ligand in further synthesis.

In summary, we have shown for the first time that ZIFs (namely zni-ZnIm$_2$ and SOD-Zn(MeIm)$_2$) can be made mechanochemically from basic zinc carbonate and simple imidazoles and that the reaction progress can be monitored in situ by measuring the change of pressure inside the milling vessel due to release of CO$_2$ gas during the reaction. In situ monitoring of the synthesis of the commercially viable ZIF-8 revealed an unprecedented feedback loop in which the newly produced ZIF-8 reacts with released CO$_2$ gas and water to form a complex zinc carbonate methylimidazolate, the structure of which has been elucidated here for the first time and provides a new addition to the already rich landscape of zinc 2-methylimidazolate phases.$^{[31]}$ Formation of the byproduct is then prevented by addition of excess ligand, enabling large scale quantitative synthesis of ZIF-8 from cheap and available starting materials, and the possibility of a totally solvent-free route for its manufacture.

**Experimental Section**
Details of experimental procedures are given in Supporting Information (SI). Large scale (ca. 15 g) mechanochemical syntheses were carried out in a Retsch PM400 mill operating at 300 rpm. Reaction mixtures were milled in a 250 mL PM GrindControl™ reaction vessel with seven stainless steel balls ($m = 44$ g, $d = 20$ mm, $V = 6$ mL). Small scale (200 mg) reactions were done in a 10 mL stainless steel milling jar with two stainless steel balls ($m = 1.3$ g, $d = 7$ mm) at 30 Hz. All samples were washed extensively with ethanol to remove potential unreacted imidazole starting material before further analysis. The vessel pressure curves, TGA, PXRD, solid-state NMR and FTIR-ATR data are given in the SI, along with details of PXRD structure determination of 1. Crystallographic data for 1 have been deposited with the Cambridge Structural Database (CCDC deposition code 1942361).

**Acknowledgements**
This work was funded by NSERC DG, CREATE and I2I grants. KTH and IB acknowledge support by the NSF (DMR-1610882). IB thanks Georgetown University (Kunin Fellowship, GSAS Dissertation Research Grant), and the ICDD (Ludo Frevel Scholarship). FRQNT Center in Green Chemistry & Catalysis is acknowledged for support. The École Nationale Supérieure de Chimie de Clermont-Ferrand is thanked for providing a summer placement at Cardiff University to F.D. The authors thank MOF Technologies Ltd. for providing a reference sample of Porolite Z8. W. Y. thanks the National Natural Science Foundation of China (21561009) for financial support.

**Keywords:** Mechanosynthesis • Ball milling • Metal-organic frameworks • Pressure • Mechanism

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