Electronic Supplementary Material (ESI) for Dalton Transactions

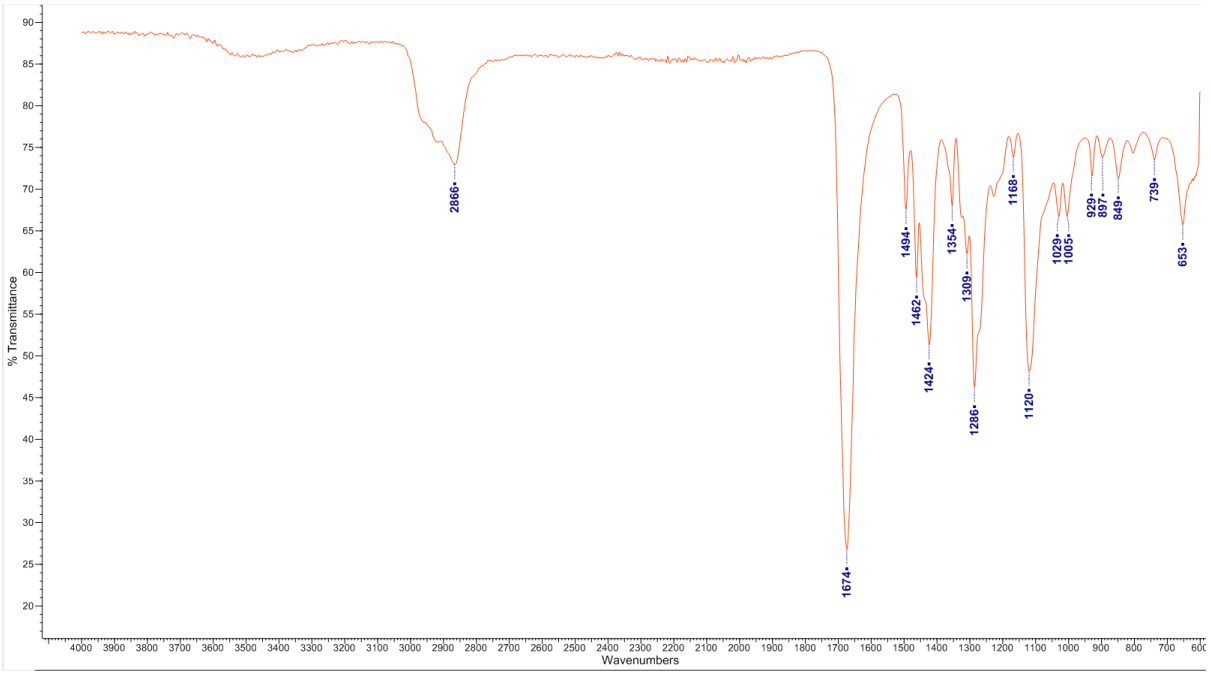
N-Alkyl Lactam Ether Podands as Versatile Alkali Metal Ion Chelants

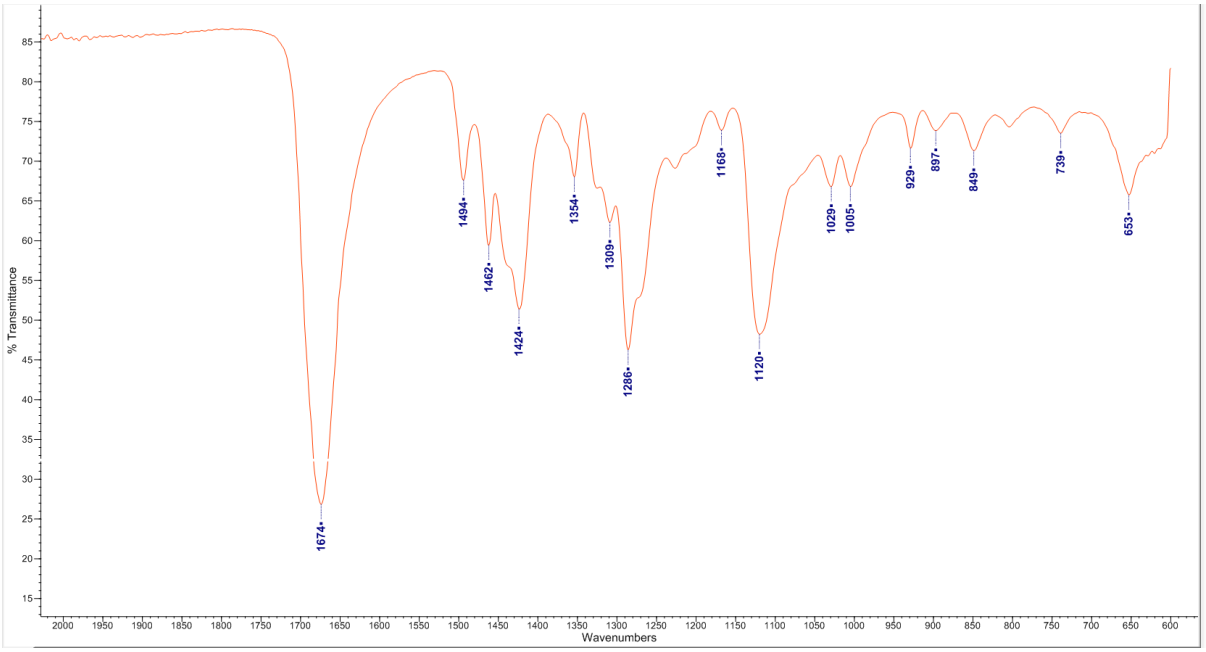
Andrea Perrin,[a] Dominic Myers,[a] Katharina Fucke,[a] Osama M. Musa[b] and Jonathan W. Steed*[a]

Electronic Supplementary Information

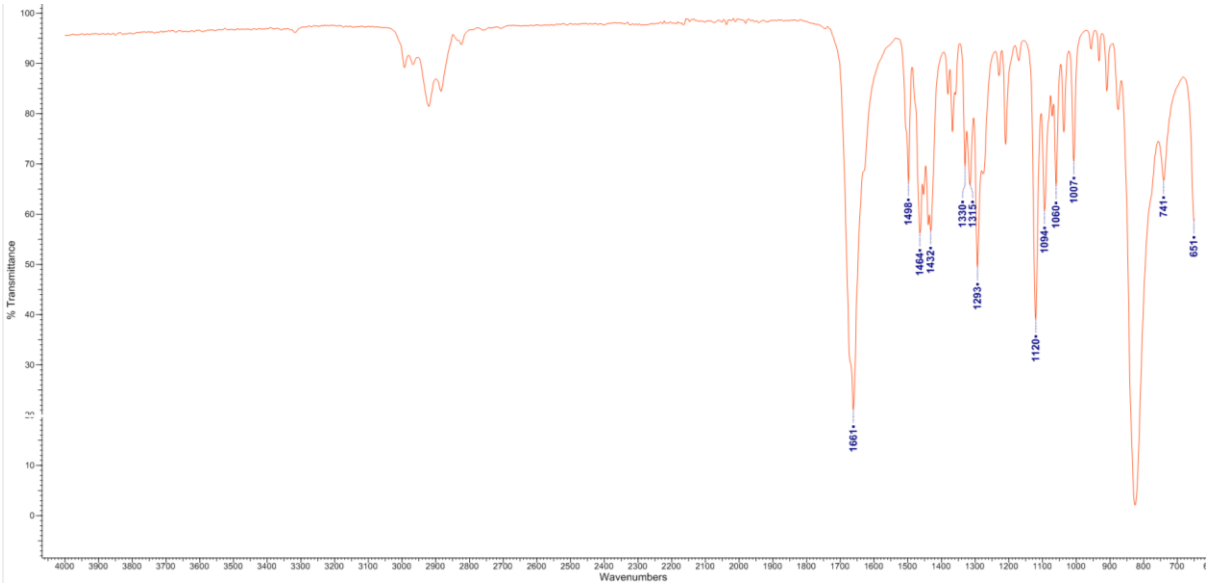
IR Spectra

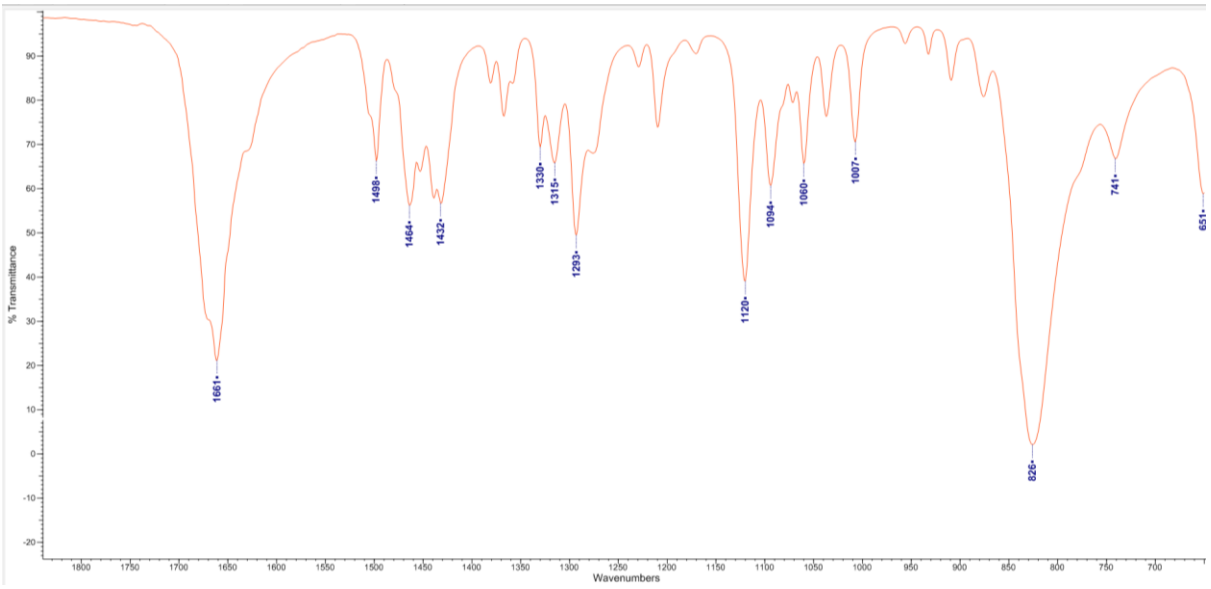
1-{2-[2-(2-oxo-pyrrolid-1-yl)-ethoxy]-ethyl}-pyrrolid-2-one (neat oil as supplied) (**1**)



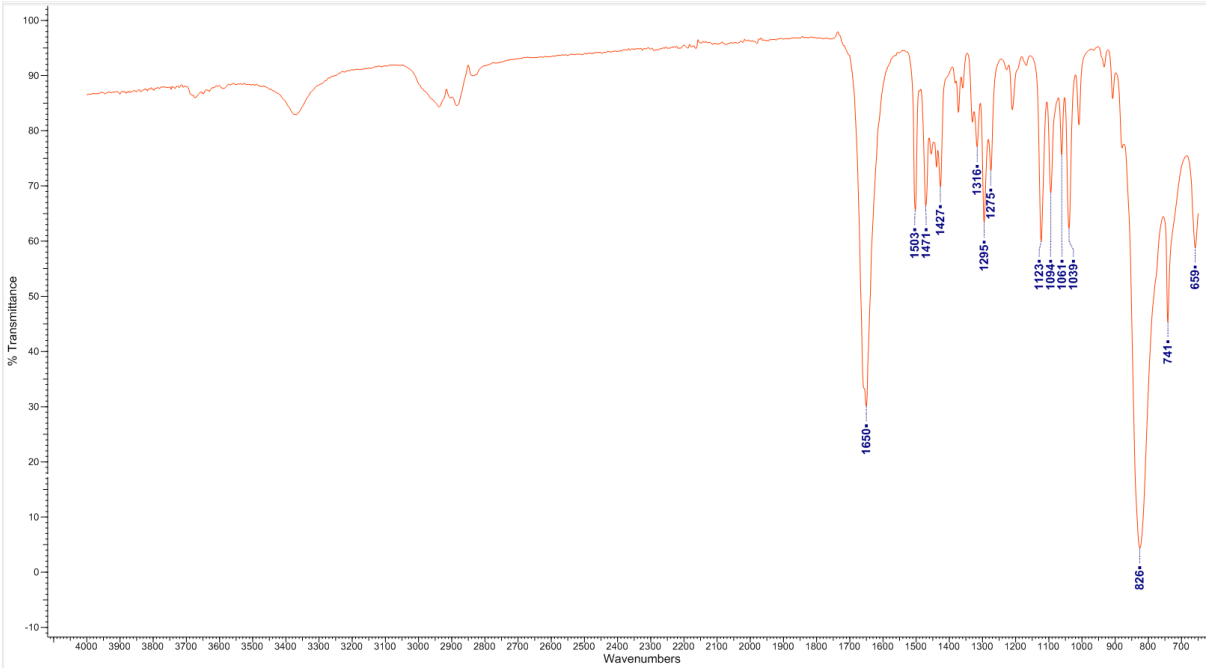


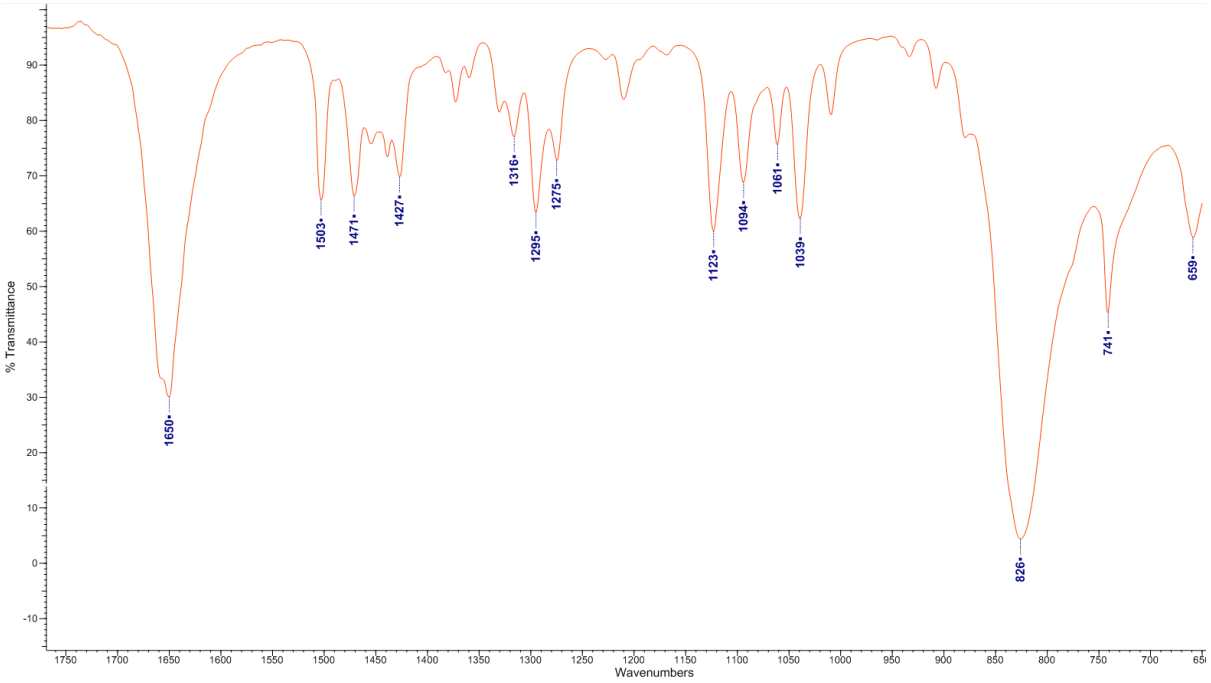
[Na(**1**)$_2$]PF$_6$ (**3**)



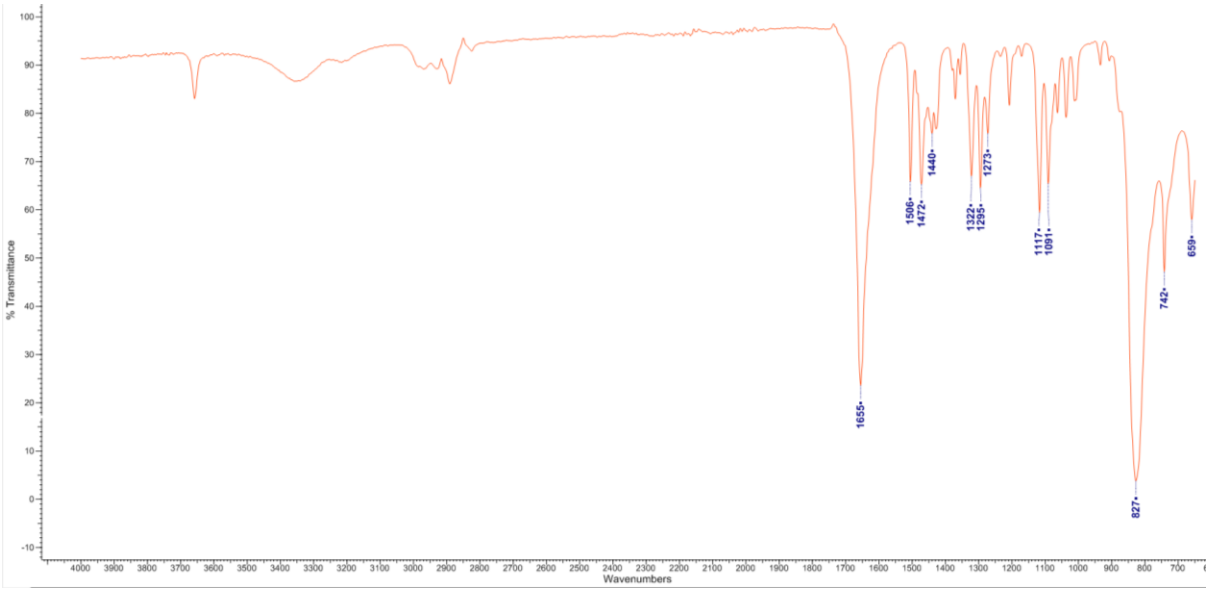


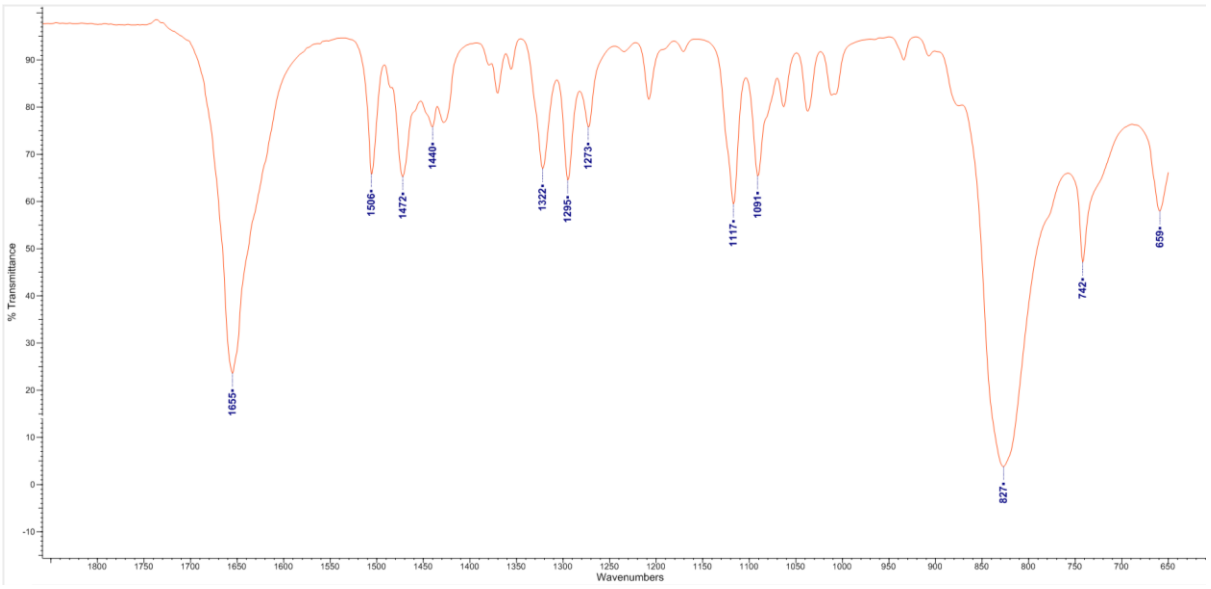
$[Na_3(\mu\text{-}\mathbf{1})_2(H_2O)_2](PF_6)_3$ (**4**)



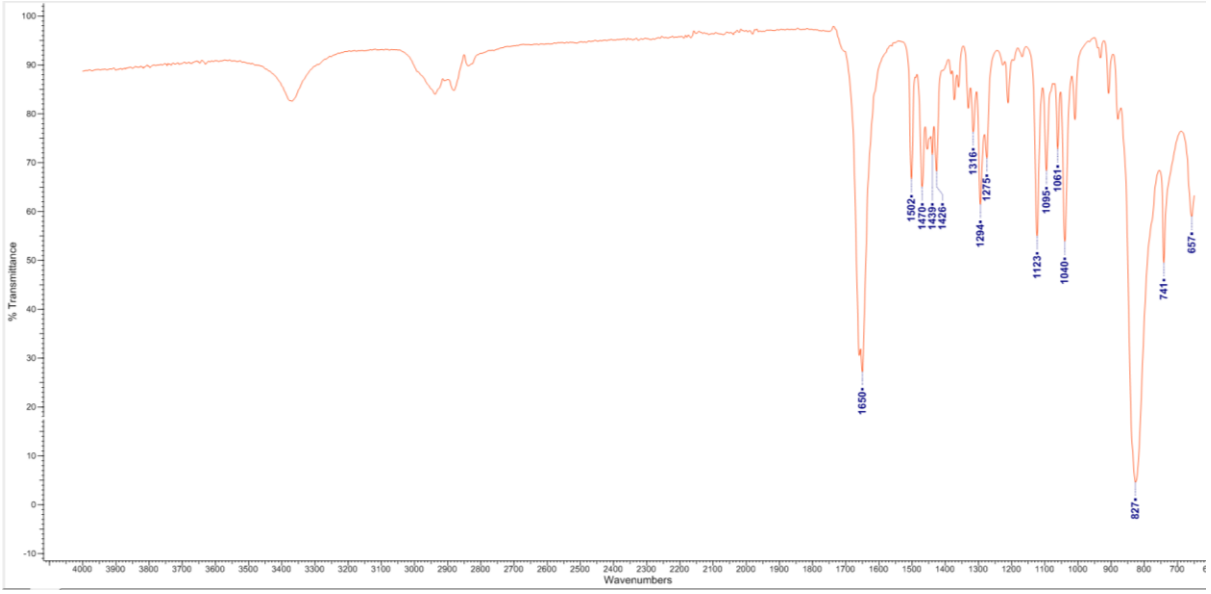


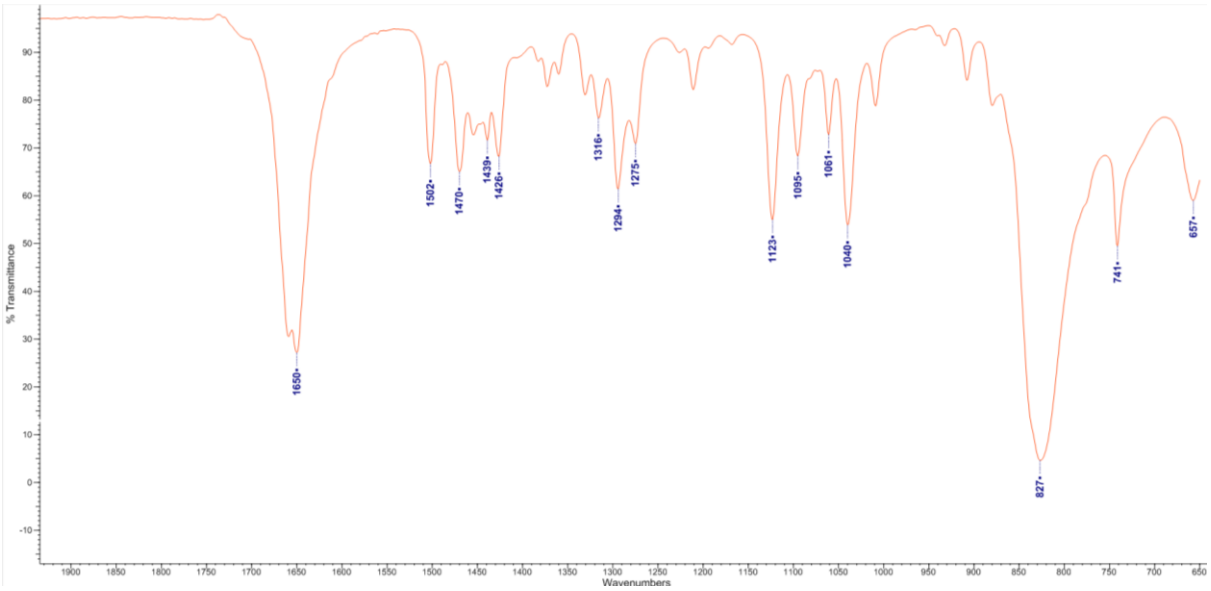
{[$Na_3(\mu_3$-**1**$)_3(\mu_2$-**1**$)$][$(PF_6)_3$]}$_n$ (**5**)



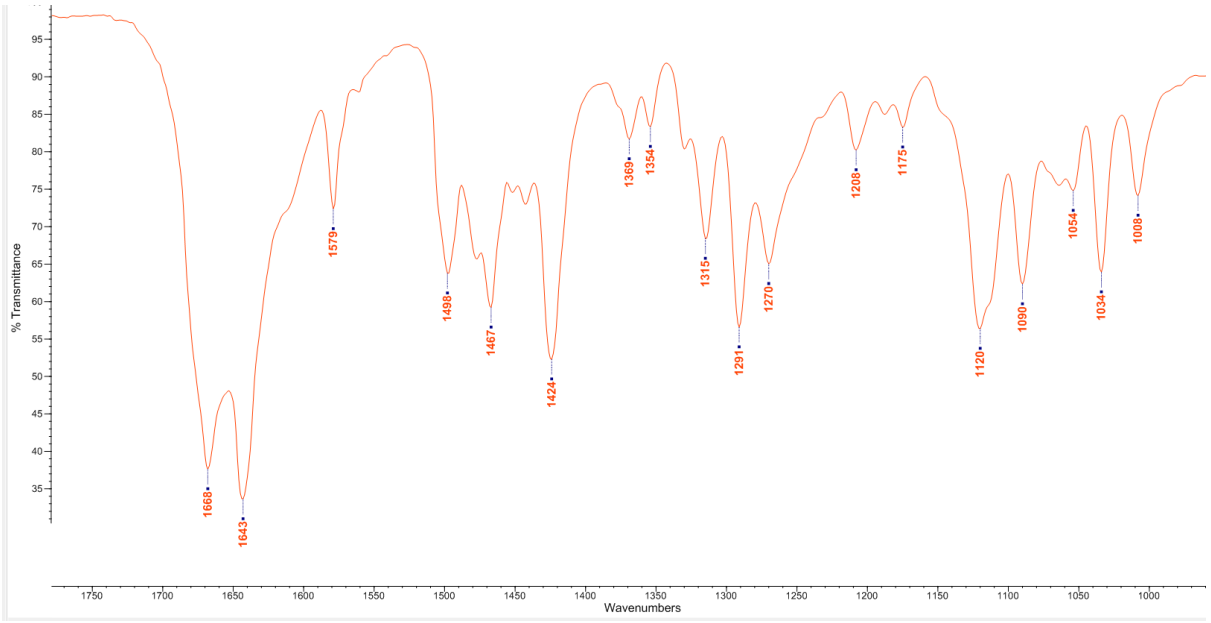


$[Na_2(\mu\text{-}\mathbf{1})_2(MeOH)_2](PF_6)_2$ (**6**)

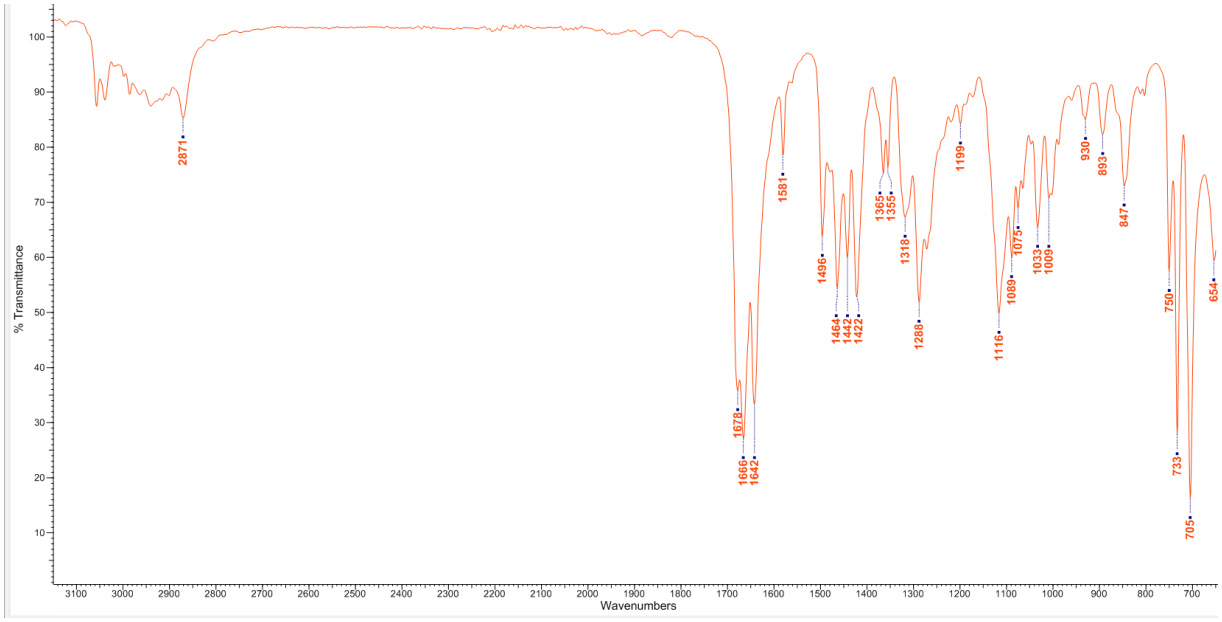


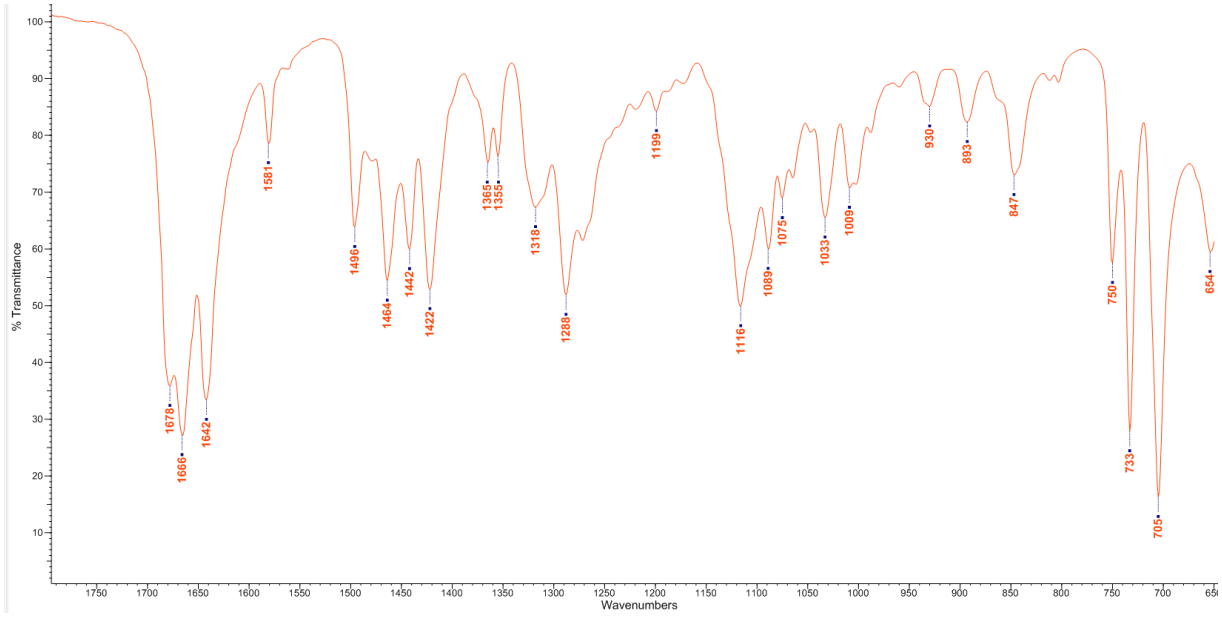


$[Na_2(\mathbf{1})_2(H_2O)_4](BPh_4)_2$ (**7**)

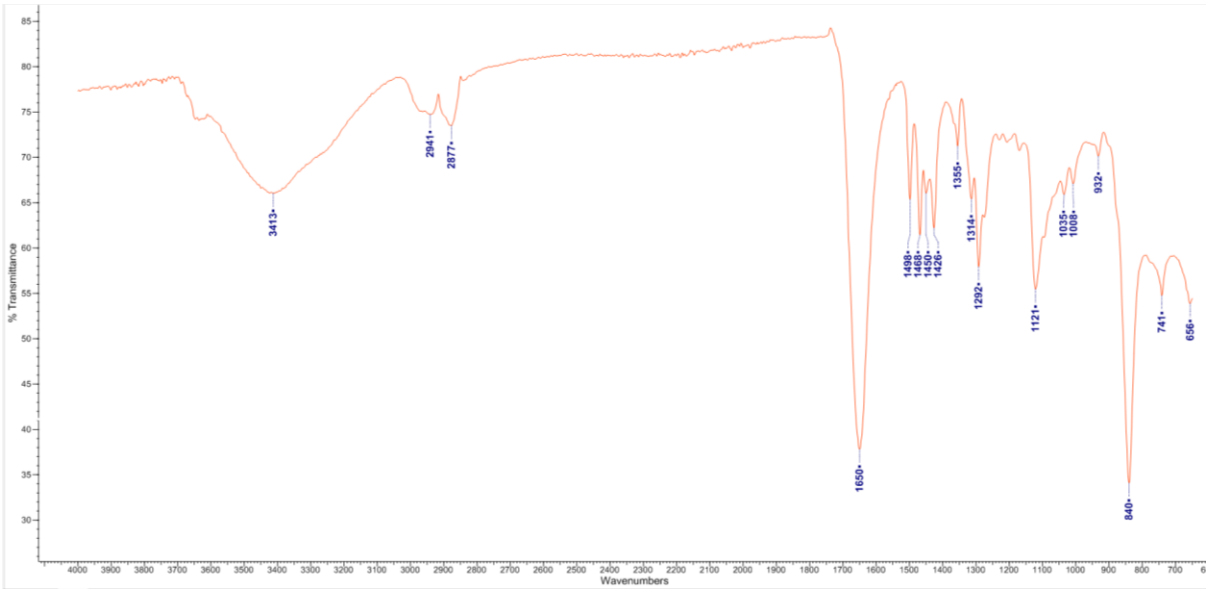


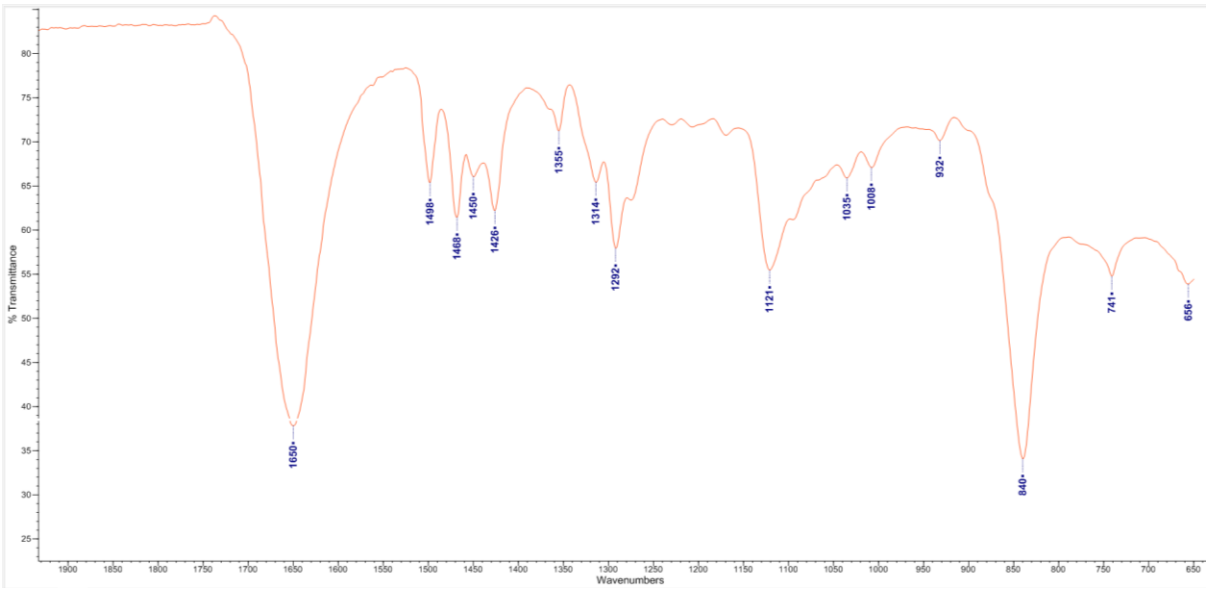
$[Na_2(\mathbf{1})_4](BPh_4)_2$ (**8**)



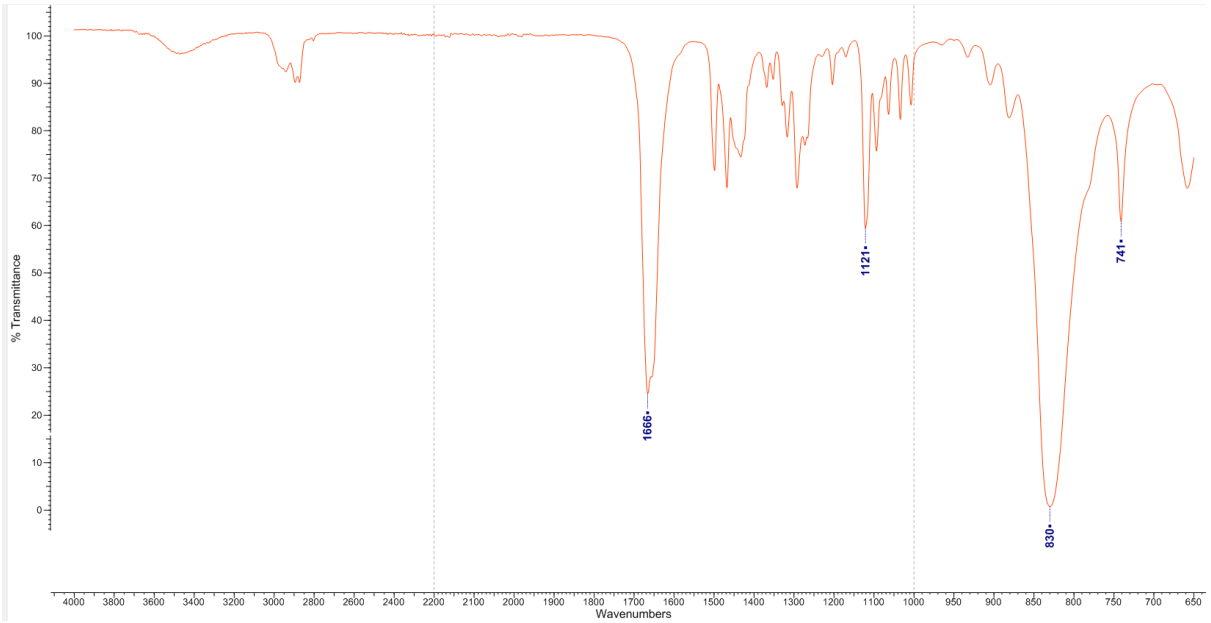


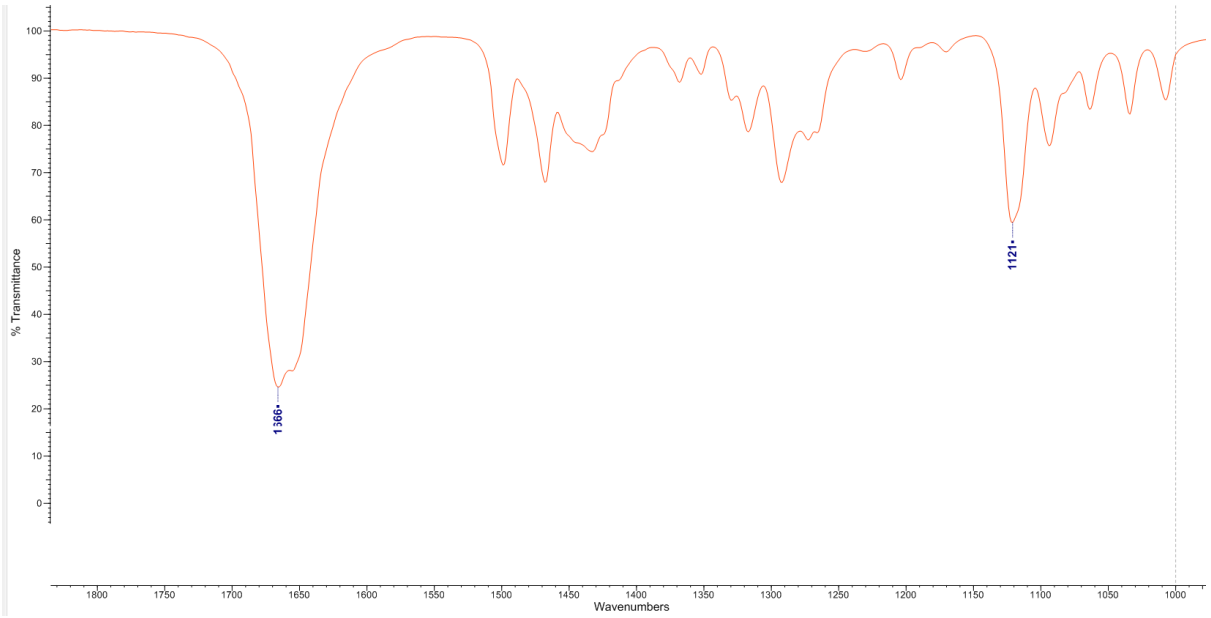
[K(**1**)$_2$]PF$_6$ (**9**)



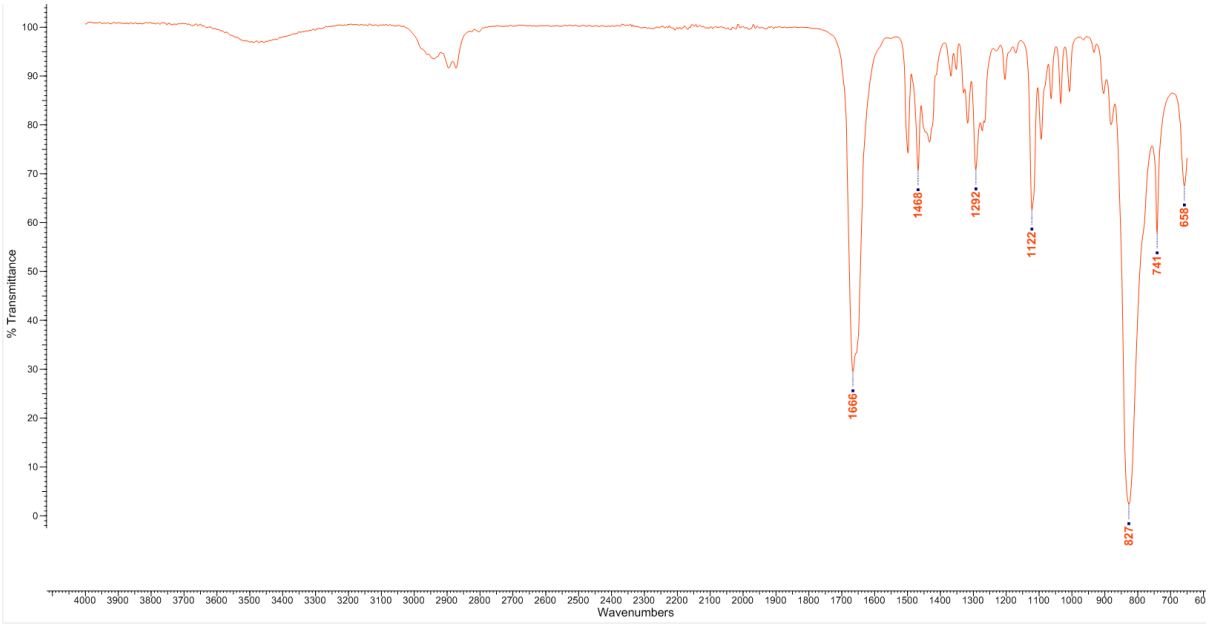


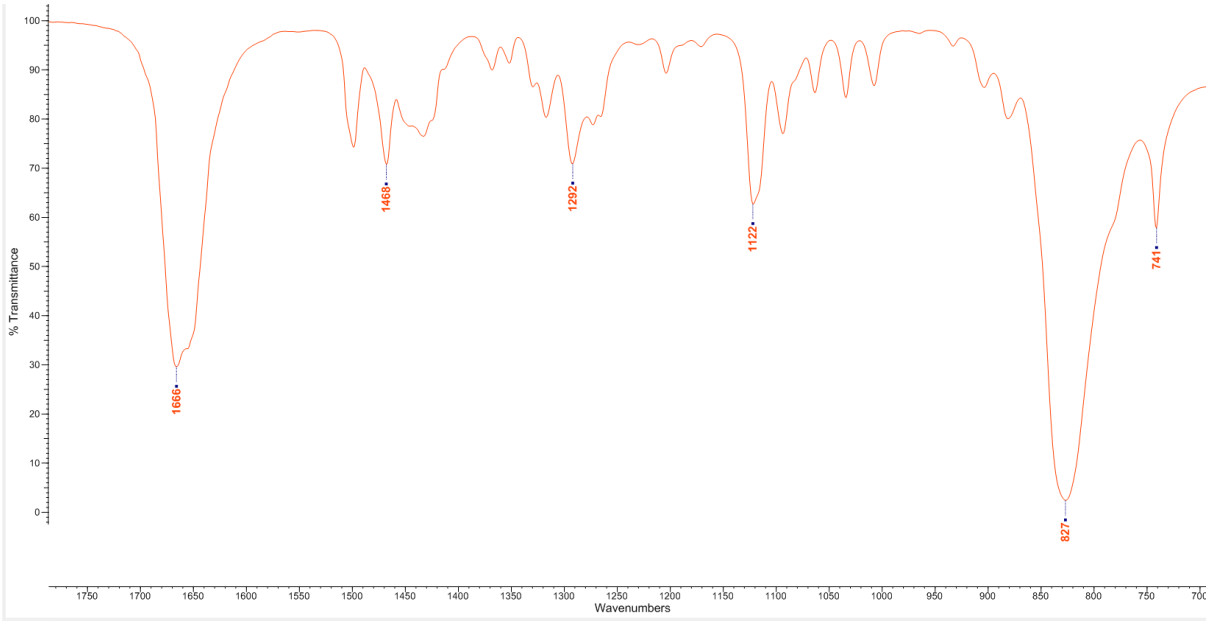
$[K_4(\mu_4\text{-}H_2O)_2(\mu\text{-}\mathbf{1})_4](PF_6)_4$ (**10a**)



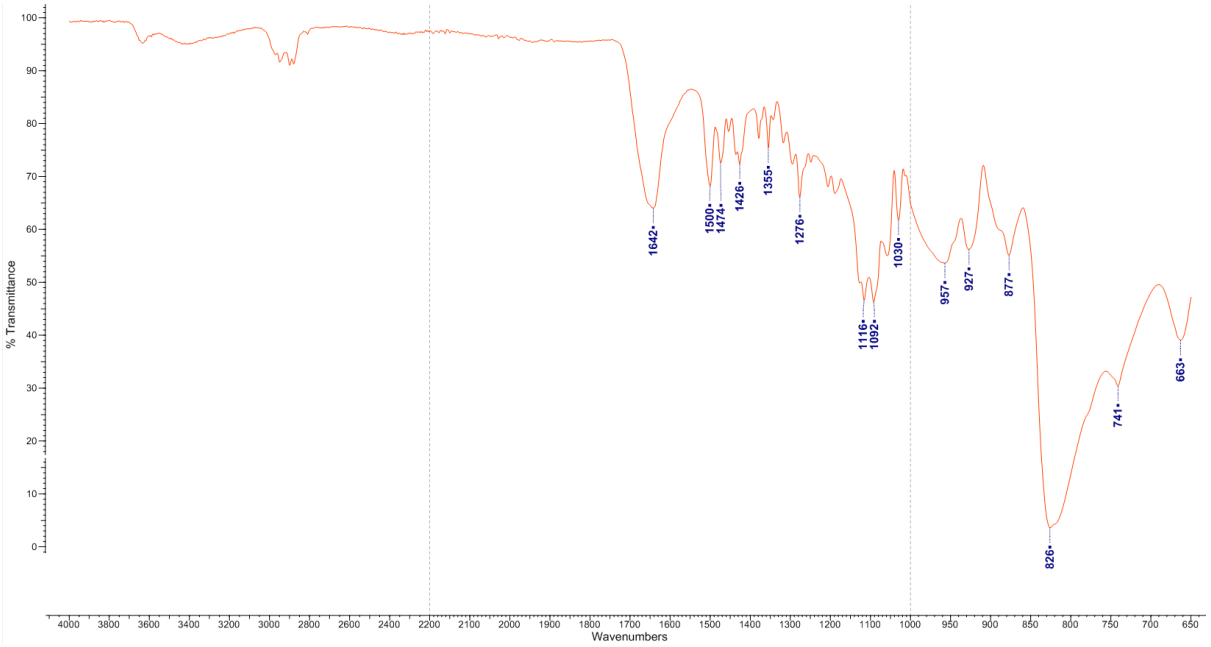


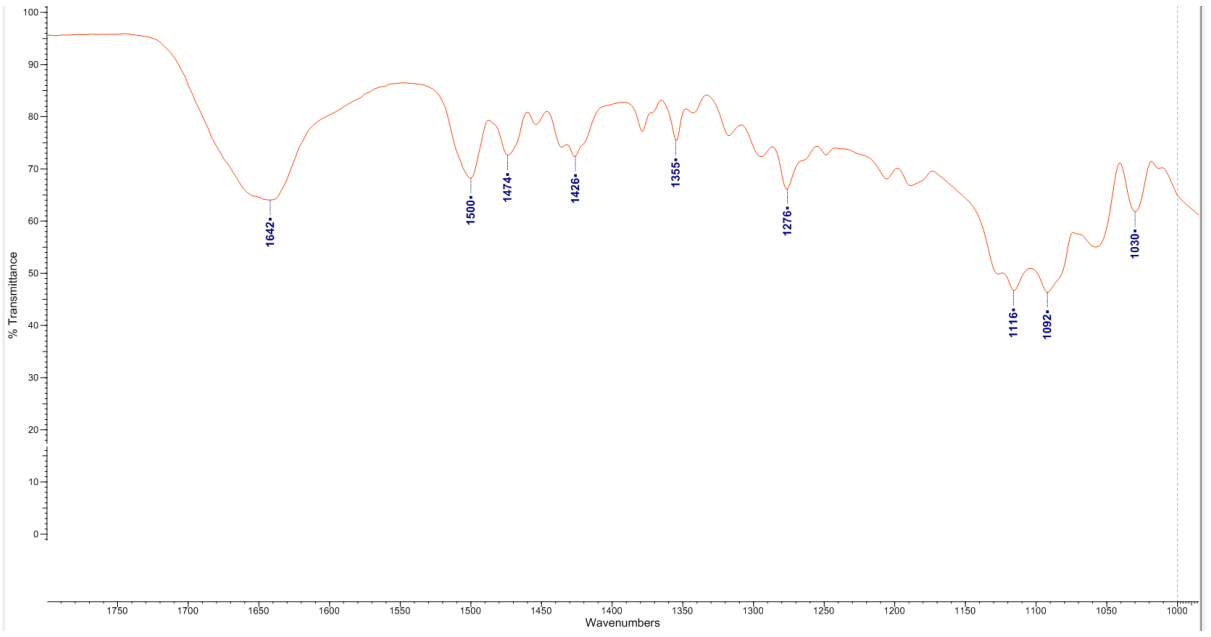
$[K_4(\mu_4\text{-}H_2O)_2(\mu\text{-}\mathbf{1})_4](PF_6)_4$ (**10b**)



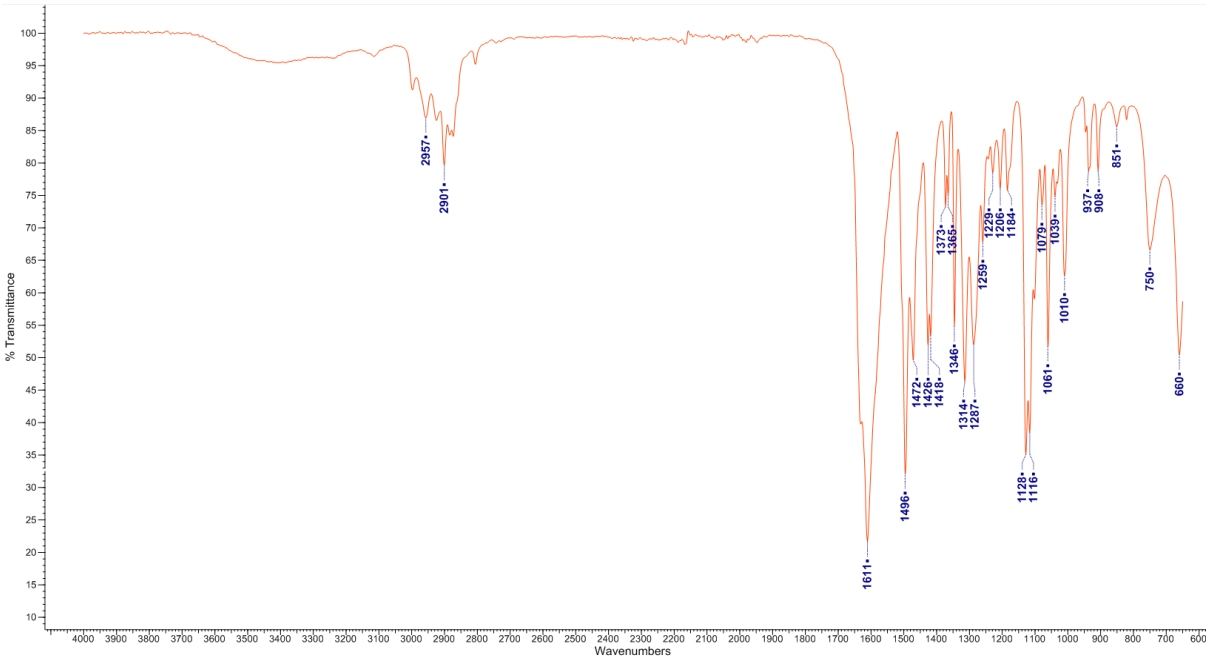


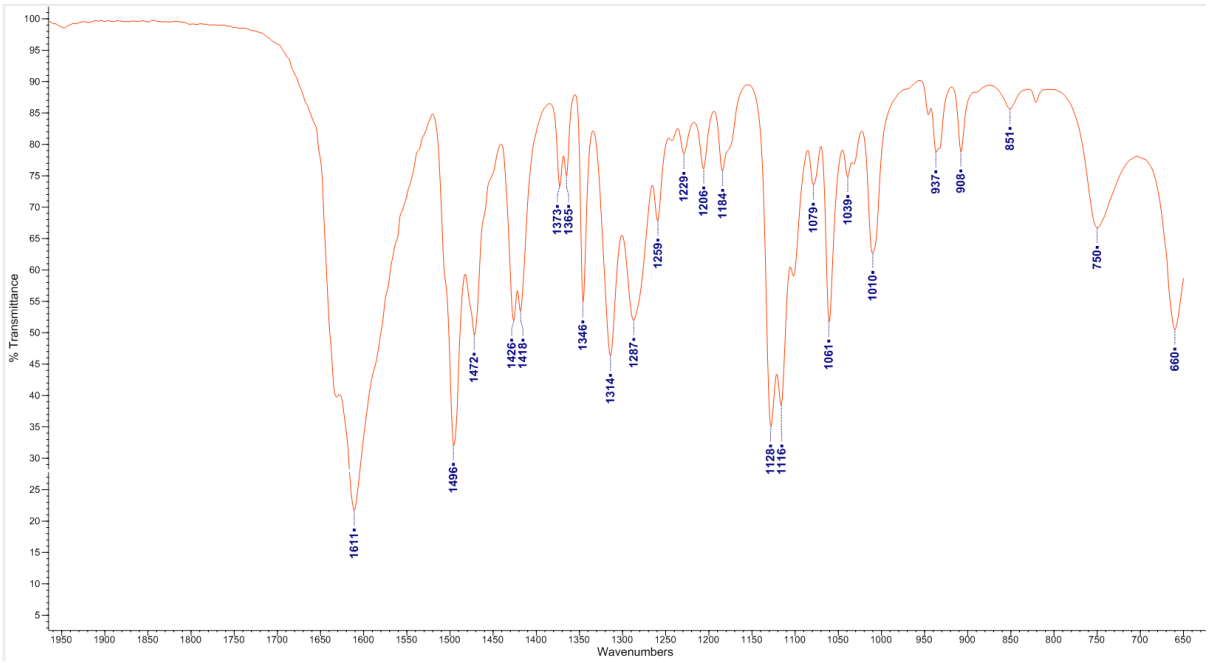
Protonation (**1**·HPF_6)



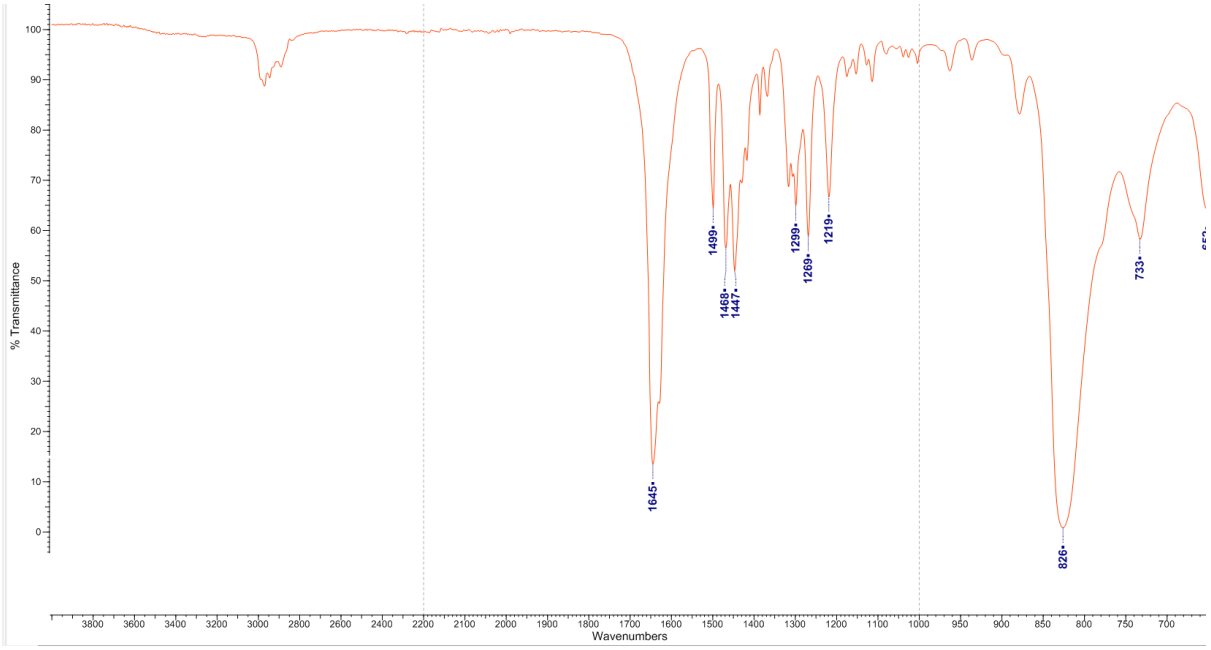


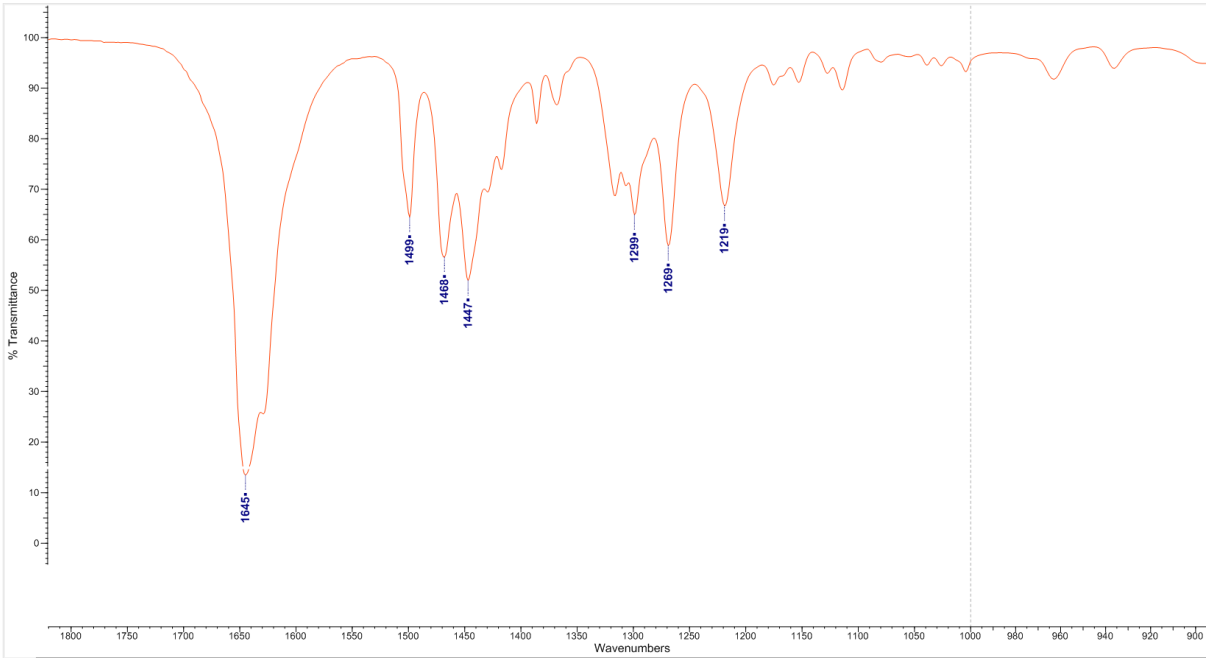
$\{ZnCl_2(\mu\text{-}\mathbf{1})\}_n$ (**11**)





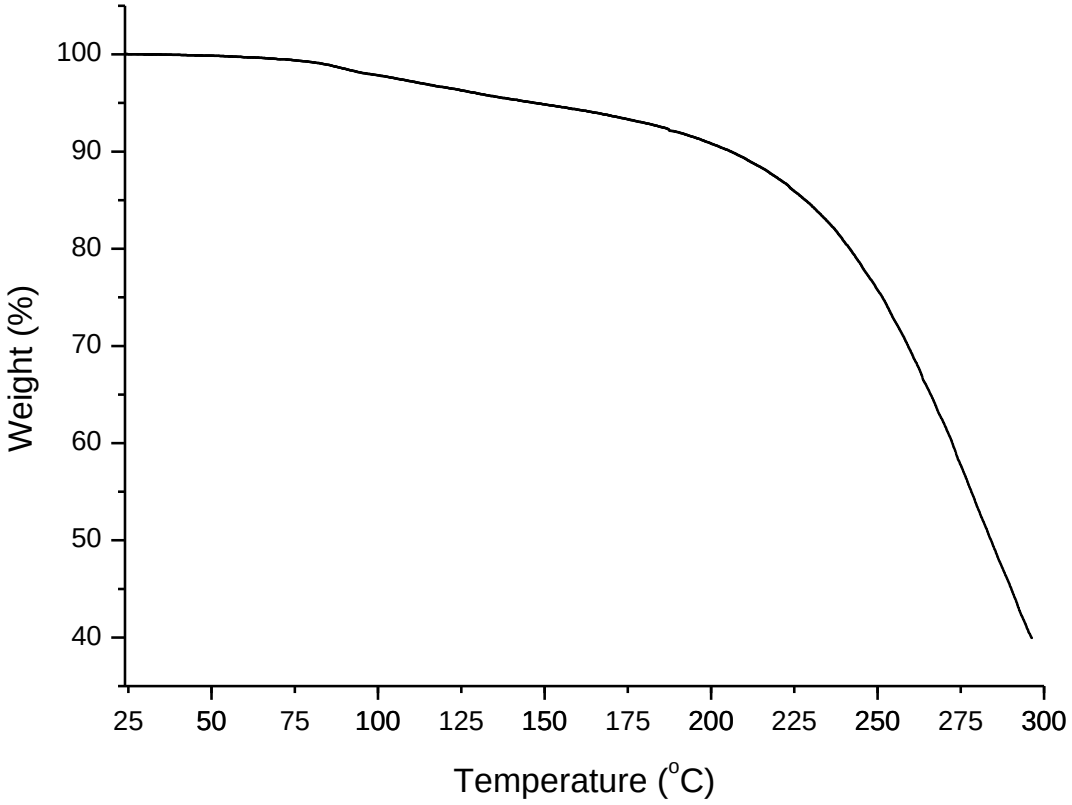
{[Na_2(μ-**2**)$_3$]($(PF_6)_2$}$_n$ (**12**)





TGA Analysis

TGA curve for $[Na_2(\mu\text{-}\mathbf{1})_2(MeOH)_2](PF_6)_2$ (**6**)



The initial loss up to 182.7 °C corresponds to a weight loss of 7.26% which equates to the loss of two molecules of methanol.

$[Na_2(\mu\text{-}\mathbf{1})_2(MeOH)_2](PF_6)_2 = C_{26}H_{48}N_4O_8Na_2P_2F_{12}$; FW = 880.60

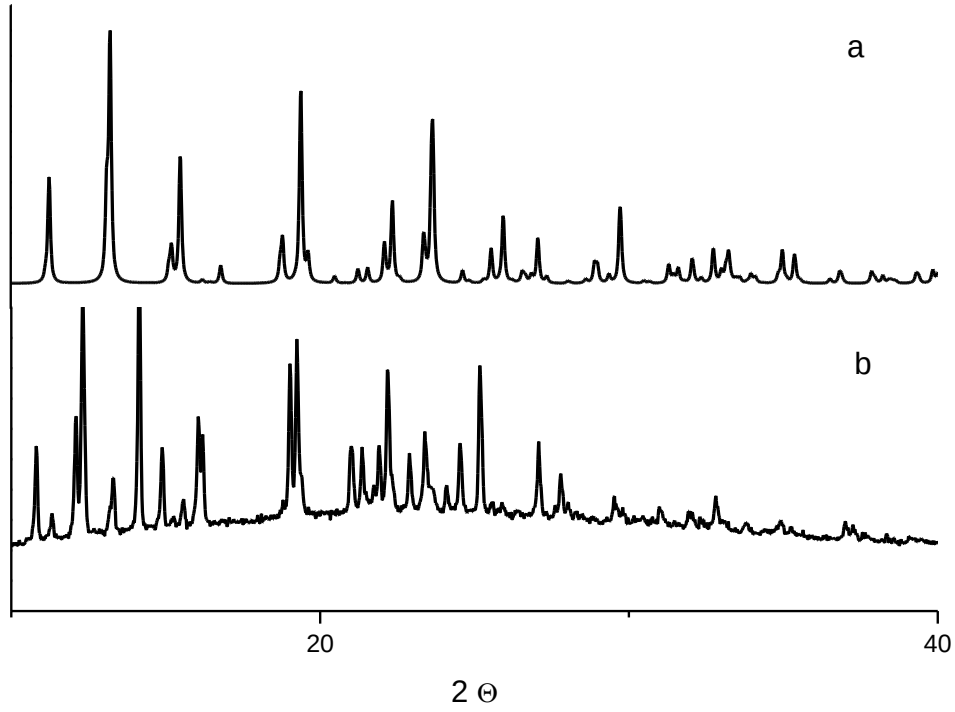
2MeOH molecules = $C_2H_8O_2$; FW = 64.1

% MeOH composition = (64.1/880.60) x 100 = 7.27%

Data for Zinc Chloride powder

Addition of 1-{2-[2-(2-oxo-pyrrolid-1-yl)-ethoxy]-ethyl}-pyrrolid-2-one directly to $ZnCl_2$ followed by addition of solvent (acetone or acetonitrile) results in the immediate formation of a white crystalline powder upon sonication. Examination of the resultant powder using PXRD (pattern b, below), and comparison to the predicted powder pattern (pattern a) based on the single crystal data for the $\{[ZnCl_2(\mu\text{-}\mathbf{1})]\}_n$ structure indicates formation of a new material.

PXRD reaction of **1** with $ZnCl_2$



(a) Calculated PXRD pattern based upon **11** $\{[ZnCl_2(\mu\text{-}\mathbf{1})]\}_n$; (b) experimental PXRD pattern of alternative zinc chloride product.

Elemental Analysis

For the new material found: C 38.19, H 5.45, N 7.29 %

Calculated for **11**: C 38.25, H 5.26, N 7.47 %

IR spectrum

IR of "new" powder: C=O 1615cm^{-1} and C-O ether 1127cm^{-1}, *cf.* **11**: C=O 1611cm^{-1} and C-O ether 1128cm^{-1}