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Azamacrocycle-Containing Gallium Phosphates: A New Class of Inorganic-Organic Hybrid Material

David S. Wragg, Gary B. Hix and Russell E. Morris*

School of Chemistry, University of St. Andrews

St. Andrews, Scotland KY16 9ST

Supplementary Material

Crystal Structure Details for $\text{GaPO}_4(\text{OH})_2\text{F}\cdot\text{Ga}(\text{C}_{10}\text{N}_4\text{H}_{24})$

The crystals were too small (max. size $30 \times 30 \times 15 \mu\text{m}$) for single crystal X-Ray data collection using a standard laboratory four circle diffractometer, so diffraction data were collected at low temperature (160K) using a Bruker AXS SMART CCD area-detector diffractometer on the high-flux single-crystal diffraction station 9.8 at CCLRC Daresbury Laboratory Synchrotron Radiation Source, Cheshire, UK. The experiment used X-rays of wavelength 0.6849\AA selected by a horizontally focusing silicon (111) monochromator and vertically focused by a cylindrically bent palladium-coated zerodur mirror.ⁱ The data set covered more than a hemisphere of reciprocal space with several series of exposures, each series with a different crystal orientation and each exposure taken over 0.2° rotation. Corrections were made for the synchrotron beam intensity decay as part of standard inter-frame scaling procedures.ⁱⁱ

The crystal structure of CYCLAM-GaPO was solved using the direct methods program SHELXS-97ⁱⁱ and refined with a full matrix least-squares technique using the program SHELXL-97.ⁱⁱ The final cycle of least-squares refinement included anisotropic displacement parameters for gallium phosphorus, oxygen, fluorine, carbon and nitrogen atoms. The hydrogen atoms of the CYCLAM were placed in chemically sensible positions and their positions recalculated after each cycle of refinement. In all 14162 integrated intensities were measured ($2.48^\circ < \theta < 28.08^\circ$), of which 2891 were unique and 2693 observed according to the criterion that $F^2 > 2\sigma(F^2)$. Final refinement of the 192 least-squares parameters converged to $wR(F^2_{\text{observed data}}) = 0.1214$, $wR(F^2_{\text{all data}}) = 0.1224$, $R(F_{\text{observed data}}) = 0.0407$, $S(F^2_{\text{all data}}) = 1.066$, max. residual electron density = $1.032e \text{ \AA}^{-3}$. Refinement on F^2 was carried out against all data; the observed criterion $F^2 > 2\sigma(F^2)$ was used only for calculating $wR(F^2_{\text{observed data}})$ and $R(F_{\text{observed data}})$ and is not relevant to the choice of reflections for refinement. The final unit cell refined to values of $a = 13.2558(5) \text{ \AA}$, $b = 10.4321(5) \text{ \AA}$, $c = 18.4382(9) \text{ \AA}$, Volume = $2549.7(2) \text{ \AA}^3$ at $T=160\text{K}$, spacegroup $Pbcn$, $\rho_{\text{calcd}} = 2.542\text{g cm}^{-3}$. Details of the final atomic coordinates and equivalent isotropic displacement are given in Table 1.

Table 1. Final atomic coordinates (\AA) and equivalent isotropic temperature factors (\AA^2) for CYCLAM-GaPO. Estimated standard deviations are given in parentheses.

Atom	x	y	z	U_{eq}
Ga(1)	0.85828(4)	0.24875(4)	0.73027(3)	0.00817(16)
Ga(2)	1.03374(4)	-0.02020(5)	0.64064(3)	0.01613(17)
Ga(3)	1.0000	0.5000	0.5000	0.00804(18)
P(1)	1.03739(8)	0.27208(11)	0.62236(6)	0.0084(2)
P(2)	0.82844(8)	-0.04431(11)	0.72430(6)	0.0085(2)
O(1)	0.7915(2)	0.0950(3)	0.72936(18)	0.0130(7)
O(2)	1.1244(3)	-0.0865(3)	0.70442(19)	0.0190(7)
O(3)	1.0584(3)	-0.1154(3)	0.56256(18)	0.0137(7)
O(4)	0.9271(2)	0.2866(3)	0.64704(18)	0.0146(7)
O(5)	0.7593(2)	0.3694(3)	0.70603(18)	0.0130(6)
O(6)	1.0619(3)	0.1335(3)	0.60451(19)	0.0199(8)
O(7)	0.9036(3)	-0.0574(3)	0.6619(2)	0.0167(7)
O(8)	1.0529(2)	0.3529(3)	0.55598(17)	0.0116(6)
O(9)	0.8922(3)	0.3191(3)	0.81706(19)	0.0177(7)
F(1)	1.0000	0.1289(4)	0.7500	0.0218(9)
N(1)	0.9197(3)	0.3770(4)	0.4353(2)	0.0119(7)
N(2)	1.1158(3)	0.4718(4)	0.4283(2)	0.0098(7)
C(1)	1.2173(3)	0.4599(5)	0.4616(3)	0.0143(9)
C(2)	0.9757(4)	0.3713(5)	0.3652(2)	0.0141(9)
C(3)	1.0872(4)	0.3604(5)	0.3822(3)	0.0151(9)
C(4)	0.8109(4)	0.4034(5)	0.4255(3)	0.0167(10)
C(5)	1.2436(4)	0.5810(5)	0.5024(3)	0.0172(10)

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  '-x, -y, -z'
  'x-1/2, y-1/2, -z-1/2'
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  '-x-1/2, y-1/2, z'

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Refinement of F2 against ALL reflections. The weighted R-factor
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on F, with F set to zero for negative F2. The threshold expression
of
F2 > 2sigma(F2) is used only for calculating R-factors(gt) etc.
and is

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not relevant to the choice of reflections for refinement. R-factors based

on F^2 are statistically about twice as large as those based on F , and R-

factors based on ALL data will be even larger.

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Ga2 Ga 1.03374(4) -0.02020(5) 0.64064(3) 0.01613(17) Uani 1 1 d . . .
Ga3 Ga 1.0000 0.5000 0.5000 0.00804(18) Uani 1 2 d S . .
P1 P 1.03739(8) 0.27208(11) 0.62236(6) 0.0084(2) Uani 1 1 d . . .
P2 P 0.82844(8) -0.04431(11) 0.72430(6) 0.0085(2) Uani 1 1 d . . .
O1 O 0.7915(2) 0.0950(3) 0.72936(18) 0.0130(7) Uani 1 1 d . . .
O2 O 1.1244(3) -0.0865(3) 0.70442(19) 0.0190(7) Uani 1 1 d . . .
O3 O 1.0584(3) -0.1154(3) 0.56256(18) 0.0137(7) Uani 1 1 d . . .
O4 O 0.9271(2) 0.2866(3) 0.64704(18) 0.0146(7) Uani 1 1 d . . .
O5 O 0.7593(2) 0.3694(3) 0.70603(18) 0.0130(6) Uani 1 1 d . . .
O6 O 1.0619(3) 0.1335(3) 0.60451(19) 0.0199(8) Uani 1 1 d . . .
O7 O 0.9036(3) -0.0574(3) 0.6619(2) 0.0167(7) Uani 1 1 d . . .
O8 O 1.0529(2) 0.3529(3) 0.55598(17) 0.0116(6) Uani 1 1 d . . .
O9 O 0.8922(3) 0.3191(3) 0.81706(19) 0.0177(7) Uani 1 1 d . . .
F1 F 1.0000 0.1289(4) 0.7500 0.0218(9) Uani 1 2 d S . .
N1 N 0.9197(3) 0.3770(4) 0.4353(2) 0.0119(7) Uani 1 1 d . . .
N2 N 1.1158(3) 0.4718(4) 0.4283(2) 0.0098(7) Uani 1 1 d . . .
C1 C 1.2173(3) 0.4599(5) 0.4616(3) 0.0143(9) Uani 1 1 d . . .
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H2B H 0.9537 0.2977 0.3371 0.017 Uiso 1 1 calc R . .
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C4 C 0.8109(4) 0.4034(5) 0.4255(3) 0.0167(10) Uani 1 1 d . . .
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H4B H 0.8028 0.4811 0.3972 0.020 Uiso 1 1 calc R . .
C5 C 1.2436(4) 0.5810(5) 0.5024(3) 0.0172(10) Uani 1 1 d . . .
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0.0087(14)
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0.0048(13)
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O9 0.0170(17) 0.0199(17) 0.0160(16) -0.0090(14) -0.0092(14)
0.0087(14)
F1 0.027(2) 0.019(2) 0.019(2) 0.000 -0.0073(18) 0.000
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0.0002(15)
N2 0.0079(17) 0.0136(18) 0.0079(17) -0.0022(14) 0.0021(14) -
0.0014(14)
C1 0.010(2) 0.021(2) 0.013(2) 0.0030(18) 0.0023(17) 0.0024(18)
C2 0.017(2) 0.015(2) 0.010(2) -0.0039(17) 0.0000(17) -0.0011(18)
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C5 0.009(2) 0.025(2) 0.019(2) 0.002(2) -0.0020(17) -0.0038(19)

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planes.

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Ga2 O7 1.811(3) . ?
Ga2 O2 1.818(3) . ?
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P2 O5 Ga1 152.6(2) 8_765 . ?
P1 O6 Ga2 137.2(2) . . ?
P2 O7 Ga2 139.8(2) . . ?
P1 O8 Ga3 144.4(2) . . ?
P1 O9 Ga1 131.4(2) 3_756 . ?
Ga1 F1 Ga1 113.67(18) . 3_756 ?
C4 N1 C2 112.7(4) . . ?
C4 N1 Ga3 117.4(3) . . ?
C2 N1 Ga3 105.7(3) . . ?
C3 N2 C1 113.7(4) . . ?
C3 N2 Ga3 106.8(3) . . ?
C1 N2 Ga3 115.1(3) . . ?
N2 C1 C5 110.2(4) . . ?
N1 C2 C3 107.9(4) . . ?
N2 C3 C2 108.0(4) . . ?
N1 C4 C5 112.1(4) . 5_766 ?
C1 C5 C4 114.5(4) . 5_766 ?

_diffn_measured_fraction_theta_max 0.930
_diffn_reflms_theta_full 28.08
_diffn_measured_fraction_theta_full 0.930
_refine_diff_density_max 1.232
_refine_diff_density_min -1.791
_refine_diff_density_rms 0.205

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#==END

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- (ii) Programs SADABS, SHELXS-97 and SHELXL-97, G.M. Sheldrick, University of Göttingen, Germany, **1997**.

