# The eCrystals Federation

**Repository Curation Service Environments (RECURSE) Workshop**
**National e-Science Centre, Edinburgh**

**4th International Digital Curation Conference**
**"Radical Sharing: Transforming Science?"**
**1-3rd December 2008**
**Edinburgh, Scotland**

**Manjula Patel**
**UKOLN, University of Bath, UK**

# Context

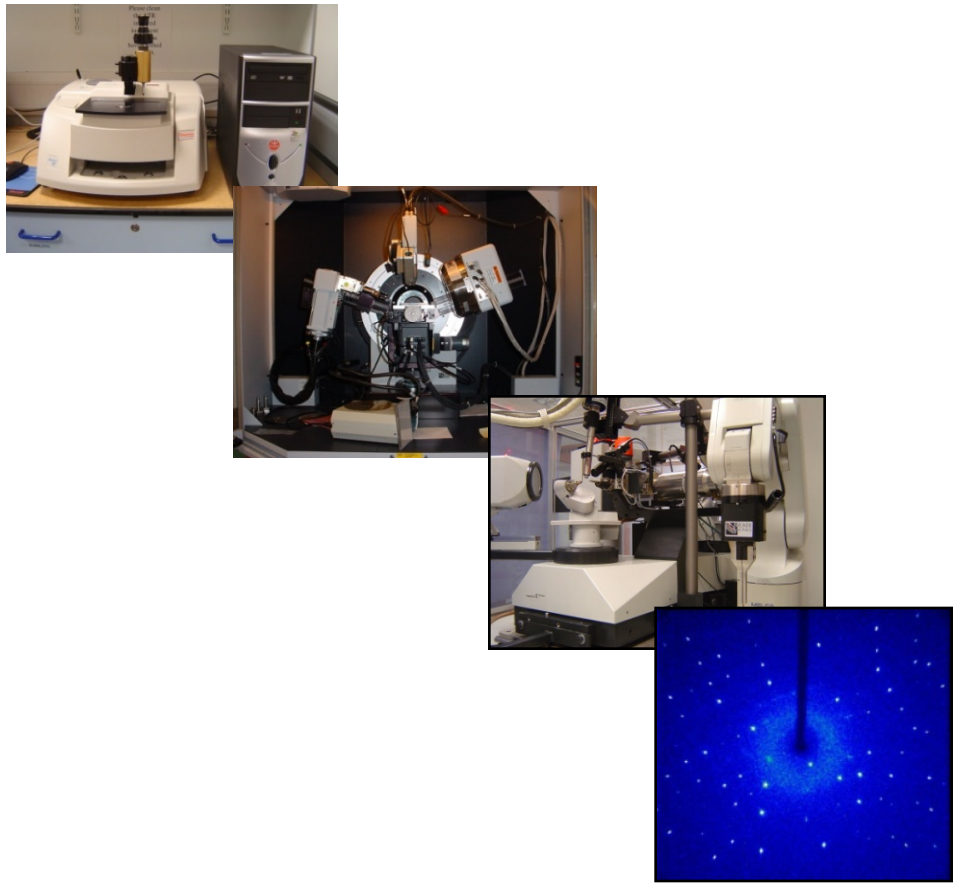
- The data deluge
  - Advances in instrumentation, data storage technologies, computational power and improvements in algorithms
  - Development of grid and cyber infrastructures
- Actual nature of science is changing
  - Mining and analysis of large datasets (e.g. Protein Data Bank, GenBank)
  - Open Science (e.g.Open Notebook Science; myExperiment)
- High quality data are the raw materials of contemporary e-science
  - Verification; Validation; Replication
  - Predictive science
  - Innovative scientific endeavour
- S. Carlson, *Lost in a Sea of Science Data*, The Chronicle of Higher Education, June 2006

  "To vet experiments, correct errors, or find new breakthroughs, scientists desperately need better ways to store and retrieve research data"

  "Data from Big Science is … easier to handle, understand and archive. Small Science is horribly heterogeneous and far more vast. In time Small Science will generate 2-3 times more data than Big Science."

# Crystallography –The Science

- Sub-discipline of chemistry
- Concerned with determining the structure of a molecule and its 3D orientation with respect to other molecules in a crystal
- Analysis of diffraction patterns obtained from X-ray scattering experiments
- Focus on laboratory based experimental technique of chemical crystallography undertaken at the EPSRC National Crystallography Service (NCS), UK

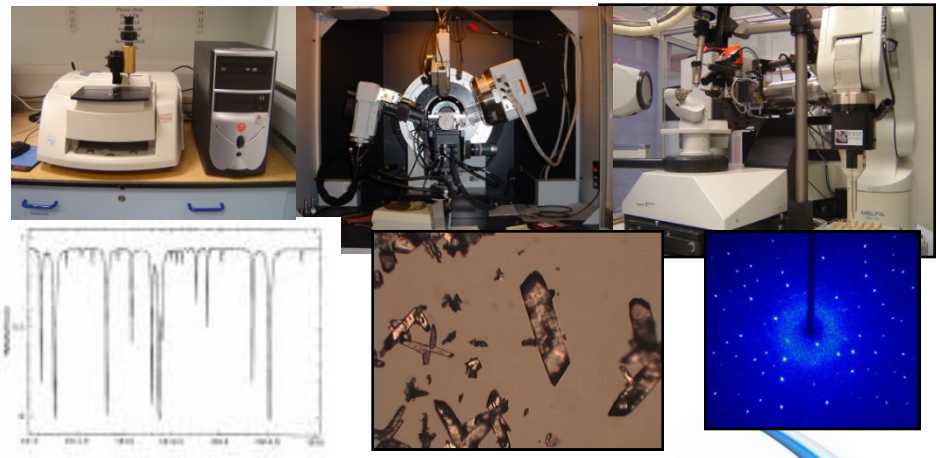


Images from Simon Coles (NCS), 2006

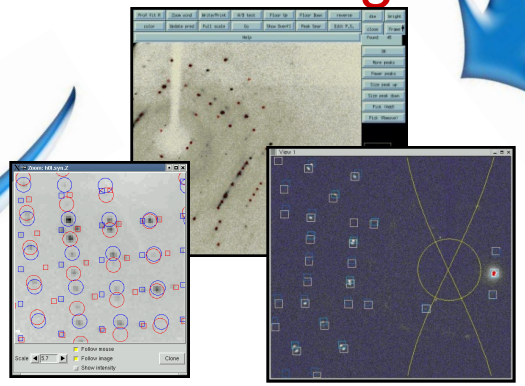
# Data Generation

## Synthesis

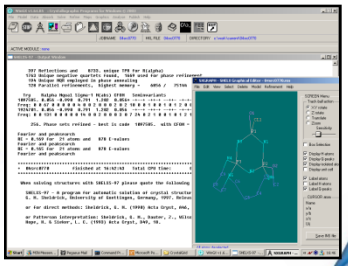
## Data Collection



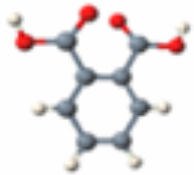
## Data Processing



## Data Workup



## Publication

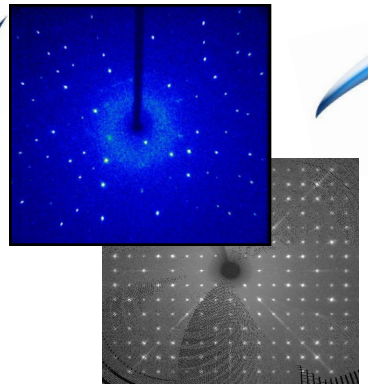
Cambridge Crystallographic Data Centre

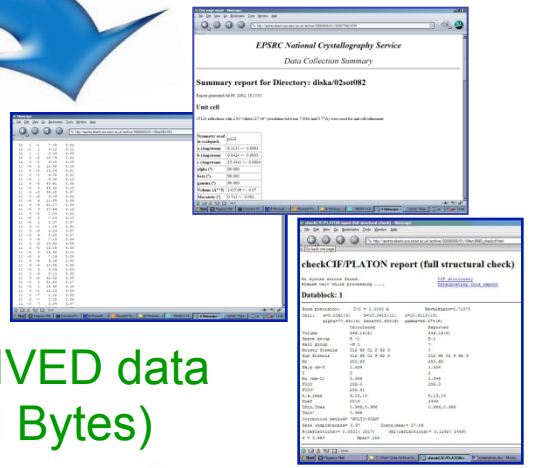
Adapted from Simon Coles (NCS), 2007

# Data Volumes

Science & Technology Facilities Council

RAW data
(G Bytes)





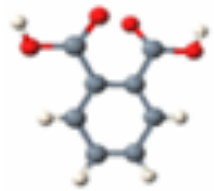

DERIVED data
(M Bytes)

Laboratory; Institution

Subject Repository; Data Centre; Public Domain

Cambridge Crystallographic Data Centre

RESULTS data
(K Bytes)




Adapted from Simon Coles (NCS), 2007

# Community & Current Practice (1)

- Relatively organised approach to data (crystallography data are highly structured)
- Convention is to share derived or reduced data, access to raw data is rare
- Crystallography Information File (CIF) is a de facto exchange standard
  - Maintained by International Union of Crystallography (IUCr)
- Heterogeneity in instrumentation and associated software
- Established system for publishing crystallographic data in UK (Cambridge Crystallographic Data Centre-CCDC)
- Other major databanks
  - Germany (inorganic molecule database)
  - Canada (metals database)
  - US (Protein Data Bank -PDB)

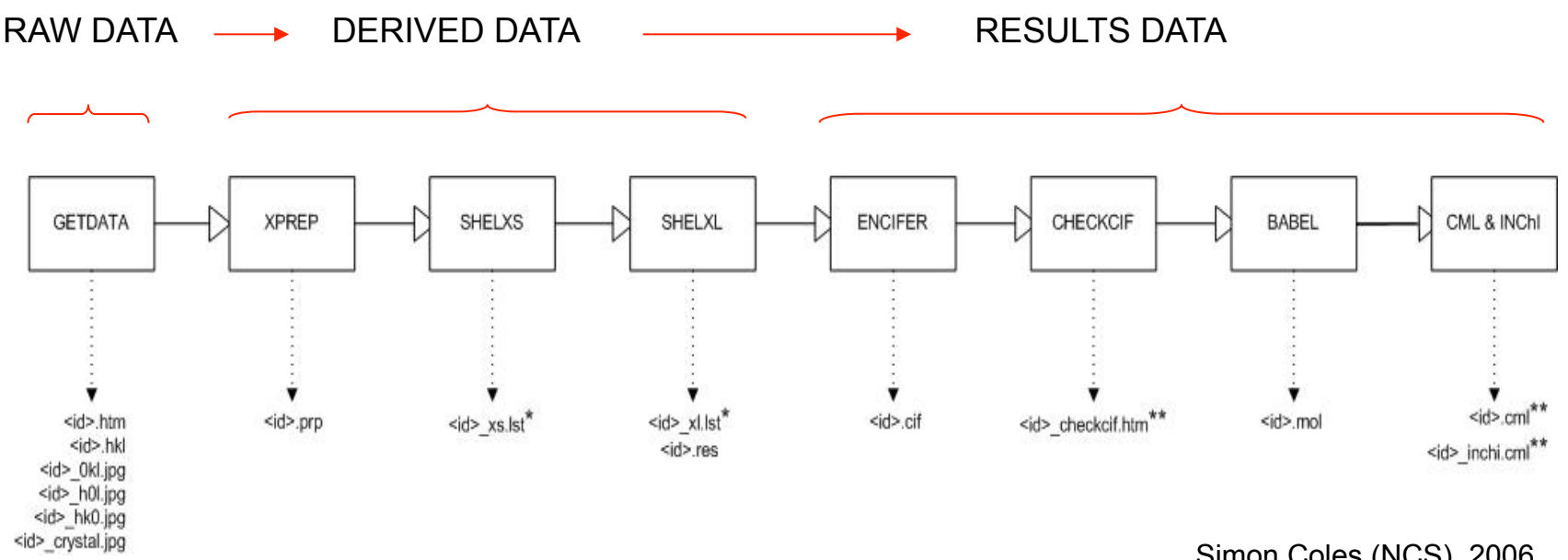
# Community & Current Practice (2)

- Publishing datasets
  - Alongside journal articles through publisher mandates
  - Researchers often wish to retain exclusive use of their data
  - Lack of career rewards with respect to data creation and publishing
- Smaller projects at greatest risk
  - Sometimes CIF retained but raw data discarded
  - Data often stored on DVDs or laptops
  - Distributed, local storage -shortage of local curation expertise
  - Quality of metadata for datasets is variable
- Open access
  - eCrystals Federation Project
  - CrystalEye
  - ReciprocalNet (US, Australia, UK)
  - Crystallography Open Database (COD)
  - Chemistry Central (open access publisher)

# Building the eCrystals Repository

- Phenomenal growth in amount of data generated from experiments
  - 40 years ago a PhD student would determine 2-3 structures for a thesis; this can now be easily achieved in a single day
- Only a small proportion is widely and easily accessible
  - Estimated that < 50% of crystal structures are published [Allen 2004]
  - Current data publication process is a bottleneck
- eBank-UK Project
  - JISC funded; three phases Sept. 2003-June 2007
  - UKOLN (lead), University of Southampton, University of Manchester
- eCrystals data repository
  - Open access and rapid dissemination of derived and results data from crystallography experiments
  - Repository platform: ePrints.org software V3
  - Supported by learned society (IUCr) and subject repository (CCDC)
- Linking research data to publications and scholarly communication
- Metadata harvesting and aggregation (OAI-PMH)

# EPSRC NCS Crystal Structure Determination Workflow

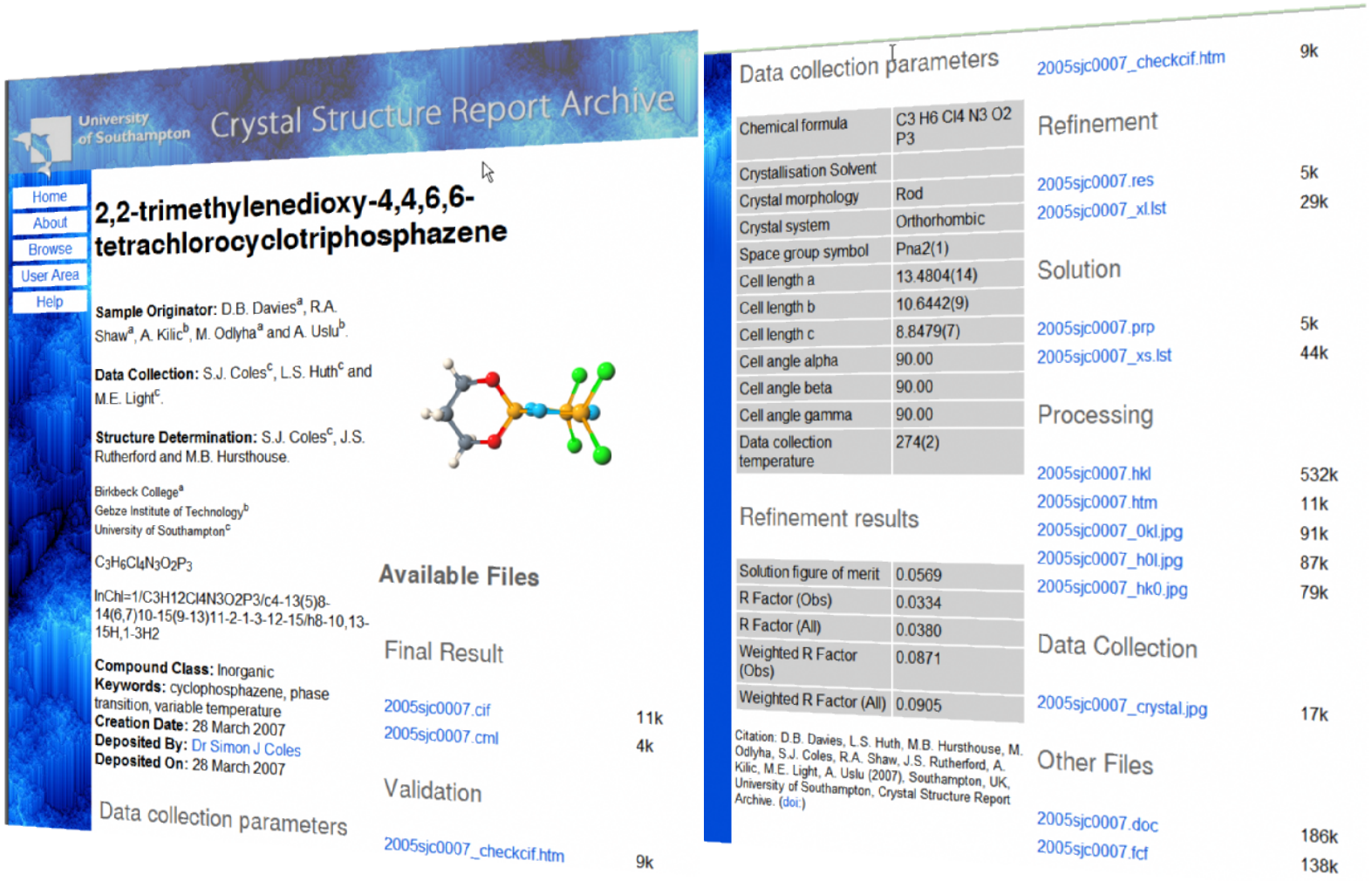



Simon Coles (NCS), 2006

- Initialisation: mount new sample
- Collection: collect data
- Processing: process and correct images
- Solution: solve structures

- Refinement: refine structure
- CIF: produce Crystallographic Information File
- Validation: chemical & crystallographic checks
- Report: generate Crystal Structure Report
- CML, INChI

# eCrystals Data Repository: Example Crystal Structure Report



University of Southampton — Crystal Structure Report Archive

Home | About | Browse | User Area | Help

## 2,2-trimethylenedioxy-4,4,6,6-tetrachlorocyclotriphosphazene

**Sample Originator:** D.B. Davies[a], R.A. Shaw[a], A. Kilic[b], M. Odlyha[a] and A. Uslu[b].

**Data Collection:** S.J. Coles[c], L.S. Huth[c] and M.E. Light[c].

**Structure Determination:** S.J. Coles[c], J.S. Rutherford and M.B. Hursthouse.

Birkbeck College[a]
Gebze Institute of Technology[b]
University of Southampton[c]

$C_3H_6Cl_4N_3O_2P_3$

InChI=1/C3H12Cl4N3O2P3/c4-13(5)8-14(6,7)10-15(9-13)11-2-1-3-12-15/h8-10,13-15H,1-3H2

**Compound Class:** Inorganic
**Keywords:** cyclophosphazene, phase transition, variable temperature
**Creation Date:** 28 March 2007
**Deposited By:** Dr Simon J Coles
**Deposited On:** 28 March 2007

Data collection parameters

Available Files

Final Result

2005sjc0007.cif 11k
2005sjc0007.cml 4k

Validation

2005sjc0007_checkcif.htm 9k

Data collection parameters

| | |
|---|---|
| Chemical formula | C3 H6 Cl4 N3 O2 P3 |
| Crystallisation Solvent | |
| Crystal morphology | Rod |
| Crystal system | Orthorhombic |
| Space group symbol | Pna2(1) |
| Cell length a | 13.4804(14) |
| Cell length b | 10.6442(9) |
| Cell length c | 8.8479(7) |
| Cell angle alpha | 90.00 |
| Cell angle beta | 90.00 |
| Cell angle gamma | 90.00 |
| Data collection temperature | 274(2) |

Refinement results

| | |
|---|---|
| Solution figure of merit | 0.0569 |
| R Factor (Obs) | 0.0334 |
| R Factor (All) | 0.0380 |
| Weighted R Factor (Obs) | 0.0871 |
| Weighted R Factor (All) | 0.0905 |

Citation: D.B. Davies, L.S. Huth, M.B. Hursthouse, M. Odlyha, S.J. Coles, R.A. Shaw, J.S. Rutherford, A. Kilic, M.E. Light, A. Uslu (2007). Southampton, UK, University of Southampton, Crystal Structure Report Archive. (doi:)

2005sjc0007_checkcif.htm 9k

Refinement

2005sjc0007.res 5k
2005sjc0007_xl.lst 29k

Solution

2005sjc0007.prp 5k
2005sjc0007_xs.lst 44k

Processing

2005sjc0007.hkl 532k
2005sjc0007.htm 11k
2005sjc0007_0kl.jpg 91k
2005sjc0007_h0l.jpg 87k
2005sjc0007_hk0.jpg 79k

Data Collection

2005sjc0007_crystal.jpg 17k

Other Files

2005sjc0007.doc 186k
2005sjc0007.fcf 138k

# Linking Data to Publications

**eBank UK Demo**

## Crystal Structure Data Reports

Crystal Structure Report of 2-(N-Ferrocenylmethylcarbamoyl)-5-(N-phenylcarbamoyl)-3,4-diphenyl pyrrole

**Creator(s):** Hursthouse, Michael B., Light, Mark E., Coles, Simon J., Horton, Peter N., Gale, Phil A., Denuault, G., Warriner, C. N.
**Date released:** 23/05/2004
**Empirical Formula:** C35H29FeN3O2
**IUPAC name:** 2-(N-Ferrocenylmethylcarbamoyl)-5-(N-phenylcarbamoyl)-3,4-diphenyl pyrrole
**CCDC code:** XUZSIU
**Compound Class:** Organic
**General keywords:** Supramolecular Chemistry
**Related article:** ?A URI citation?

**Available Datafiles**

**CIF file**
**processing Dataset**
**refinement Dataset**
**solution Dataset**

Crystal Structure Report of 2-(N-Ferrocenylcarbamoyl)-5-(methoxycarbonyl)-3,4-diphenylpyrrole

**Creator(s):** Hursthouse, Michael B., Coles, Simon J., Light, Mark E., Horton, Peter N., Gale, Phil A., Denuault, G., Warriner, C. N.
**Date released:** 23/05/2004
**Empirical Formula:** C29H24FeN2O3
**IUPAC name:** 2-(N-Ferrocenylcarbamoyl)-5-(methoxycarbonyl)-3,4-diphenylpyrrole
**CCDC code:** XUZSOA
**Compound Class:** Organometallic
**General keywords:** Supramolecular Chemistry
**Related article:** ?A URI citation?

**Available Datafiles**

**CIF file**
**processing Dataset**
**refinement Dataset**
**solution Dataset**

## Publications

A supramolecular assembly: aquatris(pentafluorophenyl)borane as its mixed dimethyl sulfone and water solvate, (H2O)B(C6F5)3Me2SO2H2O

The title compound, C18H2BF15OH2OC2H6O2, obtained by crystallization of a product formed from a reaction mixture containing B(C6F5)3 and Me2SO2 (and H2O) in hexane, was characterized in the solid state as a supramolecular assembly containing water adducts of tris (pentafluorophenyl)borane, (H2O)B(C6F5)3, linked together by a network of hydrogen bonds involving one additional H2O and one additional Me2SO2 molecule per adduct molecule.

**Creator(s):** Coles, Simon J., Hursthouse, Michael B., Beckett, Michael A., Dutton, Michael
Acta Crystallogr E Struct Rep OnlineVol 59 Issue Pt 9 pp. o1354 - o1356
**DOI:**
**Download from:** http://scripts.iucr.org/cgi-bin/getarticleid?issn=1600-5368&volume=59&fpage=o1354&details=yes

Structural investigations of phosphorus-nitrogen compounds. 5. Relationships between molecular parameters of 2,2-diphenyl-4,6-cis-oxytetra(ethyleneoxy)-4,6-R2-cyclotriphosphazatrienes (R = Cl, OCH2CF3, OPh, OMe, NHPh, NHBut) and substituent basicity constants

The syntheses and crystal structures of six new cis-ansa derivatives N3P3Ph2[O(CH2CH2O)4] R2 (R = Cl, OCH2CF3, OPh, OMe, NHPh, NHBut) are reported and the observed relationship between molecular parameters of the N3P3 ring and substituent basicity constants is discussed.

**Creator(s):** Besli, S., Coles, S. J., Hursthouse, M.B., Kilic, A., Mayer, T.A., Shaw, R. A.
Acta Crystallogr B Vol 58 Pt 6 pp. 1067 - 1073
**DOI:** 10.1107/S0108768102018608
**Download from:** http://scripts.iucr.org/cgi-bin/getarticleid?issn=0108-7681&volume=58&fpage=1067&details=yes
**Related dataset:** http://ecrystals.chem.soton.ac.uk/archive/00000062/

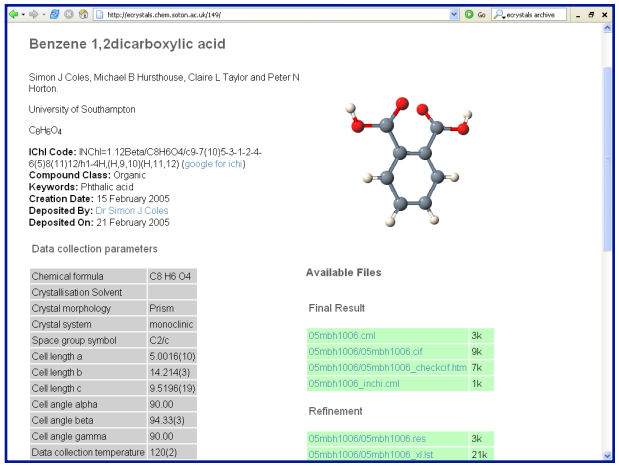
5alpha-Cholestane

The title compound, C27H48, is a steroid derivative composed of a saturated-carbon fused-ring framework with two methyl substituents and an alkyl side chain.

**Creator(s):** Coles, S. J., Hursthouse, M. B., Frampton, C. S.
Acta Crystallogr E Struct Rep OnlineVol 58 Issue Pt 4 pp. o445 - o446
**DOI:** 10.1107/S1600536802004786
**Download from:** http://scripts.iucr.org/cgi-bin/getarticleid_yard?issn=1600-5368&volume=58&fpage=o445&details=yes
**Related dataset:** http://ecrystals.chem.soton.ac.uk/archive/00000051/

Ethyl (2S*)-2-[(2R*,2'R*,5S*)-2',5-dimethyl-5'-oxoperhydro-[2,2']bifuranyl-5-yl]-2-hydroxyethanoate

The framework of K2Zn(H2P2O7)2.2H2O contains acid diphosphate-metallate layers linked by KO interactions and weak hydrogen bonds. Zn2+ cations are coordinated octahedrally by O atoms from two bidentate [H2P2O7]2- anions and two water molecules.



Benzene 1,2dicarboxylic acid

Simon J.Coles, Michael B Hursthouse, Claire L Taylor and Peter N Horton.

University of Southampton

$C_8H_6O_4$

**IChI Code:** INChI=1.12Beta/C8H6O4/c9-7(10)5-3-1-2-4-6(5)8(11)12/h1-4H,(H,9,10)(H,11,12) (google for ichi)
**Compound Class:** Organic
**Keywords:** Phthalic acid
**Creation Date:** 15 February 2005
**Deposited By:** Dr Simon J Coles
**Deposited On:** 21 February 2005

Data collection parameters

| | |
|---|---|
| Chemical formula | C8 H6 O4 |
| Crystallisation Solvent | |
| Crystal morphology | Prism |
| Crystal system | monoclinic |
| Space group symbol | C2/c |
| Cell length a | 5.0016(10) |
| Cell length b | 14.214(3) |
| Cell length c | 9.5196(19) |
| Cell angle alpha | 90.00 |
| Cell angle beta | 94.33(3) |
| Cell angle gamma | 90.00 |
| Data collection temperature | 120(2) |

Available Files

Final Result

| | |
|---|---|
| 05mbh1006.cml | 3k |
| 05mbh1006/05mbh1006.cif | 9k |
| 05mbh1006/05mbh1006_checkcif.htm | 7k |
| 05mbh1006_inchi.cml | 1k |

Refinement

| | |
|---|---|
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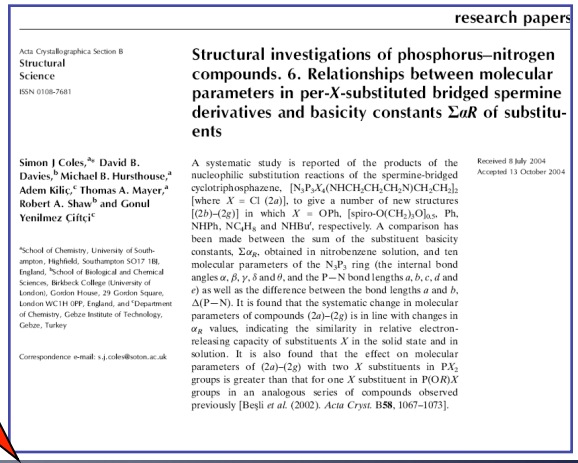
| 05mbh1006/05mbh1006_xl.lst | 21k |



research papers

Acta Crystallographica Section B
Structural Science
ISSN 0108-7681

**Structural investigations of phosphorus–nitrogen compounds. 6. Relationships between molecular parameters in per-*X*-substituted bridged spermine derivatives and basicity constants $\Sigma aR$ of substituents**

Simon J Coles,[2]* David B. Davies,[b] Michael B. Hursthouse,[a] Adem Kilic,[c] Thomas A. Mayer,[a] Robert A. Shaw[b] and Gonul Yenilmez Ciftci[c]

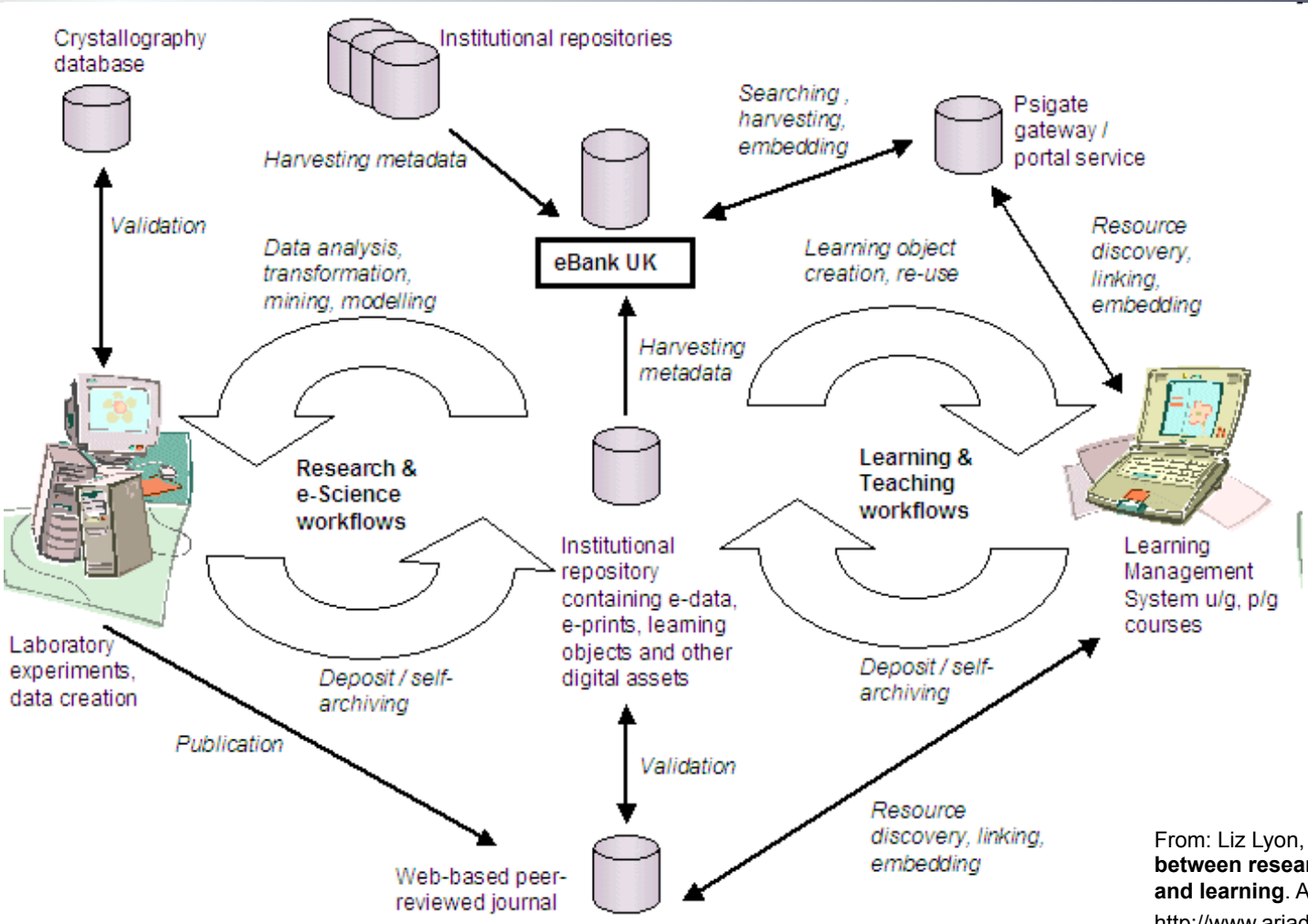
[a]School of Chemistry, University of Southampton, Highfield, Southampton SO17 1BJ, England, [b]School of Biological and Chemical Sciences, Birkbeck College (University of London), Gordon House, 29 Gordon Square, London WC1H 0PP, England, and [c]Department of Chemistry, Gebze Institute of Technology, Gebze, Turkey

Correspondence e-mail: s.j.coles@soton.ac.uk

Received 8 July 2004
Accepted 13 October 2004

A systematic study is reported of the products of the nucleophilic substitution reactions of the spermine-bridged cyclotriphosphazene, $[N_3P_3X_4(NHCH_2CH_2N(CH_2CH_2)_2]$ [where $X$ = Cl (2*a*)], to give a number of new structures [(2*b*)–(2*g*)] in which $X$ = OPh, [spiro-$O(CH_2)_2O]_{0.5}$, Ph, NHPh, $NC_4H_8$ and NHBu$^t$, respectively. A comparison has been made between the sum of the substituent basicity constants, $\Sigma\alpha_R$, obtained in nitrobenzene solution, and ten molecular parameters of the $N_3P_3$ ring (the internal bond angles $\alpha$, $\beta$, $\gamma$, $\delta$ and $\theta$, and the P—N bond lengths *a*, *b*, *c*, *d* and *e*) as well as the difference between the bond lengths *a* and *b*, $\Delta$(P—N). It is found that the systematic change in molecular parameters of compounds (2*a*)–(2*g*) is in line with changes in $\alpha_R$ values, indicating the similarity in relative electron-releasing capacity of substituents $X$ in the solid state and in solution. It is also found that the effect on molecular parameters of (2*a*)–(2*g*) with two $X$ substituents in $PX_2$ groups is greater than that for one $X$ substituent in P(O*R*)$X$ groups in an analogous series of compounds observed previously [Beşli *et al.* (2002). *Acta Cryst.* B**58**, 1067–1073].

# The Scholarly Knowledge Lifecycle




Both research and learning are cyclical processes

- Research outputs feed into and contribute to knowledge
- Research outputs are based on continuous use and reuse of data i.e. derivative in nature

From: Liz Lyon, **eBank UK: Building the links between research data, scholarly communication and learning**. ARIADNE, July 2003
http://www.ariadne.ac.uk/issue36/lyon/intro.html

# Resource Discovery & Reuse

- Simple Dublin Core
  - Crystal structure
  - Title (Systematic IUPAC Name)
  - Authors
  - Affiliation
  - Creation Date
- Qualified Dublin Core (for additional chemical metadata)
  - Empirical formula
  - International Chemical Identifier (InChI)
  - Compound Class and Keywords
- Application Profile: http://www.ukoln.ac.uk/projects/ebank-uk/schemas/
- DOI links: http://dx.doi.org/10.1594/ecrystals.chem.soton.ac.uk/145
- Rights & Citation: http://ecrystals.chem.soton.ac.uk/rights.html

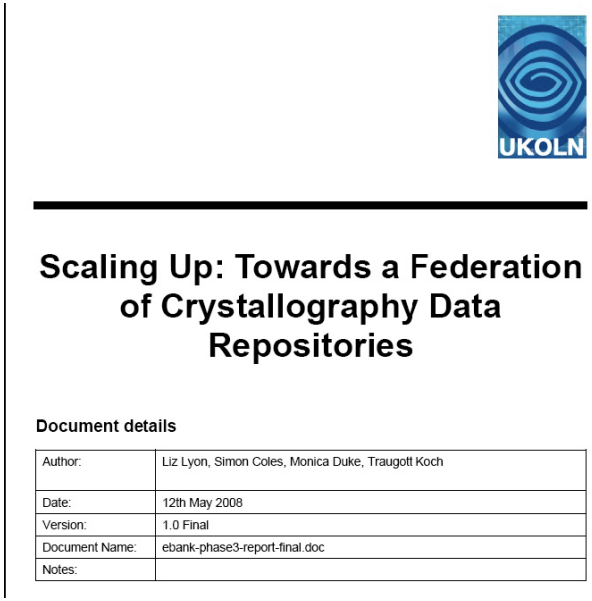
# Scaling Up: Towards a Federation

Interviews, analysis & synthesis:

IR Policy & Practice, Laboratory Practice & Workflows, Technical Interoperability & Standards, Metadata Schema & Application Profiles, Semantic Interoperability, Data Citation, Identifiers & Linking, Federation Architectures & Third Party Services, Rights & Licensing, Data Quality & Validation, Preservation, Curation & Sustainability

Selected Issues (& Recommendations):

- Diverse laboratory practice
- Instrument manufacturers have proprietary formats
- Data policy needs to reflect laboratory practice
- Data quality criteria and validation (access to raw data)
- Repository must provide control over timing of public visibility-"prior publication" problem
- No disciplinary preservation model



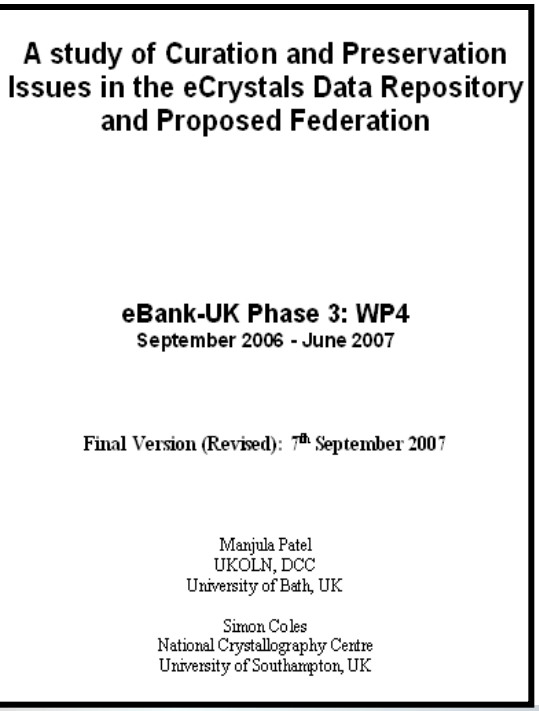
UKOLN

**Scaling Up: Towards a Federation of Crystallography Data Repositories**

**Document details**

| | |
|---|---|
| Author: | Liz Lyon, Simon Coles, Monica Duke, Traugott Koch |
| Date: | 12th May 2008 |
| Version: | 1.0 Final |
| Document Name: | ebank-phase3-report-final.doc |
| Notes: | |

# Data Curation & Preservation


A study of Curation and Preservation Issues in the eCrystals Data Repository and Proposed Federation

eBank-UK Phase 3: WP4
September 2006 - June 2007

Final Version (Revised): 7th September 2007

Manjula Patel
UKOLN, DCC
University of Bath, UK

Simon Coles
National Crystallography Centre
University of Southampton, UK

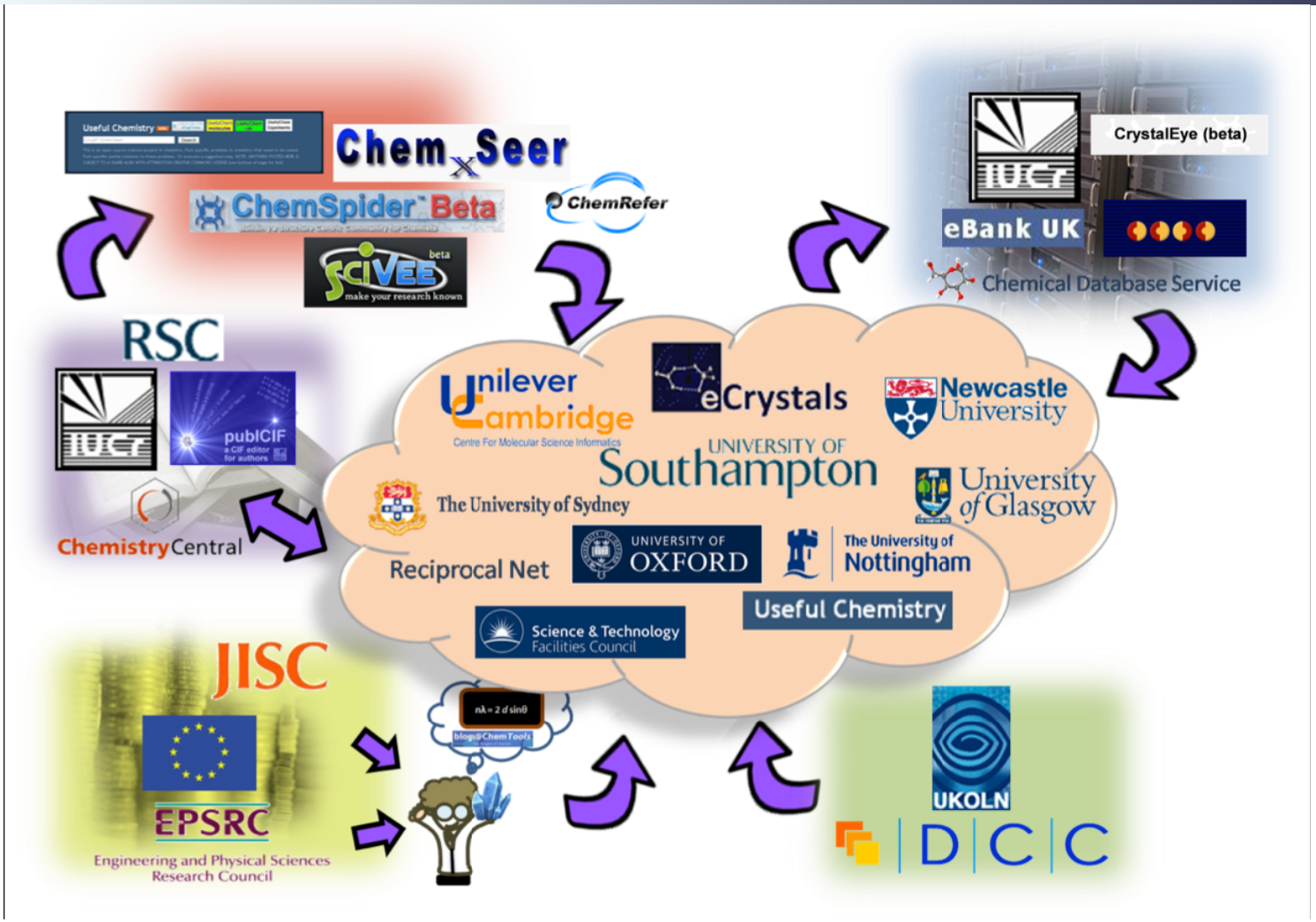
eBank-UK Phase 3: "A Study of Curation and Preservation issues in the eCrystals Data Repository and proposed Federation", Sept. 2007

- Development of preservation strategies and policies
- Audit and certification issues (TRAC, DRAMBORA, NESTOR, ISO International repository audit and certification BOF Group)
- OAIS and Representation Information for crystallography data
- eBank-UK Application Profile and preservation metadata
- e-Prints.org repository platform

# Data Curation & Preservation: Recommendations

- Develop a preservation and curation strategy and formal policies to indicate levels of service (e.g. deposit, ingest, validation, dissemination)
- Promote community-supported sustainability plan
- Self-assessment using DRAMBORA toolkit
  - Implement regular audits e.g. annually
  - Produce documentary evidence of compliance
- Maintenance and open access of critical file formats and software
  - Crystallography Information File (CIF)
  - Work-up software e.g. XPREP; SHELX{S,L}; ENCIFER; checkCIF, BABEL
  - Advocate export of raw data from instrumentation as IMG CIF
- Capture relevant Representation Information
- Capture preservation metadata (e.g. versioning; provenance)
  - OAIS Preservation Description Information
  - PREMIS Data Dictionary
  - Extend or augment eBank Metadata Application Profile
- Obtain consensus on Metadata Application Profile
- Seek to automate metadata generation, extraction and maintenance

# Building a Federation of Repositories



# eCrystals Federation Project

- eCrystals Federation Project, Nov 2007 – Mar 2009
- Builds on eBank-UK Phase 3 results
- Led by the UK National Crystallography Service (University of Southampton) with core partners at UKOLN (University of Bath), the Digital Curation Centre and the Unilever Centre (University of Cambridge) – currently 14 supporting partners.
- Integrate and embed open data repository approach into current research practice by engaging data centres, librarians, researchers, publishers and third party information providers
- Harmonise Federation metadata application profile
- Investigate aggregation issues arising from harvesting metadata from Federation repositories
- Enable the Federation of institutional repositories to interoperate with international subject archives (IUCr and CCDC) and other third party harvesters
- Develop approaches to preservation and curation of scientific data in open repositories

# Federation Interoperability

- Roll-out in 2 phases led by University of Southampton
  - Universities Sydney, Drexel, Birmingham, Newcastle with eprints.org platform
  - University Cambridge, STFC, ReciprocalNet, ARCHER with other platforms
  - Establish Federation policies, metadata application profile etc.
- Bi-directional links with derived articles in “publisher repositories”, IUCr, RSC, Chemistry Central
- StORe middleware -linking “source” and “output” repositories
- CLADDIER –linking data to publications
- OAI-ORE (Open Archives Initiative – Object Reuse and Exchange)
  - Enable distributed repositories to fully describe and exchange content
  - MicroSoft eChemistry Project

# Some challenges

- Data management plans
- Dealing with diverse laboratory practice and workflows
- Appraisal and selection
- Data provenance, audit, tracking
- Citations and versions –persistent identifiers
- Granularity of citations: dataset or values within a dataset
- Instrumentation –proprietary formats
- Access to raw data files for mining and quality control purposes
- Preservation beyond “data” e.g. workflows, blogs, discourse
- Linking across disciplines and sectors
- Collaborative social networks; also “citizen science”
- Semantic integration –controlled vocabularies, ontology etc.

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# Thanks …

**…for your attention**

**…to**

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## Questions?

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