Alfred Bader fonds Correspondence Scotland 1993-96

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Dr. Alfred Bader
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A Chemist Helping Chemists

April 2, 1996

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

The enclosed letter from Professor Tony Deeming will be self-explanatory.

You will recall that I have agreed to speak at the National Gallery of Scotland on Friday, November 29th and much look forward to that.

I had feared that I would then have to rush back to London simply because I thought that the major sales of Old Master paintings would be in London on Tuesday, December 3rd. However, I have just learned that I was mistaken and that the major sales will not be in London until the week of December 9th.

Thus, if you would not mind, we could stay in Edinburgh a little while longer and if Glasgow might enjoy hearing us also, we could be there on Monday and Tuesday, December 2nd and 3rd.

There is a Bader Award in Chemistry at Glasgow, so the academics there know me fairly well. And of course we would like to start a scheme like that at University College at Glasgow also. Could you perhaps inquire whether my visit would be welcome?

With all good wishes, I remain,

Yours sincerely,

AB/cw
Enclosure

UCL

University College London
Chemistry Department
Christopher Ingold Laboratories
20 Gordon Street
London WC1H 0AJ

Tel: 0171-387 7050
Fax: 0171-380 7463

Dr Alfred Bader
2961 North Shepard Avenue
Milwaukee
Wisconsin 53211
USA

19 March 1996

Dear Alfred,

Thank you very much for the cheque for $15,000 for the bursary scheme for 1996/97 which I have passed on. Sally and I are very happy to explain what we are doing to people in the Universities of Edinburgh, Glasgow and Birmingham. We will produce a few pages to decribe the scheme including the summer school and the costs involved. We look forward to your visit in May or July. The summer school will be on the 4th and 5th of July and of course we will be very busy on those two days but please feel free to come whenever it suits you best.

Yours sincerely,

Tony

Professor A J Deeming

Dr. Alfred Bader
924 East Juneau, Suite 622
Milwaukee, Wisconsin 53202
Phone: 414/277-0730
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A Chemist Helping Chemists

September 12, 1996

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

I am so glad to know from your letter of August 21st that you liked my book.

May I bring some copies to Edinburgh for a book-signing and if so, how many do you think reasonable? The retail price is £14.99, but the publisher requires only £7.50, so that whoever sells these could use the difference to help defray the expenses of our trip.

After my expulsion, hundreds of chemists around the world wrote to me, and many speculated why Cori kicked me out. I think that an old friend in Vienna, Dr. Paul Löw-Beer, came the closest in his analysis, copy enclosed.

Cori is so careful in taking legal advice for everything he does that I don't think for a moment that he will end on the gallows. But a man who boasts that he doesn't have a single friend, that everyone can be bought and sold, and that includes his wife and children, cannot be a happy person. And today I am far happier doing what I am doing than I was before my expulsion.

Edinburgh is one of our favourite cities, and Isabel and I so look forward to being with you.

With all good wishes for the new year, I remain,

Yours sincerely,

AB/cw
Enclosure

21. VIII. 96

Dear Alfred,

I was most pleased to receive the copy of your 'adventures'. It arrived just before going on holiday - most timely - and made fascinating reading as did your article for Chemistry in Britain. Hopefully, Couper (and Loschmidt) will be given credit for their ideas in future textbooks now that you have raised their profile.

The chapter on your expulsion from the Aldrich Board of Directors makes sad reading. What on earth was driving Tom Cori? Perhaps, to paraphrase a quotation in your book 'er wird an einem Galgen enden'!

Looking forward to your visit to Edinburgh. Meanwhile,

All best wishes to you and Isabel

Ian Gosney

FAX FROM

DR. ALFRED BADER
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March 13, 1996

Page 1 of 4

TO: Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
FAX: 131-650-4743

Dear Ian:

Thank you for your fax of today.

Isabel and I plan to be in Britain from about the 10th of November until the end of the year.

On November 20th and 25th, we must be in Sussex. Thus, one convenient time would be from Tuesday, November 26th till the end of that week. On Monday, December 2nd, I have to be in London.

Enclosed are my CV and the menu of my talks.

Of these, the obvious talk for the Chemistry Department on Wednesday the 27th at 11:00 am would be Talk #3. In this, I use a good deal of the material on Couper, which will be of special interest to a Scottish audience.

If you could stand a second talk in chemistry, perhaps combined with students from your business school, consider Talk #1. For the National Gallery, Talk #7 would, I believe, be the best.

I don't know whether the Chemistry Department in Glasgow might also like to hear Talk #3, and perhaps you could inquire. That might be on Friday, November 29th.

We will also be in England at the end of May and again in July. In May, I plan to telephone you to discuss a totally different matter.

What we think of as a pilot scheme of bursaries in Chemistry has now been running, I believe successfully, at University College for three years. We would like to use very much the same scheme at other universities and are, of course, thinking of Edinburgh and Glasgow.

The two people responsible for the scheme at University College are Professor Tony Deeming and Dr. Sally Price, and you might like to talk to them when next you are in London to get their impressions of the scheme.

With all good wishes, I remain,

Yours sincerely,

AB/cw

Enclosures

Dr. Alfred Bader
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A Chemist Helping Chemists

August 27, 1996

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

As you know, Isabel and I much look forward to being with you from November 27th on. But we have not finalized the details of our trip because we were wondering whether either or both of the Chemistry Departments in Glasgow might want to hear us also.

I have not written to anyone there because I don't really know who the persons are who will make the decisions. Could you please inquire for us? We could, for instance, come to Glasgow from Edinburgh on Sunday the 29th and return to London on the 3rd or even the 4th of December.

I hope that you don't mind that I impose on you in this way.

I wonder whether you have had a chance to talk to University College about our bursary efforts there. Of course, I plan to take the details with me to Edinburgh, and if your university was interested in trying this also, we could finalize during our visit. Glasgow also might be interested, but I have really no idea with whom to discuss this.

Dr. Douglas Lloyd at the Department of Chemistry at the University of St. Andrews would very much like to hear my talk on Couper and Loschmidt and will ask you for the exact date of that talk. Are you also able to send us the schedule of our talks in Edinburgh?

With all good wishes, I remain,

Yours sincerely,

AB/cw

Department of Chemistry
West Mains Road, Edinburgh EH9 3JJ

FAX #

Direct Dial Tel

Sent to the Jewish community details of your generous offer.
You should be hearing from Glasgow shortly; they are slow off the blocks!

To Dr. Ian Gosney

ALFRED BADER TALKS

1. History of the Aldrich Chemical Co. (A) *[overhead projector/screen]*
2. Josef Loschmidt, The Father of Molecular Modelling (A) *[overhead projector/screen]*
3. Richard Anschütz, Archibald Scott Couper and Josef Loschmidt: A Detective at Work (A) *[overhead projector/screen]*
4. The Bible through Dutch Eyes (Rembrandt and the Jews) (B) *[2 slide projectors/screens]* NB 2
5. The Adventures of a Chemist Collector (C) *[2 slide projectors/screens]*
6. Jan Lievens: Out of the Shadow (D) *[2 slide projectors/screens]*
7. The Rembrandt Research Project and the Collector (D) *[1 slide projector/screen]*
8. Chemophobia: Fear for the Future (A) *[overhead projector/screen]*

A. For chemists only

B. For art historians, theologians, Bible students

C. Mainly on art, art conservation, some chemistry

D. For art historians

March 28, 1996

Dear Ian: No word from Glasgow or Mr. Simpson — not to worry: 3 talks in Edinburgh (Nos. 1, 3 & 7) will be fine.

Best wishes
Alfred

9 X 96

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

April 8, 1993

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

Time flies, and our visit to Edinburgh is just a little more than a month away.

I have been invited to give just two lectures in the Chemistry Department at the University of Durham on Monday, May 24th. Late that afternoon, we plan to take a train from Durham to Edinburgh, and I would like to ask you to make a hotel reservation for us at a small hotel near the university for Monday and Tuesday nights, May 24th and 25th. Sometime in the afternoon of Wednesday the 26th, we plan to take a train to Glasgow.

If it fits into your schedule, I would love to be able to give at least three talks in your department. One of these should certainly be on Josef Loschmidt which will be so fitting both because the first Loschmidt Award was given to one of your colleagues and because Crum Brown worked in your department. The other two talks could be "The Adventures of a Chemist Collector" and "The Bible through Dutch Eyes." The latter could be subtitled "Rembrandt and the Jews" and might be of particular interest to members of the Jewish community. Abstracts of the first two talks are enclosed.

A further talk might be on the history of Aldrich and Sigma-Aldrich, and that might be of particular interest also to students in your business school. However, as you will be able to imagine, it isn't the easiest talk for me to give.

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

For the other three talks I will need two projectors and either one large white wall or two screens, to show two slides simultaneously.

I much look forward to being with you.

Best regards.

Sincerely,

Enclosures

H 10

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Edinburgh EH 14 4AS

031 449 5111 Mr. Mike Seed.

E4

University of Edinburgh
Dept of Chemistry
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West Mains Road
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031 667 1081 (Miss Dixon X 3425)

Sean Wilson

NB.
There are two campuses with
two different zip codes.
1 Kings Bldg & the central one.

[*Information compiled as at 28/4/83*]

UNIVERSITY★

ly established (1821) as Edinburgh School of Arts
nics Institute

[illegible] called HERIOT-WATT University

urgh, Scotland EH1 1HX
031-225 8432

F MONIFIETH, KT, Hon. LLD *Dund.*, Hon. DSc *Aston*

MH

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BSc PhD *Lond.*, FRSChem
CB, MA *Edin.*, BA *Camb.*, Hon. LLD *Strath.*, Hon.

Spelling of [illegible] tape !!!

IE UNIVERSITY—D. I. CAMERON, JP, BL *Glas.*, CA
, BL *Glas.*

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Sr. Lectr.
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Lectr.

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Camb.,
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(Biochem.) *Prof.
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Male, S. P., MSc	Lectr.
Sneddon, J.	Instr.

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Massie, W. H. S., BSc PhD *Glas.*, FRSChem	Sr. Lectr.

ties † Jt. dept. of univ. and Edinburgh Coll. of Art

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ican Studies, Centre of, see below

riculture, see also 'Associated Institution', elow

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onds, N. W., BSc, FRSEd, FIBiol	Hon. Prof.
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rison, R. R., BSc	†Lectr.
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mson, J. R., BSc	†Lectr.
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ey, J. A., BSc MA	Lectr.
, S. E., MSc	†Lectr.
ns, J. K. S., MSc	†Lectr.
h, M. L., BSc PhD	Lectr.
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McKinley, R. G., BSc PhD	†Lectr.
Copland, T., BSc	†Lectr.

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Stockwell, R. A., BSc MB BS PhD *Lond.*	Prof.
Kerr, A. W. S., MB ChB	Sr. Lectr.
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Shaw, J. P., BDS PhD	Lectr.
Trotter, Christina M., BSc *St.And.*, PhD	Lectr.
Thomson, D. A. R., BDS, FDSRCSEd	Lectr.

Archæology

Harding, D. W., MA DPhil *Oxf.*	*Abercromby Prof.
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Bonsall, J. C., BA *Sheff.*	Lectr.
Mercer, R. J., MA, FSA	Lectr.
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Archæology, Classical

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Byrom, J. B., BArch PhD	Sr. Lectr.
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Gilmour, A.	Sr. Lectr.
Higgs, M. S., PhD	Sr. Lectr.
Appleton, I., PhD	Sr. Lectr.
Ruddock, E. C., BA MSc *Lond.*	*Sr. Lectr.
Birkhans, M., MArch *Tor.*	Sr. Lectr.
Russell, T. M., BArch PhD *Sheff.*	Sr. Lectr.
Duriez, M. J.	Lectr.
Filor, S. W.	Lectr.
Macdonald, A. J., BSc PhD	Lectr.
Raman, P. G., BArch *Madr.*, PhD	Lectr.
Talbot, R. D., BSc *Sheff.*	Lectr.
Alberli, P. D., MA	†Lectr.

Art (Fine Art)

Hillenbrand, R., MA *Camb.*, DPhil *Oxf.*	Reader
Macmillan, J. D., MA *St.And.*, PhD	Sr. Lectr.
Bury, K. M., MA *Camb. & Lond.*	Lectr.
Campbell, Mrs. Jean P., MA PhD	Lectr.
Cowling, Mrs. Elizabeth G., BA *Oxf.*, MA *Lond.*	Lectr.
Hammer, M. A., BA *Lond.*	Lectr.
Higgitt, J. C., MA *Oxf. & Lond.*	Lectr.
Howarth, D. J., MA PhD *Camb.*	Lectr.
Tarr, R. P., MA *Oxf.*, DipHistArt *Lond.*	Lectr.
Tolley, T., BA *E.Anglia*	Lectr.

Astronomy

Longair, M. S., BSc *St.And.*, MA PhD *Camb.*, FRSEd	*Regius Prof.
Smyth, M. J., MA PhD *Camb.*, FRSEd	Sr. Lectr.
Brück, Mrs. Mary T., MSc *Dub.*, PhD	Sr. Lectr.
Brand, P. W. J. L., BSc PhD	Lectr.
Emerson, D., MA DPhil *Oxf.*	Lectr.
Cooke, J. A., MA *Camb.*, PhD	Lectr.

Biochemistry

......	Prof.
Bowman, I. B. R., BSc PhD *Aberd.*	Sr. Lectr.
Ramsay, W. N. M., BSc PhD	*Sr. Lectr.
Ottaway, J. H., DSc PhD *Lond.*	Sr. Lectr.
Jocelyn, P. C., MA DPhil *Oxf.*	Sr. Lectr.
Cruft, H. J., PhD	Sr. Lectr.
Ryle, A. P., BA PhD *Camb.*	Sr. Lectr.
Atkins, G. L., MA *Oxf.*, PhD *Lond.*	Sr. Lectr.
Leaver, J. L., BSc PhD	Lectr.
Stafford, Mrs. Winifred L., BSc PhD	Lectr.
Apps, D. K., MA PhD *Camb.*	Lectr.
Doyle, J., BSc *St.And.*, PhD	Lectr.
Flynn, I. W., BSc PhD	Lectr.
Nimmo, I. A., MA *Camb.*, PhD	Lectr.
O'Brien, J. A., MA *Oxf.*, PhD	Lectr.
Phillips, J. H., MA PhD *Camb.*	Lectr.
Suckling, K. E., BSc *Liv.*, PhD *Camb.*	Lectr.
van Heyningen, S., MA PhD *Camb.*	Lectr.
Whittle, Elizabeth D., MA PhD *Camb.*	(Med. Information) Lectr.
Haywood, J., BSc PhD *Open*	Lectr.
Pettigrew, G. W., BSc PhD	Lectr.

Biology, see Bot., and Zool.

Botany, see also Agric., and Forestry and Nat. Resources

Yeoman, M. M., MSc *Durh.*, PhD, FRSEd	*Regius Prof.
Davis, P. H., PhD DSc, FRSEd	(Plant Taxonomy) Prof.
Dale, J. E., BSc PhD *Lond.*, FRSEd	Reader
Leaver, C. J., BSc PhD	Reader
Dyer, A. F., DPhil *Oxf.*, BSc *Durh.*	Sr. Lectr.
Lyndon, R. F., BSc PhD *Durh.*	Sr. Lectr.
Smith, P. M., BSc PhD *Birm.*	Sr. Lectr.
Dixon, R. O. D., MSc PhD *Lond.*	Sr. Lectr.
Blyth, W., BSc PhD	Lectr.
Tulett, A. J., BSc *Lond.*	Lectr.
Kemp, R. F. O., MA DPhil *Oxf.*	Lectr.
Trewavas, A. J., BSc PhD *Lond.*	Lectr.
Mann, D. G., BSc PhD *Brist.*	Lectr.
Henderson, D. M., BSc, FRSEd, FLS	Hon. Prof.

Business Studies, see also Acctg. and Bus. Method

Hunt, N. C., CBE, BCom *Lond.*, PhD	*Prof.
Coke, S., MA *Oxf.*	(Internat. B.) Morrison Prof.
Williams, H. P., MA *Camb.*, PhD *Leic.*	(Management Sci.) Prof.
Reekie, W. D., PhD *Strath.*, BCom	Reader
McKenzie, R. M., MC, MA	Sr. Lectr.
Sams, K. I., BSc(Econ) *Lond.*	Sr. Lectr.
Closs, S. J., MA PhD	Lectr.
Droucopoulos, V., BA *Athens*, MA *N.Y.State*, PhD	Lectr.
Glen, J. J., BSc *Glas.*, MSc *Strath.*	Lectr.
Hirst, I. R. C., BA *Oxf.*, MBA *C'nell.*, PhD *Chic.*	Lectr.
Henley, J. S., BSc PhD *Lond.*	Lectr.
Ingleton, C. C. P., BSc *Brad*	Lectr.
Weber, M. H., DBA	Lectr.
White, P. J., BA MSc *Strath.*	Lectr.
Rees, Mrs. Jennifer L., BSc	Lectr.
Allen, D. E., MA *St.And.*, MPhil *Leic.*	Lectr.
Crook, J. N., BA *Lanc.*, MSc *Wales*	Lectr.
Gowrie, E. S., BA *Nott.*, MSc	Lectr
Kwiatkowski, J. W., MA *Camb.*, MSc *Birm.*, PhD *Calif.*	Lectr
Reid, D. M., BSc *Salf.*, MSc *Manc.*	Lectr.
Behrend, Hilde, BSc(Econ) *Lond.*, PhD	Prof. Emer

Celtic

Gillies, W., BA *Oxf.*, MA	*Prof.
Meek, D. E. M., MA *Glas.*, BA *Camb.*, FRHistS	Lectr
Black, R. I., MA *Glas.*	Lectr.

Chemical Engineering, see under Engin., Sch. of

K
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Macka
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Baxter
Chin
Chinne
Scott, J
Dolby,
Dow, F
Civil
Class
Comp
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Artific
Howe, J.
Plotkin,
Bundy, A
Popplest
Thomps
Compu
Michaels
FRS
Burstall,
Milner, A
Schofield,
Atkinson,
Candlin,

U of Edinburgh

Chemistry

Kemball, C., MA ScD *Camb.*, FRSChem, FRS, FRSEd — Prof.
Ebsworth, E. A. V., MA PhD ScD *Camb.*, FRSChem, FRSEd — *Crum Brown Prof.
Knox, J. H., PhD *Camb.*, DSc, FRSChem, FRSEd — (Phys. C.) Prof.
Donovan, R. J., BSc *Wales*, PhD *Camb.*, FRSChem, FRSEd — (Phys. C.) Prof.
...... — (Organic C.) Forbes Prof.
Leaver, D., BSc PhD *Leeds*, FRSEd — Reader
Tennant, G., BSc PhD *Glas.*, FRSChem, FRSEd — Reader
Anderson, D. M. W., PhD DSc, FRSChem, FRSEd — Reader
Rankin, D. W. H., MA PhD *Camb.* — Reader
Kidd, D., BSc PhD — Sr. Lectr.
Schwarz, J. C. P., MA BSc PhD *Dub.* — Sr. Lectr.
Fluendy, M. A. D., MA DPhil *Oxf.*, DSc, FRSChem, FRSEd — Sr. Lectr.
Leach, H. F., ARCS, BSc PhD *Exe.*, FRSChem — Sr. Lectr.
Sharp, J. T., BSc PhD *Lond.*, FRSChem — Sr. Lectr.
Lowe, B. M., BSc PhD *Lond.*, FRSChem — Sr. Lectr.
Bellamy, A. J., BSc PhD *Birm.* — Sr. Lectr.
Stephenson, T. A., DIC, DSc PhD, FRSChem — Sr. Lectr.
Lawley, K. P., MA DPhil *Oxf.* — Lectr.
Palmer, M. H., DSc PhD *Lond.* — Lectr.
Gould, R. O., BA *Mass.*, PhD *St.And.* — Lectr.
Sadler, I. H., BSc PhD *Lond.* — Lectr.
Whan, D. A., BSc PhD *Belf.*, DSc, FRSChem — Lectr.
Cradock, S., MA PhD *Camb.* — Lectr.
Gosney, I., BSc PhD *A.N.U.* — Lectr.
Paton, R. M., BSc PhD — Lectr.
Rowley, A. G., BSc *Sus.*, DPhil *York(U.K.)* — Lectr.
McNab, H., BSc PhD *St.And.* — Lectr.
Simpson, T. J., PhD *Brist.*, BSc — Lectr.
Heath, G. A., BSc PhD *Melb.* — Lectr.
Mackaill, A. W., BSc *H.-W.*, MEd PhD, FRSChem — Hon. Lectr.
Welch, A. J., BSc PhD *Lond.* — Lectr.
Baxter, R. L., BSc PhD — Lectr.

Chinese

Chinnery, J. D., BA PhD *Lond.* — *Sr. Lectr.
Scott, J. H. J., MA — Lectr.
Dolby, A. W. E., MA PhD *Camb.* — Lectr.
Dow, F. D. M., BA *Chinese H.K.* — Lectr.

Civil Engineering, see under Engin., Sch. of

Classics, see Greek, and Latin

Computer Science and Artificial Intelligence, School of

Artificial Intelligence

Howe, J. A. M., MA *St.And.*, PhD *Camb.* — *Reader
Plotkin, G. D., BSc *Glas.*, PhD — Reader
Bundy, A. R., BSc PhD *Leic.* — Lectr.
Popplestone, R. J., BSc — Lectr.
Thompson, H. S., MA MS PhD *Calif.* — Lectr.

Computer Science

Michaelson, S., ARCS, BSc *Lond.*, FIMA, FRSEd — Prof.
Burstall, R. M., BA *Camb.*, MSc PhD *Birm.* — Prof.
Milner, A. J. R. G., BA *Camb.* — Reader
Schofield, P. D. A., ARCS, BSc *Lond.* — *Sr. Lectr.
Atkinson, M. P., MA PhD *Camb.* — Sr. Lectr.
Candlin, Mrs. Eileen R. S., MA PhD *Camb.* — Lectr.
Rees, D. J., ARCS, BSc *Lond.*, PhD — Lectr.
Stacey, F., BSc *Brist.* — Lectr.
Tansley, J., BSc *Lond.* — Lectr.
Wight, A. S., MA PhD — Lectr.
Smith, L. D., MA *Camb.* — Lectr.
Dewar, H. M., BA BPhil *Oxf.* — Lectr.
Jerrum, M. R., MA *Camb.*, PhD — Lectr.
Buchanan, Miss I., BSc PhD — †Lectr.
Gray, J. P., BSc PhD — †Lectr.
Hansen, I. B., MSc PhD — Lectr.
Hennessy, M. C., BSc MA PhD — Lectr.

Criminology

McClintock, F. H., JP, BSc *Lond.*, MA *Camb.*, Hon. LLD *Uppsala* — *Prof.
Young, P. J., BSc *Lond* MA *Sheff.* — Lectr.
Garland, D. W., MA *Sheff.*, LLB — Lectr.

Dentistry, see below

Divinity, see also 'Constituent College', below

McIntyre, Very Rev. J., MA BD DLitt Hon. DD *Glas.*, FRSEd — (Div.) Prof.
Cheyne, Rev. A. C., MA BD BLitt *Oxf.* — (Eccles. Hist.) Prof.
Forrester, Rev. D. B., MA *St.And.*, DPhil *Sus.*, BD — (Christian Ethics) Prof.
Anderson, Rev. H., MA BD PhD *Glas.*, Hon. DD *Glas. & Edin.* — (N.T. Lang., Lit. & Theol.) Prof.
Mackey, Rev. J. P., BA *N.U.I.*, LPh DD STL *Maynooth*, PhD *Belf* — (Systematic Theol.) Prof.
Gibson, Rev. J. C. L., MA BD *Glas.*, DPhil *Oxf.* — Reader
Ross, Rev. A. C., STM *U.T.S.(N.Y.)*, MA BD PhD — Sr. Lectr.
Wright, D. F., MA *Camb.* — Sr. Lectr.
Campbell, Rev. A. V., ThD *Calif.*, MA BD — Sr. Lectr.
Auld, Rev. A. G., MA *Aberd.*, BD PhD — Lectr.
Gill, Rev. R. M., BD PhD *Lond.*, MSocSc — Lectr.
Hayman, A. P., BA PhD *Durh.* — Lectr.
Mealand, D. L., MA *Oxf.*, MLitt *Brist.* — Lectr.
Templeton, Rev. D. A., BA *Camb.*, BD PhD *Glas.* — Lectr.
Lyall, Rev. D., STM *Yale*, BSc BD PhD — †Lectr.
McGregor, Rev. T. S., MA BD — †Lectr.
O'Donoghue, Rev. N. D., MA *N.U.I.*, BD *Maynooth*, DPhil *Louvain* — Lectr.
Whaling, Rev. F., MA *Camb.*, ThD *Harv.* — Lectr.
Lewis, Rev. A. E., MA *St.And.*, ThD *Prin.*, BD — Lectr.
Page, Rev. Ruth, MA *St.And.*, BD *Otago*, DPhil *Oxf.* — Lectr.
McDonald, Rev. J. I. H., MA BD MTh *Glas.*, PhD — Lectr.
Leishman, Rev. R. M., MA — †Lectr.
Tellini, Rev. G. — Lectr.

Economics

Vandome, P., MA *Camb. & Oxf.* — (Econometrics) Prof.
Stewart, I. G., MA *St.And. & Camb.* — *Prof.
Main, B. G. M., BSc *St.And.*, MA PhD MBA *Calif.* — Reader
Wright, L. C., BA *Wales*, PhD — Sr. Lectr.
Heughan, Hazel E., MA — Lectr.
Reid, G. C., MA *Aberd.*, MSc *S'ton.*, PhD — Lectr.
Crowther, A. V., BA BPhil *Oxf.* — Lectr.
Rutherford, D., MA *Oxf.* — Lectr.
George, D. A. R., BSc *Sus.*, BPhil *Oxf.* — Lectr.
Roberts, C. J., BSc *Warw.*, MSc *Lond.*, PhD — Lectr.
Sayer, S. T., BA BPhil *Oxf.* — Lectr.
Oxley, L. T., BA *Wales*, MA *Sheff.* — Lectr.
Fransman, M., MA *Witw.*, DPhil *Sus.* — Lectr.
Clark, S. J., MSc *Lond.* — Lectr.

Education

Entwistle, N. J., BSc *Sheff.*, PhD *Aberd.*, FBPsS — *Bell Prof.
King, K. J., BA *Camb.*, DipEd *Lond.*, PhD — Reader
Alexander, D. J., MA *Keele*, MEd *Manc.* — Lectr.
Steward, T. G., BA *Manc.* — Lectr.
Pollitt, A. B., BSc MEd *Aberd.* — Lectr.
Raffe, D. J., BA BPhil *Oxf.* — Lectr.
Sharp, A., BA *Lond.*, PhD *Lanc.* — Lectr
Thomson, G. O. B., MEd *Glas.*, MA PhD — Lectr.
Jonathan, Mrs. Ruth M., BA *Liv.*, MA *Leic.*, PhD — Lectr.
Bray, T. M., BA *N'cle.(U.K.)*, MSc — Lectr.

Electrical Engineering, see under Engin., Sch. of

Engineering, School of

Chemical Engineering

...... — Prof.
Macleod, N., ARCS, BSc PhD *Lond.*, FRSEd — *Sr. Lectr.
Davidson, J. M., BSc PhD *Lond.* — Sr. Lectr.
Glass, D. H., BSc *Manc.*, PhD *Camb.* — Lectr.
Ponton, J. W., BSc PhD — Lectr.
Pritchard, C. L., MA *Camb.*, PhD *Delhi* — Lectr.

Civil Engineering

Hendry, A. W., PhD DSc *Aberd.*, FICE, FRSEd, FIStructE — *Prof.
Robertson, J. M., BSc *St.And.*, MSc *Dund.*, PhD — Reader; Dir., Microfabrication Facility
Christie, I. F., BSc *Glas.*, PhD, FICE — Sr. Lectr.
Davies, S. R., BSc *Wales*, PhD — Sr. Lectr.
Royles, R., BSc *Lond.*, PhD *Camb.* — Sr. Lectr.
Birse, R. M., BSc — Lectr.
Darling, T. Y., MA *Camb.*, FICE — †Lectr.
Fairbairn, D. R., BSc PhD — Lectr.
Forde, M. C., MSc PhD *Birm.* — Lectr.
Gardner, R. P. M., BSc, FICE — †Lectr.
McConnachie, G. L., MSc *Strath.*, BSc — Lectr.
Simpson, D., BSc — †Lectr.
Sinha, B. P., BSc *Patna*, PhD — Lectr.
Morgan, J., PhD — Lectr.
Topping, B. H. V., BSc PhD *City* — Lectr.
Hamilton, J. A. K., GM, BSc Hon. DSc, FICE, FRSEd — Hon. Sr. Lectr.

Electrical Engineering

Collins, J. H., MSc *Lond.*, FIEE, FIP, FRSEd — *Prof.
Mavor, J., BSc *City*, PhD *Lond.*, FIEE, FIP — (Microelectronics) Prof.
Owen, A. E., PhD *Sheff.*, FIP, FRSChem, FRSEd — (Phys. Electronics) Prof.
Grant, P. M., BSc *H.-W.*, PhD — Reader
Shepherd, J., BSc *Glas.*, FIEE — Sr. Lectr.
Dinnis, A. R., BSc PhD — Sr. Lectr.
Jordan, J. R., DIC, MSc *Sur.*, PhD — Sr. Lectr.
Dryburgh, P. M., BSc *St.And.*, PhD *Glas.* — Lectr.
Whittington, H. W., BSc PhD *Strath.* — Lectr.
Hannah, J. M., BSc *Strath.*, PhD — Lectr.
Jack, M. A., MSc *H.-W.*, PhD — Lectr.
Cowan, C. F. N., BSc PhD — Lectr.
Coghill, G. G., BSc *H.-W.*, BA *Open*, PhD *Kent* — Lectr.
Denyer, P. B., BSc *Lough.*, PhD — Lectr.
Kelly, R. G., BSc PhD — Lectr.
Dubbs, J. H., BSc PhD — Lectr.

Dr. Alfred R. Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

October 7, 1993

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
King's Building
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

I am so happy to have your kind letter of September 22nd.

I have had great fun studying the correspondence between Anschütz and Crum-Brown. Both were such fascinating men in pursuit of the truth, and it is just too bad that Anschütz did not find out about Loschmidt before he did his detective work on Couper.

I hope to finish my autobiography early next year, and after that plan to write a detailed essay on Anschütz, using the correspondence which you let me have.

As you know, Isabel and I visit Britain at lest twice each year, in the summer and then again in November/December. Once you have a new group of students, we will be happy to come to Edinburgh again with the proviso that you allow me to give at least three lectures.

All good wishes.

Sincerely,

22.IX.93

DEPARTMENT of CHEMISTRY
The University of Edinburgh
King's Buildings
West Mains Road
Edinburgh EH9 3JJ
Fax 031 650 4743
Telex 727442 (UNIVED G)
Email
Telephone 031 650 1000
or direct dial 031 650 4712

Dear Alfred & Isobel,

A belated thank you note for the cheque which will be spent as a stipend for a student to do some research over the vac.

No luck on the Loschmidt front. We can find no trace of him or Bauer in Crum Brown's papers so far.

You are still featuring in the Independent (see enclosure), as indeed I am, with my letter on 'handedness' to refute Jack Dunitz and Heilbronner's claim on 'looking-glass' milk.

Also enclosed is another Independent piece on the restoration of the Sistine Chapel. A very topical subject here in Europe and as professionals

you will both have opinions on the quality!

Finally Alfred, you may recollect meeting Brian Johnson on various occasions. He is of course, our new Crum Brown professor at Edinburgh and he has asked me to extend another invitation for you and Isobel to visit Edinburgh and entertain a new batch of students with one of your illuminating lectures. You will be most welcome and Brian regrets missing your last lecture.

With all best wishes
Yours sincerely

Ian

Dr. Alfred R. Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

July 30, 1993

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
King's Buildings
West Mains Road
Edinburgh EH9 3JJ, Scotland

Dear Ian:

I truly appreciated your great efforts to get such interesting books and letters to me. You must have realized my happiness when your package arrived on Friday evening, just minutes after I talked to your wife.

The material contained in the letters between Crum Brown and Anschütz has all been correctly published in the various articles on Couper, particularly in Dr. Duff's article in Chemistry in Britain. I will make certain that this material eventually goes to a library where it will be appreciated.

The more I think about Anschütz the more I appreciate his care and thoughtfulness. Without him, neither Couper nor Loschmidt would be known at all! I may well use the material to write a essay connecting Anschütz, Couper and Loschmidt.

As promised, please find enclosed my check for $1000 to your Department of Chemistry.

As I mentioned, in 1862 Bauer travelled through Europe and came to England, bringing copies of Loschmidt's little book with him. Of the two scientists who were in my mind, the likeliest recipients were Crum Brown and Faraday. I checked carefully in the Royal Institution Library, and they do not have the book. Being so small and in difficult German, it might easily have been discarded. I would be delighted to send you another check for $1000 if you could find Crum Brown's copy for me.

Again, many thanks and best wishes,

Enclosure

P.S. On the photo of ~~the~~ the Munich research group is the name - Manasse. Coincidentally,

DEPARTMENT *of* CHEMISTRY
The University of Edinburgh
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Fax 031 650 4743
Telex 727442 (UNIVED G)
Email
Telephone 031 650 1000
or direct dial 031 650 4712

21. VII. 93

Dear Alfred & Isobel,

Hopefully this find will excite you as much as it did me.

My old chum Neil Campbell, formerly Professor of Chemistry, who is 90 but still active, asked me to go through books and papers that he had left behind when he retired in 1973 and I came across this treasure trove.

I know you will provide a good home for them under the terms discussed and perhaps they will be of use for your scholarship.

All best wishes

Ian

P.S. In the photo of ~~the~~ Munich research group is the name - Manasse. Coincidentally, we have just published a paper on Manasse's 'dimer' - he got the structure wrong!

Dr. Ian Gosney and Julie Warren
24 (1F) Fettes Row
Edinburgh EH3 6RH

Dear Dr. & Mrs Bader,

Thankyou so much for the Whalley print, I love it! It was immensely kind and generous of you to think of me; you have unwittingly aided me in my campaign to get something on our walls that is more recently produced than 1820!

Sincerely,
Julie Warren.

Programme for the Visit by Dr Alfred and Isobel Bader to Edinburgh

24th May	evening	arrive at Club from Durham
25th May	11.30	visit to Talbot Rice Gallery (Mr Bill Hare) to see Dutch paintings
—	12.30	lunch at Club with Dr Patsy Campbell (Fine Art Department), Mr Bill Hare (Assistant Curator, Talbot Rice Gallery), and Julia Lloyd Williams (National Gallery)
	13.30	visit to the National Gallery with Julia Lloyd Williams to see Dutch paintings
	18.30	dinner at the Club with Professor Tom Brown and wife
	19.50 for 20.00	erev Shavuot 5753 at Synagogue Chambers, Salisbury Road, Newington (Dr Ian Liefer)
26th May	09.00	pick-up from Club by Dr Gosney
	09.30	Dr Tennant
	10.00	Professor Brown
	10.30	coffee with Professor Ramage
	11.10	LECTURE in T100
	12.30	lunch with Professor Ramage and Dr Gosney and Dr Tabor
	14.00	Back to Club train to Glasgow

TALBOT RICE GALLERY

The University of Edinburgh
Old College
South Bridge
Edinburgh EH8 9YL

Fax 031 667 7938
Telephone 031 650 1000
or direct dial 031 650 2210-3

20 MAY 1993

Dr Alfred Bader
c/o Dr Gosney
Departmnet of Chemistry
Kings Buildings
University of Edinburgh

Dear Dr Bader

I was delighted to learn from Dr Gosney that you had expressed a wish to visit the Talbot Rice Gallery during your trip to Edinburgh on 25th May.

Unfortunately I shall be out of the country on a lecture tour of Japan at that time and will therefore not be able to meet you and show you the University's collection. I have however asked Bill Hare, the Assistant Curator of the Gallery, to receive you.

I hope I may have the opportunity of meeting you at some future date.

Yours sincerely

DUNCAN MACMILLAN

Dr Duncan Macmillan Curator Bill Hare Assistant Curator

Al. Meyers to follow on Monday, 29th - quite a double act!

THE UNIVERSITY OF EDINBURGH

DEPARTMENT *of* CHEMISTRY

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Edinburgh EH9 3JJ

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Telex 727442 (UNIVED G)
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Telephone 031 650 1000
or direct dial 031 650 4712

12.v.93

Dear Alfred,

my apologies for not contacting you sooner but I have been away from the department, and in my absence your visit was not handled. However, I have now arranged your lecture to be at 11.00 am on Wednesday, May 26th. Accomodation for you and your wife has been booked as requested in our Staff Club in Chambers St, in the middle of the University. Other arrangements are also being made in connection with your lecture(s). I will be in touch again shortly. In the meantime,

FAX TRANSMITTAL SHEET

FROM: DR. ALFRED BADER

2961 North Shepard Ave.
Milwaukee, Wisconsin 53211

PHONE: (414) 962-5169

FAX: (414) 962-8322

TO: Dr. Ian Gosney
Department of Chemistry
University of Edinburgh

DATE: May 14, 1993

Dear Ian,

I so appreciate the arrangements you have made for us, and I am really excited both about being able to speak about Loschmidt and looking at 17th century Dutch paintings in two galleries.

Could you please suggest to your students that they study both the paper on Loschmidt in the February issue of Chemistry in Britain, as well as the interesting challenge and the letters to the editor in the May issue. Of course, I will be able to present a great deal of material which was not possible to include in the February article.

If, per chance, the Torrance Gallery is considering disposing of old master paintings, I would be delighted to help.

In view of all the exciting things that we are looking forward to with you, would it be possible for us to spend two nights, Monday and Tuesday nights, at the Staff Club, of course gladly at our expense.

I have two lectures to give in Durham, both Monday morning and discussions with academics in the afternoon, and we could then easily take an afternoon train from Durham to Edinburgh and thus have all of Tuesday there.

I am just leaving Milwaukee and will arrive at our English home next week Friday, May 21st. Our address and telephone number are: 52 Wickham Ave., Bexhill-on-Sea, East Sussex TN39 3ER, telephone 0424 222 223. Could you just drop me a brief note letting me know whether we may stay at the Staff Club on Monday night also.

Isabel and I much look forward to seeing you.

Best regards,

IG's fax
rec'd attached

FAX TRANSMITTAL SHEET

FROM: DR. ALFRED BADER

2961 North Shepard Ave.
Milwaukee, Wisconsin 53211

PHONE: (414) 962-5169 011 44 31 6 [illegible]

FAX: (414) 962-8322

TO: Dr. Ian Gosney
Department of Chemistry
University of Edinburgh

DATE: May 14, 1993

Dear Ian,

I so appreciate the arrangements you have made for us, and I am really excited both about being able to speak about Loschmidt and looking at 17th century Dutch paintings in two galleries.

Could you please suggest to your students that they study both the paper on Loschmidt in the February issue of Chemistry in Britain, as well as the interesting challenge and the letters to the editor in the May issue. Of course, I will be able to present a great deal of material which was not possible to include in the February article.

If, per chance, the Torrance Gallery is considering disposing of old master paintings, I would be delighted to help.

In view of all the exciting things that we are looking forward to with you, would it be possible for us to spend two nights, Monday and Tuesday nights, at the Staff Club, of course gladly at our expense.

I have two lectures to give in Durham, both Monday morning and discussions with academics in the afternoon, and we could then easily take an afternoon train from Durham to Edinburgh and thus have all of Tuesday there.

I am just leaving Milwaukee and will arrive at our English home next week Friday, May 21st. Our address and telephone number are: 52 Wickham Ave., Bexhill-on-Sea, East Sussex TN39 3ER, telephone 0424 222 223. Could you just drop me a brief note letting me know whether we may stay at the Staff Club on Monday night also.

Isabel and I much look forward to seeing you.

Best regards,

<u>FAX TRANSMITTAL SHEET</u>

ET

FROM: DR. ALFRED BADER

2961 North Shepard Ave.
Milwaukee, Wisconsin 53211

PHONE: (414) 962-5169

FAX: (414) 962-8322

TO: Dr. Ian Gosney
Department of Chemistry
University of Edinburgh

DATE: May 12, 1993
FAX 011 44 31 650 4743

Dear Ian,

As you will be able to imagine, I am so happy to have your fax of today and to know that we will be welcome in Edinburgh.

We plan to arrive in Edinburgh from Durham on Tuesday morning, May 25, and will take a cab from the station to the Staff Club on Chambers Street.

Please note the interesting article on Loschmidt and the several letters to the editor in the May issue of <u>Chemistry in Britain</u>. These should lead to an interesting discussion after my lecture at 11 a.m. on Wednesday, May 26.

Of course I would be happy to give one or two other lectures on Tuesday or Wednesday.

The lecture entitled "The Bible through Dutch Eyes" might interest an audience at the National Gallery of Scotland. I gave that talk some time ago at the National Gallery in London, and it was well received.

Thanks for all your help.

Best wishes,

Dr. Alfred R. Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

May 4, 1993

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

I do hope that my letter of April 8th, copy enclosed, did not get lost.

After writing that letter, I realized, to my chagrin, that my visit to Scotland will conflict with Shavuot. We are not orthodox, but this may well interfere with my giving the talk on "The Bible through Dutch Eyes" to the Jewish community.

We plan to leave Milwaukee on May 14, and so I would much appreciate your reply by fax whether our visit to Edinburgh will be welcome. We have already heard from Professor Connolly that our visit to Glasgow on May 27 and 28 will be.

I look forward to hearing from you.

Sincerely,

FAX: Home 414 962 8322
Office 414 277 0709

Enclosures

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

April 8, 1993

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

Dear Ian:

Time flies, and our visit to Edinburgh is just a little more than a month away.

I have been invited to give just two lectures in the Chemistry Department at the University of Durham on Monday, May 24th. Late that afternoon, we plan to take a train from Durham to Edinburgh, and I would like to ask you to make a hotel reservation for us at a small hotel near the university for Monday and Tuesday nights, May 24th and 25th. Sometime in the afternoon of Wednesday the 26th, we plan to take a train to Glasgow.

If it fits into your schedule, I would love to be able to give at least three talks in your department. One of these should certainly be on Josef Loschmidt which will be so fitting both because the first Loschmidt Award was given to one of your colleagues and because Crum Brown worked in your department. The other two talks could be "The Adventures of a Chemist Collector" and "The Bible through Dutch Eyes." The latter could be subtitled "Rembrandt and the Jews" and might be of particular interest to members of the Jewish community. Abstracts of the first two talks are enclosed.

A further talk might be on the history of Aldrich and Sigma-Aldrich, and that might be of particular interest also to students in your business school. However, as you will be able to imagine, it isn't the easiest talk for me to give.

Enclosures

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

For the other three talks I will need two projectors and either one large white wall or two screens, to show two slides simultaneously.

I much look forward to being with you.

Best regards.

Sincerely,

Enclosures

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

April 8, 1993

No reply
to this and
follow up.

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

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Sincerely,

Enclosures

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

April 8, 1993

Dr. Ian Gosney
Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland

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Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

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I much look forward to being with you.

Best regards.

Sincerely,

Enclosures

Internal Revenue Service
District Director
31 HOPKINS PLAZA
BALTIMORE, MD 21201

Department of the Treasury

Date: SEP 2 1 1987

Employer Identification Number:
52-1263286
Contact Person:
GEORGE T. SMITH
Contact Telephone Number:
(301) 962-4779

AMERICAN FRIENDS OF THE UNIVERSITY
OF EDINBURGH
214 MASSACHUSETTS AVE N E
WASHINGTON, DC 20002

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Caveat Applies:
Yes

Dear Applicant:

Based on the information you recently submitted, we have classified your organization as one that is not a private foundation within the meaning of section 509(a) of the Internal Revenue Code because you are an organization described in section 509(a)(1).and 170(b)(1)(a)(vi)

Your exempt status under section 501(c)(3) of the Code is still in effect.

This classification is based on the assumption that your operations will continue as you have stated. If your sources of support, or your purposes, character, or method of operation change, please let us know so we can consider the effect of the change on your exempt status and foundation status.

This supersedes our letter dated April 22, 1987.

Because this letter could help resolve any questions about your foundation status, you should keep it in your permanent records.

If the heading of this letter indicates that a caveat applies, the caveat below or on the enclosure is an integral part of this letter.

If you have any questions, please contact the person whose name and telephone number are shown above.

Sincerely yours,

Teddy R. Kern

Teddy R. Kern
District Director

Letter 1078(DO/CG)

AMERICAN FRIENDS OF THE UNIVERSITY

For tax years ending on or after December 31,1982, you are required to file Form 990 only if your gross receipts are normally more than $25,000. For guidance in determining whether your gross receipts are 'normally' more than $25,000, see instructions for Form 990. If a return is required, it must be filed by the 15th day of the 5th month after the end of your annual accounting period. The law imposes a penalty of $10.00 a day to a maximum of $5,000 when a return is filed late unless there is a reasonable cause for the delay.

Beginning January 1,1984 unless specifically excepted,you must pay taxes under the Federal Insurance Contributions Act (Social Security taxes) for each employee who is paid $100 or more in a calendar year.

DEPARTMENT *of* CHEMISTRY
The University of Edinburgh
King's Buildings
West Mains Road
Edinburgh EH9 3JJ

Fax 0131 650 6452
Telex 727442 (UNIVED G)
Email R.Donovan@edinburgh.ac.uk
Telephone (direct dial) 0131 650 4730

19 December 1996

Dr Alfred & Isobel Bader
52 Wickham Avenue
Bexhill-on-Sea
East Sussex
TN39 3ER

Dear Dr and Mrs Bader

Bader Bursaries

We would like to thank you most warmly for your offer to fund four Undergraduate Bader Bursaries in the academic year 1997/98 and have great pleasure in accepting your offer.

As you know we have an intensive Access programme for schools already in operation but to date we have not had the financial means to give any of these students the opportunity to study at Edinburgh. Your bursaries will make this possible for students who would normally not have been able to consider 'Higher Education' because of their financial background. Dr Lesley Yellowlees will co-ordinate the programme on behalf of the Department using a selection scheme which is already in operation within the University. A letter from Dr Yellowlees, giving further details of the scheme, is enclosed.

We thank you again for your generosity and would like to take this opportunity to invite you both to meet with us on your next visit to Edinburgh.

With best wishes for the Festive Season and the New Year.

Yours sincerely

Robert Donovan Bob Ramage Peter Sadler

Professor RJ Donovan
Head of Department

Professor R Ramage

Professor PJ Sadler

HEAD OF DEPARTMENT Professor R J Donovan CChem FRSC FRSE

UNIVERSITY
of
GLASGOW

DAVID DEWAR MACNICOL,
Ph.D., D.Sc., F.R.S.E.

11th October, 1996.

Dr. Alfred Bader
52 Wickham Avenue
Bexhill
West Sussex TN39 3ER
U.K.

Dear Alfred,
I very much look forward to meeting yourself and Isabel when you visit Glasgow. A discussion with you on a new, very versatile!, magic-crystal bottle would be very much appreciated by me. The big jump in potential applicability arises from the stable nano-scale cavities which have tetrahedral symmetry ($\underline{T}$; 2,3) and tightly enclose C_{60}, $PbEt_4$, etc. etc etc!

[Please see reprint.]

With Best Personal Regards,
Yours sincerely,
David

DEPARTMENT OF CHEMISTRY
Joseph Black Building, University of Glasgow, Glasgow G12 8QQ
Telephone: 0141-339 8855 *Ext* *Fax:* 0141-330 4888 *Telex:* 777070 UNIGLA

triethylamine. The reaction mixture was stirred for 18 h and evaporated to dryness in vacuo. The residue was washed with ethanol, dissolved in a 1/1 ethanol/acetonitrile solution and treated with an excess of $n\mathrm{Bu_4NCl}$ in order to ensure complete exchange of any other possible ammonium cations such as $\mathrm{Ph(Me)_3N^+}$. The solution was evaporated to dryness in vacuo, washed with ethanol and recrystallized from acetonitrile. The LSI mass spectra of the resulting products were identical to those of **3** and **4**.

Received: January 5, 1996 [Z 8709 IE]
German version: *Angew. Chem.* **1996**, *108*, 1631–1634

Keywords: mass spectrometry · organic–inorganic hybrid composite · rhenium compounds

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[1] S. Uriel, K. Boubekeur, P. Batail, J. Orduna, E. Canadell, *Inorg. Chem.* **1995**, *34*, 5307–5313.

[2] a) R. Chevrel, M. Sergent, J. Prigent, *J. Solid State Chem.* **1971**, *3*, 515; b) T. Hughbanks, R. Hoffmann, *J. Am. Chem. Soc.* **1983**, *105*, 1150–1162; c) S. C. Lee, R. H. Holm, *Angew. Chem.* **1990**, *102*, 868; *Angew. Chem. Int. Ed. Engl.* **1990**, *29*, 840–856; d) T. Saito, N. Yamamoto, T. Nagase, T. Tsuboi, K. Kobayashi, T. Yamagata, H. Imoto, K. Unoura, *Inorg. Chem.* **1990**, *29*, 764–770; e) D. H. Johnston, D. C. Gaswick, M. C. Lonergan, C. L. Stern, D. F. Shriver, *Inorg. Chem.* **1992**, *31*, 1869–1873; f) N. Perchenek, A. Simon *Z. Anorg. Allg. Chem.* **1993**, *619*, 103–108; g) S. J. Hilsenbeck, V. G. Young, Jr., R. E. McCarley, *Inorg. Chem.* **1994**, *33*, 1822–1832; h) G. M. Ehrlich, C. J. Warren, D. A. Vennos, D. M. Ho, R. C. Haushalter, F. J. DiSalvo, *Inorg. Chem.* **1995**, *34*, 4454–4459.

[3] a) O. M. Yaghi, M. J. Scott, R. H. Holm, *Inorg. Chem.* **1992**, *31*, 4778–4784; b) J.-C. Gabriel, K. Boubekeur, P. Batail, *Inorg. Chem.* **1993**, *32*, 2894–2900

[4] The LSI mass spectra were recorded as described previously [1]. Helium was used as the collision gas in CID experiments [8] and the pressure was adjusted to reduce the main beam intensity to 50% of its original value. The spectra were recorded by linked scans [9] at constant B/E and B^2/E for daughter-ion and precursor-ion spectra, respectively.

[5] X-ray crystal structure analysis for **1**: $\mathrm{C_{33}H_{75}N_3S_6Cl_8Re_6}$, $M_r = 2075.13$, orange crystals, monoclinic, space group $P2_1/n$, $a = 12.895(3)$, $b = 11.468(5)$, $c = 18.365(4)$ Å, $\beta = 90.87(2)°$, $V = 2715.5(1.5)$ Å^3, $Z = 2$, $\rho_{calcd} = 2.54\ \mathrm{g\,cm^{-3}}$, $\mathrm{Mo_{K\alpha}}$ radiation, empirical absorption corrections ($\mu = 141.2\ \mathrm{cm^{-1}}$), 5943 independent reflections with ω-2θ scans were measured up to $\theta = 27°$ at 230 K on an Enraf–Nonius CAD4 diffractometer. The structure was solved by the Patterson method. The full matrix least-squares refinements of 266 parameters for 3056 reflections with $I \geq 3\sigma(I)$ converged at $R = 0.036$ and $R_w = 0.045$ (GOF = 1.297). Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no CCDC-179-51. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: Int. code +(1223) 336-033; e-mail: teched@chemcrys.cam.ac.uk).

[6] Preliminary crystallographic data for **6**: $\mathrm{C_{35}H_{81}N_3SiCl_8Se_6Re_6}$, $M_r = 2365.78$, orange platelike crystals, monoclinic, space group $P2_1/c$, $a = 24.948(1)$, $b = 11.7636(6)$, $c = 22.279(1)$ Å, $\beta = 111.287(1)°$, $V = 6092(1)$ Å^3, $Z = 4$, $\rho_{calcd} = 2.58\ \mathrm{g\,cm^{-3}}$; 20696 reflections were measured with $\mathrm{Mo_{K\alpha}}$ radiation up to $\theta = 27°$ at 295 K on a Siemens CCD-area detector based SMART X-ray diffractometer. The structure was solved by direct methods. Experiments to determine the precise structure and its triclinic low-temperature modification, are currently in progress and will be published separately.

[7] a) R. J. P. Corriu, V. Huynh, J. J. E. Moreau, M. Pataud-Sat, *Tetrahedron Lett.* **1982**, *23*, 3257–3260; b) R. J. P. Corriu, V. Huynh, J. J. E. Moreau, *Tetrahedron Lett.* **1984**, *25*, 1887–1890.

[8] a) K. Levsen, H. Schwarz, *Angew. Chem.* **1976**, *88*, 589–599; *Angew. Chem. Int. Ed. Engl.* **1976**, *15*, 509–519; b) F. W. McLafferty, P. F. Bente, R. Kornfeld, S.-C. Tsai, I. Howe, *J. Am. Chem. Soc.* **1973**, *95*, 2120–2129.

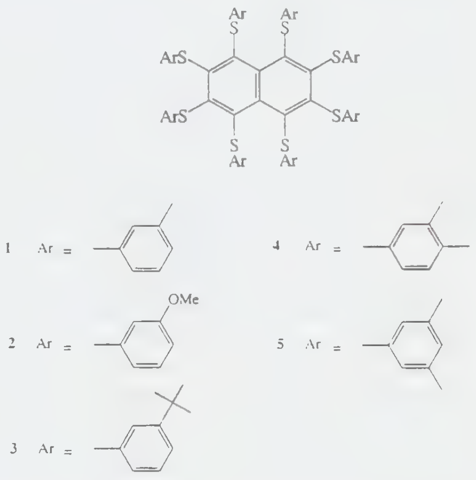
[9] R. K. Boyd, *Spectros. Int. J.*, **1982**, *1*, 169–200.

Octakis(3,4-dimethylphenylthio)naphthalene: A Designed Spider Host of Unparalled Versatility[**]

Gary A. Downing, Christopher S. Frampton, James H. Gall, and David D. MacNicol*

The exactly D_2-symmetric conformation of the spider host[1] octakis(*m*-tolylthio)naphthalene **1** is instrumental in maintaining an open tetragonal host lattice, which, remarkably, is stable even in the absence of a guest.[2] This key conformation is of the *aabbaabb* type[2] where *a* and *b* denote side chains projecting above and below the mean plane of the naphthalene core, respectively. Based on a knowledge of the crystal structure of **1**, we have sought to reproduce this key conformation by judicious modification, with a view to producing related clathrates with larger cavities suitable for versatile storage of guests[3] and lattice-controlled reactivity studies.[4] Salient results from this line of enquiry are now reported.

Initial studies involved replacement of the *meta*-methyl group in the side chains of **1** by the polar methoxy function[5] or the bulky *tert*-butyl group,[6] as in **2** and **3**; however, these changes were found to attenuate severely inclusion ability, highlighting the importance of the specific nature of the *meta* substituent in controlling host packing. In view of this, the *meta*-methyl group was retained, and octakis(3,4-dimethylphenylthio)naphthalene **4** and octakis(3,5-dimethylphenylthio)naphthalene **5**, both possessing an additional methyl group in their side chains, were targeted and prepared. Compounds **4** and **5** were found to be at the opposite ends of the inclusion ability spectrum, a direct reflection of their molecular conformations, as described below.



The introduction of a second *meta*-methyl substituent, as in **5**, is incompatible with a D_2 conformation analogous to that of **1**

---

[*] Dr. D. D. MacNicol, Dr. G. A. Downing, J. H. Gall
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ (UK)
Fax: Int. code +(141)330 4888

Dr. C. S. Frampton
Roche Research Centre
P. O. Box 8
Welwyn Garden City
Hertfordshire AL7 3AY (UK)

[**] We thank the EPSRC (UK) for support (to G. A. D.).

owing to severe *trans*-ring interactions arising from the second methyl group. This leads to the formation of a highly asymmetric *abbaabab* conformation, previously uncategorized,[2] in which two of the *peri*-related side chains have a *syn*-arrangement.[7] Compound **5** has shown no evidence of inclusion compound formation. By contrast, the sterically undemanding introduction of a *para*-methyl substituent as in **4** leaves the parent D_2 host conformation of **1** essentially unperturbed (see below), and the new compound **4** possesses truly versatile inclusion properties.

The first indication of the remarkable inclusion behavior of **4** became apparent during its isolation in the course of which ruby-red cubes containing components of the solvent (40–60° petroleum ether) were obtained on evaporation of chromatographic fractions overnight. Host **4** rapidly revealed an impressive ability to include guest species and these ranged in size from thiophosgene to squalene (Table 1). Moreover, guest classes ranged through hydrocarbons, ethers, alcohols, ketones, amines, amides, and halocarbons, for example. Particularly significant, however, is the propensity of **4** to enclathrate volatile, reactive and toxic materials, such as THF, CCl_4, thiophosgene, tri-*n*-butylphosphane, hexamethylphosphoramide (HMPA), and tetraethyllead. In order to elucidate the origin of the novel properties displayed by **4**, which indicated unusual host packing and void geometry, single-crystal X-ray analyses of the 1,4-dioxane, *t*BuOH, $PbEt_4$, and $(CH_3)_3SiC{\equiv}CSi(CH_3)_3$ adducts were carried out.[8] The adducts of **4** crystallize in the cubic space group $Pn\bar{3}$, a transformation from the tetragonal packing of **1**, which is unique for any pure organic molecular system, as established by reference to the Cambridge Crystallographic Data Base.[9]

Table 1. Representative inclusion compounds formed by **4** [a].

Guest	Host–guest ratio	Guest	Host–guest ratio
thiophosgene	1:2 [b]	methyl ethyl ketone	1:1.2
1,4-dioxane	1:1.9	*m*-xylene	1:1.1
1,4-thioxane	1:1.9	ethyl acetate	1:1.0
trichloroethylene	1:1.8	eugenol	1:1.0
CH_3CCl_3	1:1.6	isobutyrophenone	1:1.0
5-chloropent-1-yne	1:1.6	(*R*)-(+)-1-phenylethylamine	1:1.0
pyridine	1:1.5	eucarvone	1:0.8
DMF	1:1.5	*R*-(+)-limonene	1:0.7
THF	1:1.4	hexamethyldisilazane	1:0.7
benzene	1:1.4	tri-*n*-butylphosphane	1:0.6
toluene	1:1.4	$(CH_3)_3SiC{\equiv}CSi(CH_3)_3$	1:0.6
N-methylmorpholine	1:1.3	$(CH_3)_4Sn$	1:0.3
CCl_4	1:1.3 [c]	$(CH_3CH_2)_4Pb$	1:0.3
benzyl alcohol	1:1.2	HMPA	1:0.3
tert-butyl alcohol	1:1.2	squalene	1:0.3
o-xylene	1:1.2	C_{60}	[d]

[a] Analysis method ^1H NMR spectroscopy unless stated otherwise. [b] Thermogravimetric analysis. [c] Microanalysis. [d] See ref. [11].

The host molecule of **4**, located on a point of 222 symmetry, is constrained to have exact D_2 symmetry, and both enantiomeric conformations are present in the crystal. Figure 1 shows the atomic numbering scheme of the asymmetric unit, and Figure 2 the $PbEt_4$ adduct with a view along one of the twofold rotation axes. The close correspondence of this *aabbaabb* conformation[2] to that of **1** (1,4-dioxane adduct) is indicated by comparison of the related torsion angles [°] (values for **1** in parentheses): C1-C9-C9′-C1′ − 27 (− 27), C9-C1-S1-C11 + 45 (+42), C-1-S1-C11-C12 + 49 (+ 50), C1-C2-S2-C22 + 94 (+ 94), and C2-S2-C21-C22 + 11 (+11).[9b] There are two large, enantiomerically related, cavities per unit cell; these voids are chiral with symmetry 23(T), and each void is capable of including one bulky guest such as tetraethyllead or up to six[10] medium-sized guests, for example, 1,4-dioxane or 1,4-thioxane.

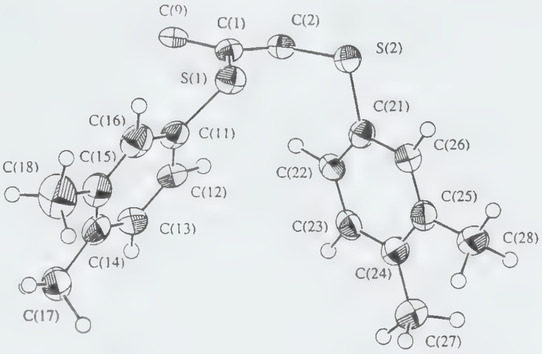


Fig. 1. View of the asymmetric unit in crystals of **4** showing atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level; hydrogen atoms are drawn as small circles of arbitary radius. Selected bond lengths [Å] and angles [°]: C(1)–C(2) 1.377(8), C(2)–C(2′) 1.42(1), C(1)–C(9) 1.444(7), C(9)–C(9′) 1.44(2); C(2)-C(1)-C(9) 120.4(6), C(1)-C(2)-C(2′) 120.2(4), C(1)-C(9)-C(9′) 116.8(4), C(1)-C(9)-C(1′) 126.3(8), C(9)-C(1)-S(1) 121.0(5) [8b].

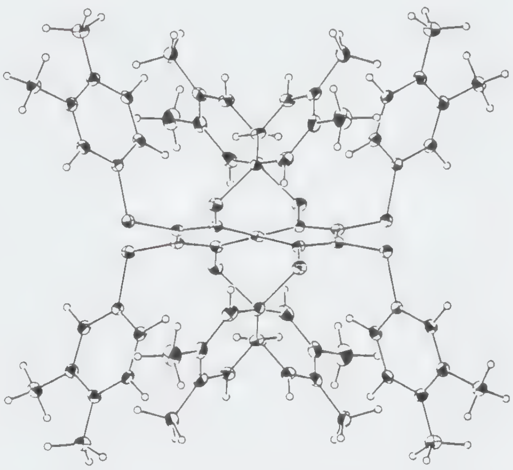


Fig. 2. Structure of **4** in its Et_4Pb clathrate. The view along the twofold rotation axis, which is coincident with the central C–C bond, illustrates the D_2-symmetric conformation of **4**.

The host–guest packing in the $PbEt_4$ adduct of **4** is shown in Figure 3. The six host molecules of **4** shown are centred round a point of $\bar{3}$ symmetry; there are, however, four such equivalent $\bar{3}$ points surrounding each cavity and, as a consequence, guest escape is blocked in all directions. The lead atom of the guest is located on a crystallographic twofold axis, hence, owing to statistical disorder, it appears as an octahedral distribution in Figure 3; the ethyl groups of the guest are highly disordered and are not shown.[11]

Recent work in the literature has described the effective control of reactions involving single molecules as well as dimerizations and polymerizations by the lattice.[4] The availability of discrete nanoscale cavities in **4**, capable of including several guest molecules, opens up the exciting possibility of carrying out controlled oligomerizations and related processes. Work along these lines is currently in progress.

Professor J D Connolly,

UNIVERSITY
of
GLASGOW

[illegible]

University of Glasg
Telephone 0141 339 8855 Ext 5499 Fax 0141 330 4888 **e-mail joec@chem.gla.ac.uk**

FAX FROM

DR. ALFRED BADER
Suite 622
924 East Juneau Avenue
Milwaukee, Wisconsin 53202
Telephone: 414/277-0730
Fax: 414/277-0709

October ~~30~~ 31, 1996

Page 1 of 3

TO: Profesor J.D. Connolly, FRSE
Department of Chemistry, University of Glasgow
FAX: 44-141-330-4888

Dear Joe:

Thank you so much for your fax of October 21st.

As you will see from the enclosed, Sir Harry Kroto and I have been friends for a long time, and I would love to be able to congratulate him personally.

Might the Chemistry Department and your Business School not also be interested in my talk #1, *The History of Aldrich*? Also, Art History and your museum might be interested in talks #4, #5 and #7. We will, of course, bring all our slides and transparencies with us, and there would be not additional costs.

I much look forward to being with you and remain, with best personal regards,

Yours sincerely,

AB/cw

Enclosures

G. W. Kirby, ScD, FRSE
Regius Professor of Chemistry

UNIVERSITY
of
GLASGOW

14 October 1996

Dr. Alfred Bader
1961 North Shepard Avenue
Milwaukee
Wisconsin 53211
USA

Dear Alfred

Alfred Bader Prize

This year's recipient is Vikki C. Pearson, who was awarded a first class honours degree in Chemistry with Medicinal Chemistry this summer. She came top of her class by a substantial margin and has now begun research with Professor David Robins, supported by a University Scholarship.

I notice that Vikki is our first female prize winner, and a very charming one too, if an ageing, male academic is allowed nowadays to make observations of this kind!

I shall retire from the Regius Chair on 31 December 1996, so the administration of the Prize will pass to my successor (not yet announced) next year. However, I shall continue here part-time as Professor of Organic Chemistry for one more calendar year, and possibly thereafter in an honorary capacity. I should like to take this opportunity to thank you formally once more for your support of our postgraduate students (7 so far).

With best wishes,

Yours sincerely,

Gordon

DEPARTMENT OF CHEMISTRY
The University, Glasgow G12 8QQ, Scotland, U.K.
Telephone: 0141-339 8855 *Ext* 4416/4417 *Telex:* 777070 UNIGLA *Fax:* 0141-330 4888

6 Braidpark Drive,
Giffnock,
Glasgow G46 6NB.

23 May 1996.

Dear Dr. Bader,

I have just returned from a short holiday to find your letter waiting. I look forward very much to meeting you when you come to this beautiful country (I refer to Scotland here) in November, and to hear what you have to say about the two men who made such important contributions to the understanding of organic chemistry.

Perhaps you would be kind enough to let me know if you will be speaking at Glasgow University as well as Edinburgh when or if that is arranged. It is certainly fitting in a way that Edinburgh should be involved where the Chair in Organic Chemistry is named after Crum Brown, and where he and Couper were in the chemistry department together for a short time, though there does not seem to be any evidence that they knew each other.

With kind regards,

Yours sincerely,

G. W. Kirby, ScD, FRSE
Regius Professor of Chemistry

UNIVERSITY
of
GLASGOW

10 October 1995

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee
Wisconsin 53211
USA

Dear Alfred,

This year's recipient of the Alfred Bader Prize is Alistair P. Rutherford. Alistair was awarded a First Class Honours Degree this summer and was placed top of the Chemistry (our main Honours course) list. Naturally, we were pleased that he decided to remain with us!

He is now starting postgraduate research, supported by a University Scholarship (awarded in open competition), under the supervision of Professor Joe Connolly.

Many thanks for returning the book so promptly, inscribed with your kind comments.

With best wishes,

Yours sincerely,

Gordon

DEPARTMENT OF CHEMISTRY
The University, Glasgow G12 8QQ, Scotland, U.K.
Telephone: 0141-339 8855 *Ext* 4416/4417 *Telex:* 777070 UNIGLA *Fax:* 0141-330 4888

9e

6 Braidpark Drive,
Giffnock,
GLASGOW G46 6NB

12th October 1995

Dear Dr. Bader,

Thank you for your letter of 29th September enclosing a copy of your Vienna paper which was waiting for me on my return from holiday in Aberdeenshire. I found the paper most interesting and was particularly intrigued by your quote from R.B Woodward's lecture - praise indeed for Couper.

It is unfortunate that the hundredth anniversary of poor Couper's death passed unnoticed a few years ago. I should have thought of it and contacted the Post Office then about a commemorative stamp as you suggest. Since the two hundredth anniversary of his birth will not fall until the year 2031, it will have to be left to others to plead his case.

It was a great pleasure to read the glowing review of your autobiography in Chemistry in Britain - the kind that most authors can only dream of.

My paper on the Crums (which contains some material on A.C.B.) is still with the British Journal for the History of Science from whom I haven't heard for some months, but if and when it appears I shall send you a copy.

I note your comments about a possible lecture on Couper at Glasgow University.

With kind regards,

Yours sincerely,

(Dr. D.G. Duff)

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

A Chemist Helping Chemists

September 27, 1995

Professor G.W. Kirby
Department of Chemistry
University of Glasgow
Glasgow G128QQ
U.K.

Dear Gordon:

Your kind letter of September 18th has given me a great deal of pleasure, and I return the book as requested.

As you perhaps know, Isabel and I spend 89 days a year in Britain, usually in November, December, June and July. Of course, I have ample stocks of the book in our home in Sussex, and if other chemists wanted signed copies, I could supply these easily from Bexhill.

With all good wishes to you and your associates, I remain,

Yours sincerely,

AB/cw

Enclosure

G. W. Kirby, ScD, FRSE
Regius Professor of Chemistry

UNIVERSITY
of
GLASGOW

18 Sept '95

Dear Alfred,

My copy of your book has just arrived from the University Bookshop. I wonder if you would be so kind as to sign it for me?

I intend to keep it for a good, continuous 'read', although I have already been tempted to dip into the text here and there. What I found has whetted my appetite for the remainder!

With best wishes,

Yours sincerely,

Gordon

DEPARTMENT OF CHEMISTRY
The University, Glasgow G12 8QQ, Scotland, U.K.
Telephone: 0141–339 8855 *Ext* 4416/4417 *Telex:* 777070 UNIGLA *Fax:* 0141–330 4888

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

A Chemist Helping Chemists

September 29, 1995

Dr. David Duff
6 Braidpark Drive
Giffnock,
Glasgow G46 6NB
Scotland

Dear Dr. Duff:

The Loschmidt Symposium in Vienna went very well indeed, and the Austrian postal authorities even printed a stamp showing Loschmidt. Now I look forward to a British stamp depicting Couper.

I gave two papers, one dealing with Loschmidt alone and the other a bridge between Anschütz, Couper and Loschmidt. A copy of that paper is enclosed.

I much look forward to receiving a copy of your paper from the British Journal for the History of Science.

As you perhaps know, my wife and I spend three months a year, usually June, July, November and December, in Britain. Perhaps someday the Chemistry Department at Glasgow will invite me to come to Scotland to speak about Couper.

With all good wishes, I remain,

Yours sincerely,

AB/cw

Enclosures

6 Braidpark Drive,
Giffnock,
GLASGOW G46 6NB

14th June 1995.

Dear Dr. Bader,

I was delighted to receive your letter this morning. I do appreciate the kindness in sending me the fascinating collection of copies of the correspondence relating to Couper's whereabouts. My German is poor so I shall have to enlist some help.

Glancing through, I was touched by Crum Brown's letter to Anschütz describing his recuperation from a four year illness and sitting up in bed knitting - a great hobby of his, and one he shared with Clark Maxwell. I suppose you will have seen the examples of Crum Brown's intricate knitting in Edinburgh?

In my last letter I think I mentioned that I was researching scientists in the Crum dynasty - including A.C.B. - and I'm now awaiting to hear from the British Journal for the History of Science if they are going to publish. If they agree, I shall send you a copy.

Best wishes for the Vienna lecture and I look forward to catching up with your autobiography.

Many thanks again.

Kind regards

Yours sincerely,

(Dr. D.G. Duff)

P.S. Note on my address, the district = GIFFNOCK and the postal code as above.

D.G.D

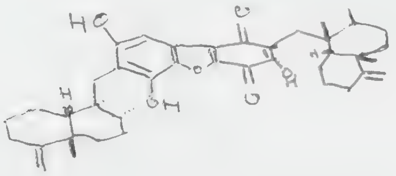
FLAT 5
60 PARTICK HILL RD
GLASGOW
G11 5AB

27TH OCTOBER 1993

Dear Dr Bader,

I would like to thank you for your support and encouragment as this years benefactor of the Alfred Bader prize in Organic Chemistry from the University of Glasgow. It came as a very pleasant surprise to me when Prof. Kirby informed me of the award as it has been a long time since my degree results.

I began work for my PhD at the beginning of October with Dr Colvin as my supervisor. The task which I have been set is to synthesize



a diterpenoid extracted from a marine sponge.

Once again thankyou for your support

Yours sincerely

Andrew Ross

G. W. Kirby, Sc.D.

Regius Professor of Chemistry

Chemistry Department
The University
Glasgow G12 8QQ
Telephone: 041-339 8855
Ext. 4416
Telex: 777070 UNIGLA

GWK/ja

20 October 1993

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee
Wisconsin 53211
USA

Dear Alfred,

This year's winner of the Alfred Bader Prize is Andrew R. Ross, who is remaining here to carry out research with Dr. Ernie Colvin, while supported by an SERC Studentship.

Andrew was awarded a First Class Honours Degree in Chemistry and achieved the highest mark (93%) in the organic paper.

I hope that you and your wife are still in the best of health and enjoying your quasi-retirement. It was a great pleasure meeting you both again on the occasion of your lectures in Glasgow.

With best regards,

Yours sincerely,

Gordon

Professor C. J. W. BROOKS
DSc, FRSC, FRSE

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
TEL.: 041-339 8855
EXT. 6583
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FAX: 041-330 4888

Mon. May 10 1993

Dr A.R. Bader,
c/o Chemistry Department,
Harvard University

Dear Alfred,

I received your letters of May 4 on Friday, but have not been able to contact Ian Gosney either then or today. Just now I have heard from Bob Ramage's secretary that Ian is away, probably at a Heterocyclics meeting in England, but may be back tomorrow – so I'll try again then.

Meanwhile, Joe was about to mail the enclosed letter and I suggested sending it to Harvard, where we hope you will receive it on Alfred Bader Day. All your friends here will wish to congratulate you on the honour – well deserved – of this Day, and to wish you and Isabel an extremely happy visit to 12 Oxford St., hopefully in the warm May sunshine that I remember from my first experience there in 1950.

Best regards,

Yours sincerely,

Charles

Dr. Alfred R. Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

May 4, 1993

Prof. Charles J. W. Brooks
Department of Chemistry
Glasgow University
Glasgow G12 8QQ, Scotland

Dear Charles:

May I ask you for your help in the following matter. Quite some time ago I was asked by your University and by Edinburgh whether I could give some talks at the end of May.

As you perhaps know, Prof. Connolly has made arrangements for my talks at your University on the 27th and 28th of May, and I plan to be at the University of Durham on Monday, May 24th.

I had left the 25th and 26th open for lectures in Edinburgh, and my letter to Ian Gosney of April 8th, copy enclosed, will be self-explanatory. Oddly, I have not heard from Ian, and I wonder whether, per chance, mail has gotten lost, or worse--Ian is sick. May I impose on you to inquire and then to let me know.

We plan to leave Milwaukee on May 14th, and on Monday, May 17th, the Harvard Chemistry Department is honoring me with an "Alfred Bader Day" to which I much look forward. We plan to fly to Gatwick on the 20th, stay at our Sussex home (0424 222 223) on Friday and Saturday, and then travel by train on Sunday from Bexhill to London and thence to Durham.

Many thanks for your help.

Sincerely,

Enclosures

Professor J.D. Connolly, FRSE

UNIVERSITY
of
GLASGOW

Your Ref:
My Ref:

Trip file

Dr Alfred Bader,
2961 North Shepard Avenue,
Milwaukee,
Wisconsin 53211,
USA.

23rd April 1993.

Dear Dr Bader,

Thank you for your letter of 8th April. I have made reservations for you in our Staff Club for the nights of 26th-28th May. The rate for a double room is £47.50 per night. I have been trying desperately to complete two reviews and a chapter and as a result I have not yet arranged the programme of visiting speakers in detail. I shall send you the proposed timetable in due course once I check on exam commitments etc. It will be easy to have two lectures - Loschmidt and the adventures - and I shall certainly try to fit in the third.

Charles, David and Gordon will all be here to greet you but Karl will be in Prague *en route* for Berlin. We are all looking forward to your visit.

Best wishes,

Yours sincerely,

Joe Connolly.

CHEMISTRY DEPARTMENT
University of Glasgow, Glasgow G12 8QQ

Telephone: 041-339 8855 *Ext* 5499 *Fax:* 041-330 4888

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

April 8, 1993

Professor J. D. Connolly
Chemistry Department
University of Glasgow
Glasgow G12 8QQ
Scotland

Dear Professor Connolly:

Our visit to Glasgow University is a little more than a month away. We plan to leave Edinburgh by train on Wednesday, May 26, to arrive in Glasgow in the early evening. Could you please make reservations for us at a simple hotel near the university for Wednesday, Thursday and Friday nights. We plan to take a train back to London on the 29th.

If it fits into your schedule, I would love to be able to give at least three talks in your department. One of these could be on "Joseph Loschmidt--the Father of Molecular Modelling." The other two talks could be "The Adventures of a Chemist Collector" and "The Bible through Dutch Eyes." The latter could be subtitled "Rembrandt and the Jews" and might be of particular interest to members of the Jewish community. Abstracts of the first two talks are enclosed. For these three talks I will need two projectors and one large white wall or two screens in order to show two slides simultaneously.

A further talk might be on the history of Aldrich and Sigma-Aldrich, and that might be of particular interest also to students in your business school. However, as you will be able to imagine, it isn't the easiest talk for me to give.

Kave - Dr David MacNicol

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

While at your University, we hope to have a chance to see old friends, Professors Brooks, Kirby, Overton and Dr. MacNicol.

I much look forward to being with you.

Sincerely,

Enclosures

6 BRAIDPARK DRIVE,
GIFFNOCK,
GLASGOW G46 6NB
U.K.

April 7th 1993.

Dear Dr. Bader,

Many thanks for sending me copies of the correspondence arising from your most interesting article on Loschmidt. I held off writing to you to see if the April volume of Chemistry in Britain would carry it, but this was not the case. What struck me when I wrote my piece on Couper was the interest it aroused from all over the place – that may have been your experience too.

Two further points are of interest:

i) The common role played by Anschütz in the rehabilitation of both Couper + Loschmidt.

ii) The fact that both C. + L. studied philosophy before tackling fundamental problems in chemistry

I trust Chemistry in Britain will publish the correspondence in full – there may even be more to come.

Best wishes in your continuing work on Loschmidt.

Yours sincerely,

David Dutt

Kan–

Dr Ian MacNico

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

April 19, 1993

Dr. David Duff
6 Braidpark Drive
Giffnock
Glasgow G46 6NB
Scotland

Dear Dr. Duff:

Thank you so much for your letter of April 7th.

I plan to be speaking on Loschmidt in the Chemistry Department at the University in Glasgow either on the 27th or 28th of May, and it would give me great pleasure if I could meet you then and chat about Couper and Loschmidt. Professor J. D. Connolly is coordinating the lectures of my visit.

Ms. Catherine O'Driscoll, Features Editor at <u>Chemistry in Britain</u>, initially thought that the two letters about Couper and reply by Professor Noe and me would be in the April issue. Then, however, <u>Chemistry in Britain</u> received an article from Professor Alan J. Rocke of Case Western Reserve University in Cleveland, stating that our arguments are deeply flawed. I do not think that Professor Rocke is correct, and we replied carefully. It may well be that <u>Chemistry in Britain</u> will want to publish the two letters and Professor Rocke's article and our two replies in one and the same issue.

When Anschütz studied the work of Couper he did not yet know anything about Loschmidt. But it is interesting that without Anschütz the work of Couper and Loschmidt would now not be known.

Your second point that both Couper and Loschmidt went from philosophy to tackling fundamental problems in chemistry is both interesting and important.

Kam

Dr. Ian MacNicol

Once the correspondence and Professor Rocke's article have appeared, do consider writing an overview of both Couper and Loschmidt in a letter to Chemistry in Britain.

I very much look forward to meeting you personally.

Sincerely,

c: Ms. Catherine O'Driscoll

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
TEL.: 041-339 8855
EXT
TELEX: 777070 UNIGLA
FAX: 041-330 4888

Ext. 5499

1st December, 1992

Dr. Alfred Bader,
52 Wickhem Avenue,
Bexhill-on-Sea,
East Sussex TN39 3ER.

Dear Dr. Bader,

I have talked to Ian Gosney and we agree that the last week in May, 1993 would be a good time for your visit. Edinburgh has its lectures on Thursdays/Wednesdays while we normally have ours on Thursdays/Fridays. We can settle the detailed programme in due course. In the meantime Ian and I shall talk to our contacts in the art galleries.

Best Wishes for 1993,

Yours sincerely,

Joe Connolly

Prof. J.D. Connolly

E Tuesday - Wed.

G Th - Fri

Kirby

Chas Brooks

Kane Overton

Dr David MacNicol

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

February 1, 1993

Mr. John McKendrick
5 Mayfield Road
Stevenston, Ayrshire
KA20 4AQ Scotland

Dear Mr. McKendrick:

I am so happy to have your letter telling me that you won the Bader Prize.

My wife and I much look forward to being at your university during the last week of May, and it would give me great pleasure if I could then congratulate you, personally.

Best wishes.

Sincerely,

5 MAYFIELD ROAD
STEVENSTON
AYRSHIRE
KA20 4AQ

Dear Sir,

I am writing to thank you for your most generous donation to Glasgow University, which is known as the Bader prize. I am this year's recipient of the prize and was very happy to recieve it.

I am working for Professor D. J. Robins in the area of lysine biosynthesis, which is partly funded by ICI. We hope in this project to obtain detailed knowledge of the mechanisms and stereospecificities of the enzyme catalysed reactions and thence to obtain inhibitors which would be of use as antibiotics or herbicides but would not hav any adverse effects on humans since onl plants and bacteria can synthesise lysine. This is an area where very little work has been undertaken and at present we have only managed to study the first enzyme in any detail, but we hope to get further into the pathway in the future.

I hope that this project will provide me with a good background knowledge of synthetic reactions and a whole breadth of practical experience. This I feel would be a good grounding as I hope to undertake a post-Doctoral post after I finish at Glasgow.

I would once again like to thank you for your generous donation to the University and I would like to thank you personally for the Prize which was much appreciated.

Thank you

John McKendrick.

Dr. Alfred Bader
2961 North Shepard Avenue
Milwaukee, Wisconsin 53211

February 1, 1993

re - Glasgow

Mr. John McKendrick
5 Mayfield Road
Stevenston, Ayrshire
KA20 4AQ Scotland

Dear Mr. McKendrick:

I am so happy to have your letter telling me that you won the Bader Prize.

My wife and I much look forward to being at your university during the last week of May, and it would give me great pleasure if I could then congratulate you, personally.

Best wishes.

Sincerely,

[illegible]

Dear Sir,

I am writing to thank you for your [illegible]

[illegible]

[illegible] we hope [illegible]

the pathways [illegible] the future.

I am sure that this project [illegible] [illegible] [illegible] knowledge [illegible] [illegible] of [illegible] [illegible] [illegible] [illegible] benefits of [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible] [illegible].

I would also like to thank you for your generous invitation to all. It certainly was a wonderful time to thank you personally for the [illegible] [illegible] were very appreciated.

Thank you

Professor C. J. W. BROOKS

Chemistry Department
The University
Glasgow G12 8QQ
Tel: 041-339 8855
Ext. ~~373~~ 6583

December 14, 1992

Dr A. R. Bader

Dear Alfred,

We are very pleased to hear from Joe Connolly that you and Isabel plan to visit Glasgow again in May, and we look forward to seeing you then

I am sure you know that Steve Ley's letter published in C & EN was one of a very large number of indignant communications, including one from me, that followed your rift with the Sigma-Aldrich board. We trust that the strength of support from your friends and colleagues was of some value in mitigating the annoyance and insult that you suffered

We were impressed earlier by your successful purchase of the portrait of Johannes Uyttenbogaert at Sotheby's. No doubt you were also aware of the "campaign" of Mr Konstam referred to in the enclosed press cutting

With our warmest greetings and best wishes for Christmas and the New Year,

Yours sincerely.

Charles & Gillian

Chemistry Department
The University
Glasgow G12 8QQ
Telephone: 041-339 8855
Ext. 4416
Telex: 777070 unigla

ick, who is remaining
ile supported by an

l the highest marks in
ed that he elected to

oth disappointed and
least because it will
I hope you and your
in the future.

Yours sincerely,

Gordon

DEPARTMENT *of* CHEMISTRY
The University of Edinburgh
King's Buildings
West Mains Road
Edinburgh EH9 3JJ

Fax 031 650 4743
Telex 727442 (UNIVED G)

Email
Telephone 031 650 1000
or direct dial 031 650 4712

8. X. 92

Dear Alfred,

I have just returned from a summer sabbatical in France to rumours that you have been asked to relinquish your Chairmanship of Aldrich. I have no idea what brought this about, but if it is true, I and indeed all of my colleagues in Edinburgh are devastated. To us, you are Aldrich! Your unselfish nature and frequent visits to Edinburgh epitomizes Aldrich as a company, or ratter used to.

I wish you and your wife well, and remember you are both welcome in Edinburgh at any time.

Have a good fast, on this day of atonement,

All best wishes,
Ian Gosney

Dr. Alfred Bader
Chairman Emeritus

September 11, 1991

Prof. Karl H. Overton
Chemistry Department
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Karl:

Thank you for that delightful book by Gerard Hoffnung. Isabel likes the triangle the best because it reminds her of the triangle she played in a rhythm band when she was a little girl. Never having played any instrument, I like so many so well that I just can't pick.

My calendar for this autumn has filled up so very much that we couldn't plan to visit Scotland, much as we would like to. Might a visit early in July be welcome? Or, would November of 1992 really be better?

A list of possible talks is enclosed; the one you will, I believe, enjoy the most is the one on Josef Loschmidt.

Fond regards from both of us,

Alfred Bader
AB:mmh
Enclosure

SIGMA-ALDRICH
P.O. Box 355 Milwaukee Wisconsin 53201 USA Telephone (414) 273-3850 Cable Aldrichem TWX 910-262-3052 Telex 26-843

Chemistry Department

University of Glasgow

Summer Term 93

COLLOQUIA

Dr. A. BADER

"THE ADVENTURES OF A CHEMIST COLLECTOR"

2.00p.m.
THURS. 27th. MAY

"JOSEPH LOSCHMIDT- THE FATHER OF MOLECULAR MODELLING"

4.00p.m.
FRIDAY 28th. MAY

ORGANIC LECTURE THEATRE

all welcome

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
TEL.: 041-339 8855
EXT
TELEX: 777070 UNIGLA
FAX: 041-330 4888

Sunday Nov 26

Dr & Mrs A-R Bader

Dear Alfred and Isabel,

I hope you have had a smooth journey to the north, and trust that the room at No. 1 will be satisfactory. There is a small kitchen on the ground floor which allows you to make tea, coffee etc.

If you arrive without having had dinner, please phone us at home (942-0514). Karl Overton and his friend Myra (a professional 'cellist) will be joining us for an informal meal this evening, and you would be welcome to share this or to come later for coffee (+ 'drinks', if you are not abstaining!)

Tomorrow morning you may care to phone Prof Kirby (internal extension 4416) or me (6583). Karl, David MacNicol and I will all be rather heavily committed to teaching but David's is mainly limited to 10-12 I believe.

Yours sincerely, Charles

ALDRICH®
Chemists Helping Chemists in Research and Industry
aldrich chemical company, inc.

Dr. Alfred Bader
Chairman

November 6, 1990

Mr. John Maguire
c/o Dr. D. G. Morris
Department of Chemistry
Glasgow University
Glasgow G12 8QQ, Scotland

Dear Mr. Maguire:

Thank you for your gracious letter of October 26th.

You wouldn't believe how many chemists, including many at Glasgow University, have helped us over the years. The Bader Prize is just a small way to say thank you.

I very much look forward to meeting you personally when next we visit your department.

Best personal regards.

Sincerely,

Alfred Bader
AB:mmh

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

34 King Street,
Clydebank,
Dunbartonshire,
Scotland.
G81 1DX

26th October 1990

Dear Dr. Bader,

I have recently started a Ph.D in Organic Chemistry at Glasgow University on the "Synthesis and Investigation of Potential Organic Conductors", supervised by Dr. D.G. Morris. Recently Prof. Kirby asked me along to his office where I was informed that I had received "The Alfred Bader Prize in Organic Chemistry." To say I was taken aback is an understatement, especially since I had no idea that such a prize existed. I would therefore like to thank you for the Trust fund which was presented to Glasgow University.

Of the £1,000 prize I have already given my mum and brother part of the sum, as for the rest I shal either buy an electric keyboard or spend it on a holiday next year.

Once again I would like to thank you, and I shall let you know of my trials and tribulations as a research student.

Yours gratefully,

John Maguire.

Professor C. J. W. BROOKS
DSc, FRSC, FRSE

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
TEL.: 041-339 8855
EXT. 6583
TELEX: 777070 UNIGLA
FAX: 041-330 4888

Thursday Nov. 30 1989

Dr A. R. Bader

cc SB

Dear Alfred,

Just a note to report that Gordon Kirby has just returned, and I have given him notes on your proposed offer of a Prize. He is very pleased, and hopes that G.U. will be able to respond favourably – anyway he will contact you soon.

Your lectures were very much appreciated – several colleagues have told me of their enjoyment of 'The Detective's Eye', and of their disappointment that teaching duties prevented attendance on the Monday. Perhaps next time we should go for post-5 p.m. in all instances.

I enclose a page of notes that I made for a discussion with Ernie Colvin recently. He advised me that the preparation of chiral silicon halides (or other electrophilic reagents analogous to these) would be beyond the ability of a Final Year student. We agreed to continue thinking about the problem, and if any better ideas emerge, we will contact you or your colleague Dr Steven Branca, whose helpful notes you sent earlier.

With best regards to Isabel and yourself,

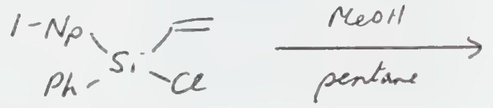
Yours sincerely,

Charles

Nucleophilic displacements at silicon.

from RJP Corriu *et al*. Top. Stereochem. 1984, 15, 43–

p.82.



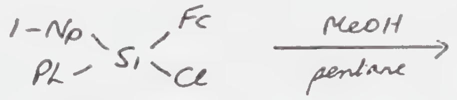
R_3SiOMe 100% retention

(A) $[\alpha]_D$ −5.7° (CCl_4) $[\alpha]_D$ −9.3°

RJP Corriu *et al*. J. Organometal. Chem. 1968, 14, 291

Bull. Soc. Chim. Fr. 1972, 4, 1490

(Apparent contradiction on p108 "chlorosilanes react with inversion, whatever the nucleophile".)



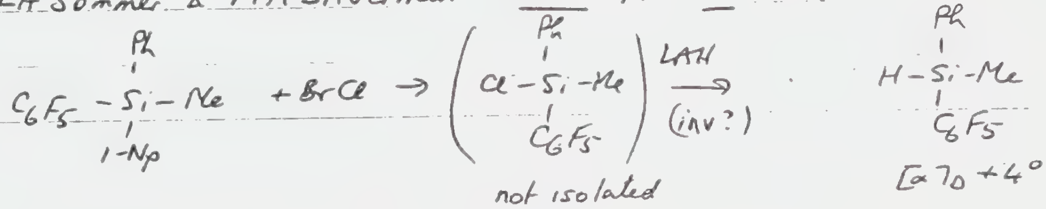
$[\alpha]_D$ + 33.8° (CCl_4)

R_3SiOMe Predominant inversion

$[\alpha]_D$ −14.8°

R_3SiH seems to be more stable than R_3SiCl e.g.

LH Sommer & MA Silverman JOC 1973 38 636.



$[\alpha]_D$ +4°. (B)

Though less convenient than halides, hydrides could still be used to form silyl ethers if sufficiently base-stable. [Cyclic ethers form easily e.g.

].

Compounds A & B are of low enough mol. wt. to allow GLC study of derived ethers (with e.g. monoterpenols etc.)

We have examined only (±) Ph MeO tBu SiBr which is of rather limited value: several types of chiral alcohols give diastereomers that separate by GLC so one can distinguish these from any achiral alcohols [illegible] is present in a mixture.

G. W. KIRBY, SC.D.

Regius Professor of Chemistry

CHEMISTRY DEPARTMENT
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TELEPHONE: 041-339 8855
EXT. 4416
TELEX: 777070 UNIGLA

GWK/ja

30 November 1989

Dr. A.R. Bader
52 Wickham Avenue
Bexhill-on-Sea
East Sussex
TN39 3ER

Dear Alfred,

I was very sorry that the Strathclyde virus prevented my attending your lectures and enjoying your company at dinner. I have heard enthusiastic reports of both your talks, which only increases my disappointment.

I was very pleased to hear from Charles Brooks the outline proposal for your new annual Prize for a student proceeding to study for a Ph.D. in Organic Chemistry in this Department. I think this is an excellent idea and personally welcome the special requirement of the student. When I receive details from you I shall take the matter up with the University administration in the hope that the University Court will accept the special, and probably unprecedented, conditions. I feel that a prize amounting to 10% of the capital is perfectly reasonable with current high interest rates although the University would probably prefer a more conservative figure. You may be sure that I shall try very hard, if necessary, to persuade them.

With best wishes,

Yours sincerely,

Gordon

Chemists Helping Chemists in Research and Industry

aldrich chemical company, inc.

Dr. Alfred Bader
Chairman

November 8, 1989

Not needed
[illegible]

Prof. C. J. W. Brooks
Department of Chemistry
Glasgow University
Glasgow G12 8QQ, Scotland

Dear Charles:

Thank you for your gracious letter of October 27th.

I will be happy to give whatever lectures you would like, wherever they will be most convenient. We plan to arrive in Glasgow on Sunday evening, November 26th, and hope that the College Club will reserve the room for late arrival.

We plan to spend Monday and Tuesday mainly at Glasgow University, but I have written to Prof. Pauson at the University of Strathclyde and would like to spend about an hour with him sometime on Monday or Tuesday. I think that one of our Ph.D. chemists from Aldrich Limited will come to Glasgow and Edinburgh to be with us, and if so, he will most likely come by car. Thus, transportation both to Strathclyde and Edinburgh should be no great problem, and I am asking Aldrich Limited to make room reservations in Edinburgh.

Of course, Isabel and I very much look forward to being with you. I don't know of any university anywhere which has ever received us as kindly as you and your associates did on our first visit last year. And, of course, there is the added attraction of being able to chat with Prof. Overton about our high school in Vienna. He and Carl Djerassi and I went to that school at pretty much the same time!

Best personal regards.

Sincerely,

Alfred Bader
AB:mmh

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

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27th October, 1989

Dr. Alfred R. Bader,
Chairman,
Sigma-Aldrich,
P.O. Box 355,
Milwaukee,
Wisconsin 53201,
U. S. A.

Dear Alfred,

I received the FAX from Ms. Hassmann, and have reserved your accommodation at No.1 The Square - the College Club rooms - for Sunday and Monday, November 26/27. I enclose some information leaflets: the Hunterian Art Gallery is just across the road from No.1, and should be open at 10 a.m. on the 27th.

Miss Pace (History of Art) 'phoned me this morning to say that Professor Tait was concerned that they might not attract a sufficient audience for your lecture. Accordingly, after consulting Professor Kirby, I have provisionally booked the same lecture room in this Department that is scheduled for your Monday talk. I hope the new proposed time of 5.15 p.m. on Tuesday will not cause you undue difficulty: as you know, trains to Edinburgh are frequent, and you could arrive within 60-70 min. of leaving this Department. The great benefit of the later time is that many of us could attend, as our teaching duties end at 5 p.m. The History of Art colleagues, and other interested colleagues, will of course come over too.

With best regards,

Yours sincerely,

Charles

Professor C.J.W. Brooks

P.S. Do you have any plans for Tuesday lunch – e.g. do you wish me to contact Peter Pauson at U. Strathclyde?
C

Dr. Alfred Bader
Chairman

October 19, 1989

Prof. Charles J. W. Brooks
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Charles:

Thank you for your gracious fax of yesterday.

I will be happy to present a talk on how we select new products on Monday, November 27th, at 4:15 p.m. Of course, I would also be delighted to give a talk at the Department of the History of Art.

We won't make any plans for the evening of the 27th, although I very much hope also to have a little time with Prof. Carl Overton. When I first talked to him last year, I had hoped that he might be one of Isabel's relatives; her family, Overton, comes from Britain. We then discovered that we are not related, but that we went to the same high school in Vienna! That distinction we share with Sigmund Freud and Carl Djerassi.

May I ask you for your help in one other matter? We plan to come up on the Sunday and stay until late Tuesday afternoon when we will go to Edinburgh. Could I impose on you to reserve for Isabel and me a double room, preferably with one large bed. We are always very uncomfortable in three or four star hotels, but much prefer quite modest hotels. However, we have gotten so old that we also prefer a toilet and shower in the room, and that is not always easy to get in one or two star hotels. I would very much appreciate any help you can give us in making such a reservation, of course at our expense. Ideally, the hotel should be within walking distance of the University, if such is at all possible.

We very much look forard to being with you and your associates.

Best regards.

As always,

Alfred Bader
AB:mmh

SIGMA-ALDRICH

P.O. Box 355 Milwaukee Wisconsin 53201 USA Telephone (414) 273-3850 Cable Aldrichem TWX 910-262-3052 Telex 26-843

Professor C.J.W. BROOKS

Chemistry Department
The University
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EXT. ~~XX~~ 4373/6583

ET

13th October, 1989

Dr. Alfred Bader,
Chairman,
Aldrich Chemical Co., inc.,
P.O. Box 355,
Milwaukee,
Wisconsin 53201,
U. S. A.

Dear Alfred,

Many thanks for your letter of September 19 confirming that you plan to be in Glasgow during November 26th-28th. I have ascertained from British Rail that the Glasgow/London fare for holders of Senior Railcards (i.e. those aged over 60 who have purchased these) is expected to be £18, and I hope this rate holds for London/Glasgow too! We are very much looking forward to your visit.

I appreciated the comments kindly made by Dr. Stephen Branca about the possible routes to chiral silyl ethers. I had noted the report (Topics in Stereochem. 1984, 15, 43) that chiral compounds of type R_3SiX, including halides, were optically stable but were racemised in the presence of nucleophiles. However, it seemed possible that the preservation of configuration might be improved by making some R groups quite bulky. Dr. Branca's suggestions of using trialkylsilanes or trialkylsilyl ethers as reagents are more attractive, and the latter type, as he indicates, might offer scope for development of chiral reagents to be used in transetherification. I am sure this is an aim worth pursuing, and I hope you will have time for a short discussion when you come.

With best regards to Mrs. Bader and yourself,

Yours sincerely,

Charles

cc SB

Dr. Alfred Bader
Chairman

October 11, 1989

Prof. Charles J. W. Brooks
Chemistry Department
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Charles:

Since writing to you abouot my visit to Glasgow on the 27th and 28th of November, a number of English universities have invited me to give various lectures. For instance, University College has asked me to speak on "Challenges at Sigma-Aldrich" on November 21st, King's College on "Adventures of a Chemist-Collector" on November 23rd, and Oxford University on "Adventures of a Chemist-Collector" and perhaps "The Bible through Dutch Eyes" on November 30th.

Hence, I will be taking my trusty slides with me, and I am wondering whether you might like me to give a talk or two at Glasgow. You would have the choice from a number of talks as described on the enclosed. Of course there would be no cost to the department whatsoever, as I will be in Glasgow anyway, but I would appreciate a cup of tea.

Talk or no talk, I very much look forward to being with you.

Best personal regards.

As always,

Alfred Bader

AB:mmh

Enclosure

SIGMA-ALDRICH

P.O. Box 355 Milwaukee Wisconsin 53201 USA Telephone (414) 273-3850 Cable Aldrichem TWX 910-262-3052 Telex 26-843

Via Veritas Vita

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ET

26th September, 1989

Dr. Alfred Bader,
Chairman,
Aldrich Chemical Company, inc.,
P.O. Box 355,
Milwaukee,
Wisconsin 53201, U.S.A.

Dear Dr. Bader,

Many thanks for your recent letter which I received on returning from holiday on the West Coast of Scotland. Remarkably good weather for so late in the season!

I look forward very much to discussions during your visit to Scotland in November. Fortunately Du Pont has come up with postdoctoral support for the fluorine work and also I have a new keen Ph.D. student, Gary Downing, just starting. So things are showing a marked up-turn!

With best regards,

Yours sincerely,

David Dewar MacNicol

Dr. D.D. MacNicol

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aldrich chemical company, inc.

Dr. Alfred Bader
Chairman

September 19, 1989

Prof. Charles J. W. Brooks
Chemistry Department
University of Glasgow
Glasgow G12 8QQ, Scotland
Dear Charles:

I am so happy to have your kind letter of September 6th and very much look forward to being in Glasgow from November 26th through the 28th. Surely in Scotland our reason for wanting to come in November will be particularly appreciated: train fares in November are really very low.

I am glad that you are interested in our 33762-5, bromo-tert.-butylmethoxyphenylsilane, which we make ourselves in Milwaukee. I wish I knew just how to resolve such a compound; do note our Dr. Stephen Branca's comments, enclosed. Let me discuss this with you when I see you.

Best personal regards.

Sincerely,

Alfred Bader

AB:mmh

Enclosure

cc: Dr. Stephen Branca

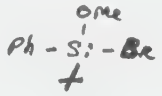
P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

To Alfred

From Steve

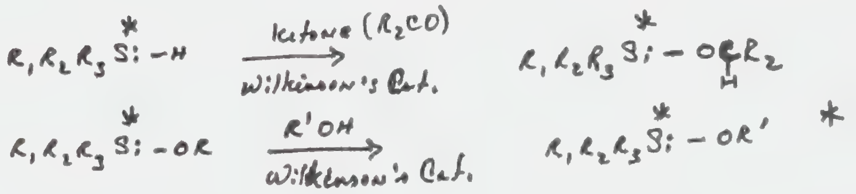
Date 9/18/89

Subject attached letter ex J. Glasgow:

① 

$$Ph-\overset{OMe}{\underset{+}{Si}}-Br$$

: As far as I know the only way a chiral form of this product can be prepared is via resolution (since it is made from $Ph_2\underset{+}{Si}{-}OMe$); the compound has no acid or base site to use for resolution; halosilanes readily racemize thus even if possible it would not be configurationally stable.

② 

$$R_1R_2R_3\overset{*}{Si}-H \xrightarrow[\text{Wilkinson's Cat.}]{\text{ketone }(R_2CO)} R_1R_2R_3\overset{*}{Si}-O\underset{H}{C}R_2$$

$$R_1R_2R_3\overset{*}{Si}-OR \xrightarrow[\text{Wilkinson's Cat.}]{R'OH} R_1R_2R_3\overset{*}{Si}-OR' \quad *$$

* equation seems to be the best method for derivatizing ROH with chiral Silicon; there may be a better more recent method but I'm not aware of any.

Can ~~Ms. Shaw~~ Prof. Brooks suggest a specific compound?

SL

Professor C.J.W. BROOKS

Chemistry Department
The University
Glasgow G12 8QQ
TEL: 041-339 8855
EXT.~~XXX~~ 4373/6583

Dr. Alfred Bader,
Chairman,
Aldrich Chemical Co. Inc.,
P.O. Box 355,
Milwaukee,
Wisconsin 53201, U.S.A.

6th September, 1989

Dear Alfred,

I was very pleased to receive your letter of August 24, which arrived just as I was discussing with David MacNicol the prospects for your visit. I have spoken with Prof. Gordon Kirby (to whom you wrote as well) and he confirms that you would be most welcome to visit us in November. The dates are not ideal, as most of the organic staff will have intensive teaching duties - e.g. our 4th Year Techniques Course begins on November 27. No doubt we can check later, via Dr. Bob Smith, regarding the timing of your visit, to ensure that it will be as productive as possible. I am reminding colleagues of your interest in research chemicals, and I hope to collect a few samples of my own to show to you.

SB
✓

Dr. John Cole and I are currently using your interesting reagent No. 33,762-5 (bromo-t-butyl-methoxyphenylsilane) in a model study of the gas chromatographic behaviour of the diastereomeric ethers it forms with chiral alcohols. In several instances, good peak resolution is observed, and we are wondering whether this reagent, or a related chiral silane, could be obtained as a single enantiomer. The availability of such a compound would greatly facilitate work on the stereochemistry of silylation. From the limited investigations in this area, it seems that silicon shows surprising configurational stability in, for example, trialkylsilanes (cf. R.J.F. Corrin, et al., Topics in Stereochem., 1984, 15, 43). It may be possible to employ suitable chiral silylating agents in the gas chromatographic analysis of enantiomeric alcohols: as far as I know, this has not yet been achieved, whereas of course chiral acylating agents of many kinds have found extensive applications, especially in drug metabolite analyses by GC-MS.

I look forward to the pleasure of seeing you again in November.

With best regards,

Yours sincerely,

Heather Shaw

pp. Professor C.J.W. Brooks

PROFESSOR K. H. OVERTON, D.Sc., F.R.S.E.

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
TEL.: 041-339 8855
EXT. 4414

ET

13th. September 1989

Dr. Alfred Bader,
Chairman,
Aldrich Chemical Company Inc.,
P.O. Box 355,
Milwaukee 53201,
U.S.A.

Dear Alfred,

I was delighted to get your letter and to see that you and your wife are going to be in Glasgow at the end of November. I was really disappointed that we discovered our congenerity (I have just made that up - I think)* too late on your last visit. I have made a note of the dates in my diary and greatly look forward to spending a good reminiscing evening with you both.

Very sincerely,

Karl

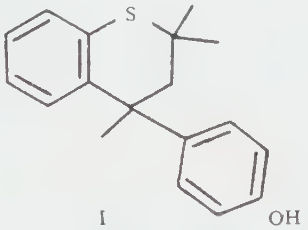
P.S. Siegmund Freud I did not know about. But it has just occurred to me that Norbert Brainin might well be another candidate for distinguished membership; I must ask him.

* You must have had an extraordinary man called Noel (even the name didn't strike me as funny at the time) for Latin? His head was entirely glabrous, as I remember and he stumped about in semi-military fashion, re-inforcing the declensions by applying a book to one's head in time - or am I making that up? And Biener for History - the Versetzungsman!

(Reprinted from Nature Vol. 245, No. 5421, p. 146 only, September 21, 1973)

Clathration as a Practical Method for Handling Dimethylmercury

THE interest in dimethylmercury in connexion with studies of environmental pollution prompts us to report a convenient and comparatively safe method for handling this volatile and extremely toxic compound. We have found that dimethylmercury may be stored, until ready for release, in the form of an inclusion compound, the organometallic guest being accommodated in the relatively large, approximately hour-glass shaped, cavity[1,2] formed by the clathrate[3] host, 4-*p*-hydroxyphenyl-2,2,4-trimethylthiochroman (Structure I).



I

This represents the first successful attempt to prepare a stable inclusion compound containing dimethylmercury, although diethylmercury and longer chain mercury dialkyls have been reported[4] to form inclusion compounds with urea; diethylmercury has also been included[5] in the tri-*o*-thymotide chanel structure.

The highly crystalline adduct, prepared by recrystallization of unsolvated[3] (Structure I) from neat liquid dimethylmercury (caution), is found by NMR analysis to have a molecular ratio of host to guest of 6:1 (microanalysis requires 10.35% Hg; found 10.42% Hg). This host-to-guest ratio corresponds to single occupancy[1-3] of the clathrate voids.

The crystals suffer no detectable loss of guest (NMR analysis) even when pumped for several days under vacuum, the closed nature of the clathrate cage[3] preventing escape of dimethylmercury*. Grinding the crystals does, however, liberate sufficient guest to be detectable by mass spectrometry. Thus when crystals of the adduct in an evacuated flask attached to the inlet of a mass spectrometer are finely ground by using a magnet to agitate a small steel ball in the flask, the mass spectrum of dimethylmercury is observed. The minimal interference found

* Crystals of this type are stable for years!

from the spectrum of the host makes this an attractive method for obtaining mass spectra of guest molecules in clathrates.

The dimethylmercury 4-*p*-hydroxyphenyl-2,2,4-trimethylthiochroman clathrate is very convenient for preparing solutions of known concentration in dimethylmercury, preweighed quantities of the adduct merely being dissolved in the appropriate solvent.

Controlled release of organometallic guests from Structure I and other host lattices promises to be of considerable value in, for example, organic synthesis—especially where guest materials are difficult, or hazardous, to handle in the free state.

R. J. CROSS
J. J. MCKENDRICK
D. D. MACNICOL

Department of Chemistry,
University of Glasgow,
Glasgow G12 8QQ

Received July 2, 1973.

[1] MacNicol, D. D., Mills, H. H., and Wilson, F. B., *Chem. Commun.*, 1332 (1969).
[2] MacNicol, D. D., and Wilson, F. B., *Chem. Commun.*, 786 (1971).
[3] MacNicol, D. D., *Chem. Commun.*, 836 (1969).
[4] Bähr, G., and Meier, G., *Z. anorg. allg. Chem.*, **294**, 22 (1958).
[5] Lawton, D., and Powell, H. M., *J. chem. Soc.*, 2339 (1958).

Printed in Great Britain by Flarepath Printers Ltd., St. Albans, Herts.

Professor C.J.W. BROOKS

Chemistry Department
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6th September, 1989

Dr. Alfred Bader,
Chairman,
Aldrich Chemical Co. Inc.,
P.O. Box 355,
Milwaukee,
Wisconsin 53201, U.S.A.

Dear Alfred,

I was very pleased to receive your letter of August 24, which arrived just as I was discussing with David MacNicol the prospects for your visit. I have spoken with Prof. Gordon Kirby (to whom you wrote as well) and he confirms that you would be most welcome to visit us in November. The dates are not ideal, as most of the organic staff will have intensive teaching duties - e.g. our 4th Year Techniques Course begins on November 27. No doubt we can check later, via Dr. Bob Smith, regarding the timing of your visit, to ensure that it will be as productive as possible. I am reminding colleagues of your interest in research chemicals, and I hope to collect a few samples of my own to show to you.

SB

Dr. John Cole and I are currently using your interesting reagent No. 33,762-5 (bromo-t-butyl-methoxyphenylsilane) in a model study of the gas chromatographic behaviour of the diastereomeric ethers it forms with chiral alcohols. In several instances, good peak resolution is observed, and we are wondering whether this reagent, or a related chiral silane, could be obtained as a single enantiomer. The availability of such a compound would greatly facilitate work on the stereochemistry of silylation. From the limited investigations in this area, it seems that silicon shows surprising configurational stability in, for example, trialkylsilanes (cf. R.J.F. Corrin, et al., Topics in Stereochem., 1984, 15, 43). It may be possible to employ suitable chiral silylating agents in the gas chromatographic analysis of enantiomeric alcohols: as far as I know, this has not yet been achieved, whereas of course chiral acylating agents of many kinds have found extensive applications, especially in drug metabolite analyses by GC-MS.

I look forward to the pleasure of seeing you again in November.

With best regards,

Yours sincerely,

Heather Shaw

pp. Professor C.J.W. Brooks

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Dr. Alfred Bader
Chairman

August 24, 1989

Prof. Charles J. W. Brooks
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Prof. Brooks:

You will have realized how very much I enjoyed seeing you again after so many years, last December.

You and your associates treated us so kindly that we already look forward to coming back to the University of Glasgow, probably on Monday and Tuesday, November 27th and 28th. I do hope that a visit with you at that time will be convenient.

Also, if, per chance, you will then have any research samples which you no longer require, I would be happy to purchase them for our Library of Rare Chemicals.

Best personal regards,

Alfred Bader

AB:mmh

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

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Dr. Alfred Bader
Chairman

August 24, 1989

Dr. David MacNicol
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Dr. MacNicol:

Thanks to your thouoghtfulness, we really enjoyed our visit to the University of Glasgow last December, and we already look forward to being with you again this coming November, probably on November 27th and 28th.

I very much hope that a visit with you at that time will be convenient and that we can discuss your fascinating "crystal bottles."

Also, if you should be able to find any other research samples, we would be glad to purchase them for our Library.

Best personal regards.

Sincerely,

Alfred Bader

AB:mmh

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Dr. Alfred Bader
Chairman

August 24, 1989

Prof. G. Kirby
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Prof. Kirby:

Isabael and I still remember with great pleasure our visit with you last December.

Your and your associates' reception was so kind--even though that was our first visit to Glasgow--that we have decided to try and make our visit to Glasgow an annual affair.

We very much look forward to being with you during the last week in November, probably on November 27th and 28th.

You will have realized how shaken we were when you told us that so many interesting research samples in your department were discarded. If any others should have turned up, we would be happy to purchase them.

I much look forward to seeing you again.

Sincerely,

Alfred Bader

AB:mmh

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Dr. Alfred Bader
Chairman

August 22, 1989

Prof. Karl Overton
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Prof. Overton:

You will have realized how very sorry Isabel and I were that we couldn't get to spend more time with you during our visit to Scotland last year.

We are now planning to be in Glasgow at the beginning of the last week of November, probably on November 27th and 28th, and it would give us great pleasure if we could get together then.

The only person to whom I have been able to chat about The Sperleum is Carl Djerassi at Stanford, and it will be such fun to compare notes with you.

I read just the other day that the graduates of The Sperleum in Vienna held a reunion where the most distinguished graduate mentioned was an assistant director of the police in Vienna. Clearly, the authorities in charge had not heard of Carl Overton or Carl Djerassi or Sigmund Freud.

We much look forward to seeing you.

Best personal regards,

Alfred Bader

AB:mmh

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CHEMISTRY DEPARTMENT
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EXT.

29th March 1989

Dr. Alfred Bader,
Aldrich Chemical Company Inc.,
P.O. Box 355,
Milwaukee,
Wisconsin 53201,
U. S. A.

Dear Dr. Bader,

Many thanks for your letter of March 20, 1989 and also for sending, via Gillingham, the four most interesting samples from the Gilman Collection which have arrived safely. These are of great interest to us.

Although the odour of thiophosgene is faint, I agree that we should have a first-rate product to meet the usual stringent Aldrich standards! The sales of this particular "Crystal Bottle" product might be low anyway and I think that Al_2Me_6 might be better. A hydrocarbon host for this guest has already been designed and awaits synthesis.

Leakage in general with these hosts does not present a problem. I enclose a reprint of some of our early work on dimethylmercury enclathrated in 4-p-hydroxyphenyl-2,2,4-trimethylthiachroman, published in Nature (London), 1973. The product is (fortunately!) odourless and guest release may be achieved as described (or simply by melting under vacuum).

With best wishes. I look forward to meeting you again, in November.

Yours sincerely,

David Dewar MacNicol

Dr. D.D. MacNicol.

(*Reprinted from Nature Vol.* 245, *No.* 5421, *p.* 146 *only,*
September 21, 1973)

Clathration as a Practical Method for Handling Dimethylmercury

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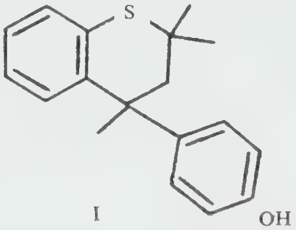

I

The highly crystalline adduct, prepared by recrystallization of unsolvated[3] (Structure I) from neat liquid dimethylmercury (cation), is found by NMR analysis to have a molecular ratio of host to guest of 6:1 (microanalysis requires 10.35% Hg; found 10.42% Hg). This host-to-guest ratio corresponds to single occupancy[1–3] of the clathrate voids.

The crystals suffer no detectable loss of guest (NMR analysis) even when pumped for several days under vacuum, the closed nature of the clathrate cage[3] preventing escape of dimethylmercury*. Grinding the crystals does, however, liberate sufficient guest to be detectable by mass spectrometry. Thus when crystals of the adduct in an evacuated flask attached to the inlet of a mass spectrometer are finely ground by using a magnet to agitate a small steel ball in the flask, the mass spectrum of dimethylmercury is observed. The minimal interference found

* Crystals of this type are stable for years!

from the spectrum of the host makes this an attractive method for obtaining mass spectra of guest molecules in clathrates.

The dimethylmercury 4-*p*-hydroxyphenyl-2,2,4-trimethylthiochroman clathrate is very convenient for preparing solutions of known concentration in dimethylmercury, preweighed quantities of the adduct merely being dissolved in the appropriate solvent.

Controlled release of organometallic guests from Structure I and other host lattices promises to be of considerable value in, for example, organic synthesis—especially where guest materials are difficult, or hazardous, to handle in the free state.

R. J. CROSS
J. J. MCKENDRICK
D. D. MACNICOL

Department of Chemistry,
University of Glasgow,
Glasgow G12 8QQ

Received July 2, 1973.

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Dr. Alfred Bader
Chairman

March 20, 1989

Dr. David D. MacNicol
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Dr. MacNicol:

Thank you for your very kind letters of February 21 and February 24.

In the meantime, I hope that the four samples made by associates of Prof. Henry Gilman, which we sent via Gillingham, have reached you.

It might well be better to wait until you have perfected the inclusion of thiophosgene in such a way that the odor of thiophosgene is not quite obvious. When you have succeeded, I would appreciate your sending me a small quantity via Aldrich Limited.

Do you have exact instructions how to get compounds such as thiophosgene out of your "Crystal Bottle?"

Of course, we are happy to wait for an article for our Aldrichimica Acta until you feel that the time is right.

Isabel and I plan to come to England twice this summer, but will probably not be able to visit Scotland until November. Of course, we very much look forward to seeing you then.

Best personal regards,

Alfred Bader

AB:mmmh

cc: Mr. Philip Hadley, Aldrich Gillingham
Dr. Stephen Branca

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

P.S. Samples have now arrived from Phil Hadley. Many Thanks!

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
TEL.: 041-339 8855
EXT. 5289

24th February, 1989

Dr. Alfred Bader,
Chairman,
Sigma-Aldrich,
P.O. Box 355,
Milwaukee,
Wisconsin 53201,
U. S. A.

Dear Dr. Bader,

Many thanks for your letter of February 17, 1989. I was very pleased to hear you find the research samples of interest and enclose, as requested, clarification of the structures of a few compounds.

By this time you should have received my letter dated 21 February, 1989. Do you think that I should send the sample containing thiophosgene to Philip Hadley in Gillingham in its present state or would you prefer further development?

Looking forward very much to meeting you and Isabel again, later this year.

With best wishes and kind regards,

Yours sincerely,

David Dewar MacNicol

Dr. D.D. MacNicol

Encl.

P.S. Samples have now arrived from Phil Hadley. Many Thanks!

CHEMISTRY DEPARTMENT
THE UNIVERSITY
GLASGOW G12 8QQ
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EXT. 5289

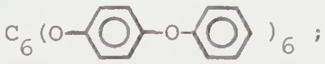
Dr. Alfred Bader,
Chairman,
Sigma-Aldrich,
P.O. Box 355,
Milwaukee,
Wisconsin 53201,
U.S.A.

21st February, 1989

Dear Dr. Bader,

I have just returned from an ACS conference in Florida and am trying to catch up with teaching and a mountain of mail. First of all many thanks indeed for your most kind letter of January 3, 1989. It was a great pleasure to meet yourself and Isabel on your visit to Scotland! Many thanks also for the £1000 cheque and the magnificent Aldrich Libraries of Spectra which are proving very useful to us.

I am extremely keen to collaborate with you on "The Crystal Bottle" venture. We have now included thiophosgene in



unfortunately the chlorine ratio by microanalysis is a bit high! This probably means that some $S=CCl_2$ is in macroscopic defects rather than in the (singly occupied) closed clathrate cages. The golden yellow crystals, as a result, disappointingly retain a slight, yet perceptible odour of thiophosgene. I am afraid this may reduce the desirability of the present product. However I feel these difficulties can certainly be overcome by modest adjustments of the host structure and in principle many useful and labelled guests should be transportable by this method. Our Industrial Liaison people are looking at the possibility of registering the name "The Crystal Bottle", following your much-appreciated suggestion.

The idea of a review article in Aldrichchimica Acta on the inclusion theme greatly appeals to me - perhaps a good time for this (if it would fit in with your plans) might be just after the appearance of Volumes 4 and 5 of Inclusion Compounds, currently in preparation.

Wishing you all the best and kind regards,

Yours sincerely,

David Dewar MacNicol

Dr. D.D. MacNicol

P.S. Many thanks also for the generous gift of the four samples which you are sending via Phil Hadley!

ALDRICH
Chemists Helping Chemists in Research and Industry
aldrich chemical company, inc.

Dr. Alfred Bader
Chairman

February 17, 1989

Dr. David MacNicol
Department of Chemistry
Glasgow University
Glasgow G12 8QQ, Scotland

Dear Dr. MacNicol:

As you know, we so enjoyed our visit with you in December.

We have now had a chance to work through that very interesting collection of research samples which you gave me, and I am really delighted by the varied and interesting structures.

There are ten compounds, beautiful in color and also of interest, where I cannot be certain exactly what the structures are because some shorthand notations were used. Enclosed please find the structures as we see them on the labels, with the corresponding numbers on the bottles. Where you know what the structures are, could you please just clarify them on the sheet and return it to me.

We already look forward to being back in Glasgow later this year.

Best personal regards.

Sincerely,

Alfred Bader

AB:mmh

Enclosure

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

¶ Small amounts of related products were also isolated.
# Dimethyl formamide.

checked by infrared and Raman spectroscopy, and by ^1H, ^{13}C

Dr. Alfred Bader
Chairman

January 3, 1989

Dr. David MacNicol
Department of Chemistry
Glasgow University
Glasgow, Scotland

Dear Dr. MacNicol:

Our visit with you on December the 6th was really the high point of our visit to Scotland.

This, of course, for many reasons: Your great personal kindness, your help in allowing us to purchase the collection of old research samples, and most importantly, your telling us about the exciting new synthetic possibilities through being able to offer compounds like thiophosgene as a MacNicol inclusion compound. We very much look forward to working with you in this field and hope that you will allow us to market a number of the inclusion compounds and to advertise them widely. In time, we also hope that you might find it possible to publish a review article of this chemistry in our Aldrichimica Acta. Perhaps the apt name, "The Crystal Bottle" should be registered in the Trade Marks Office. In the meantime, you will have received the check for £ 1000 and the spectra libraries which Philip Hadley has sent to you from Gillingham.

This was our first visit to Glasgow, but we will try to make certain that it is not our last, and I already look forward to visiting with you again next year. In the meantime, please keep in mind to let me know whenever you have a number of undergraduate students working with you, as Isabel and I would like to help them through our personal fund set up with the Royal Society of Chemistry.

Many thanks for all your help, and all good wishes for the new year.

Sincerely,

Alfred Bader
AB:mmh
cc: Dr. Stephen Branca
Mr. Philip Hadley

SIGMA-ALDRICH

P.O. Box 355 Milwaukee Wisconsin 53201 USA Telephone (414) 273-3850 Cable Aldrichem TWX 910-262-3052 Telex 26-843

† Small amounts of related products were also isolated.
# Dimethyl formamide.

checked by infrared and Raman spectroscopy, and by ^{1}H, ^{13}C

File

Reprinted from Nature, Vol. 332, No. 6159, pp. 59-61, 3 March 1988

New and unexpected reactivity of saturated fluorocarbons

David D. MacNicol & Colin D. Robertson

Department of Chemistry, University of Glasgow,
Glasgow G12 8QQ, UK

The carbon–fluorine bond, noted for its strength[1], has been of chemical interest[2] even before Moissan's discovery of elemental fluorine. The most electronegative element, fluorine is uniquely capable of replacing all the hydrogen atoms in every hydrocarbon system[3], yielding materials such as polytetrafluoroethylene renowned for their combination of rare physical properties, outstanding chemical inertness and high thermal stability[4], with significant modern technological applications[5,6]. Reaction of saturated fluorocarbons requires extreme conditions[1], for example treatment with metals at ~500 °C[7] or high-energy irradiation[8]. Following an observation by MacNicol and McGregor (unpublished data) of an unprecedented reactivity under mild conditions we now report specifically the efficient transformation of perfluorodecalin (1) to known host molecules by arenethiolate nucleophiles in dipolar aprotic solvent. This chemistry is not only of fundamental mechanistic interest, but also creates new synthetic possibilities in various fields. For example, preparing novel hosts for inclusion chemistry[9], a subject attracting much current attention.

Typical results for the reaction of (1), used as a commercial mixture of *cis* and *trans* isomers, with the sodium salts (2a–c) at ambient and higher temperatures, mostly performed in 1,3-dimethylimidazolidin-2-one (DMEU), are summarized in Table 1. The isolated products, octakis(phenylthio)naphthalene (3a) and octakis(*m*-tolylthio)naphthalene (3b) had a melting point at 205–206 °C and 156–157 °C respectively (both as unsolvated forms)[10,11]; these materials also had spectroscopic properties (infrared, ^{1}H NMR, ^{13}C NMR, and mass spectrometry) identical with those of authentic materials prepared from octafluoronaphthalene, the structures of which have been unambiguously established by single-crystal X-ray crystallography[10,11]. Compound (3c), octakis(*m*-methoxyphenylthio)naphthalene had spectroscopic properties (infrared, ^{1}H NMR, ^{13}C NMR and mass spectrometry) fully in accord with its assigned structure, these being identical to those obtained from material prepared by the authors (unpublished data) from octafluoronaphthalene. For the reaction of (2a) with (1) at ambient temperature little reaction is observed as red coloration, after one month. The process is significantly accelerated by increasing the reaction temperature to ~60–70 °C without promoting by-product formation. Above this temperature replacement of arylthio substituents by hydrogen occurs, as indicated by ^{1}H NMR, ^{13}C NMR and mass spectrometry.

Table 1 Reactions of perfluorodecalin (1) with sulphur nucleophiles (2)

Reagent*†	Solvent	Temperature	Time	Product	Yield‡ (%)
(2a)	DMEU	ambient	several months	(3a)	17§‖
(2a)	DMEU	60–70 °C	10 days	(3a)	65
(2a)	DMF#	60–70 °C	10 days	(3a)	55
(2b)	DMEU	ambient	8 weeks	(3b)	20‖
(2c)	DMEU	ambient	several months	(3c)	5‖¶

* With respect to (1), 36 molar equivalents were employed.

† The salt was preformed and dried before use.

‡ Yields based on (1) and not optimized. A large quantity of diaryldisulphide was formed in each case. Also, fluoride was detected by ion-selective electrode.

§ Unreacted (1) has an unaltered *cis/trans* ratio, by ^{19}F NMR.

‖ Most of (1) recovered unreacted.

¶ Small amounts of related products were also isolated.

\# Dimethyl formamide.

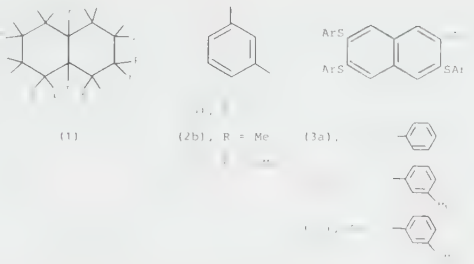


Fig. 1 Structure of the ~~un~~saturated fluorocarbon (1), the sodium salts (2a–c) and the isolated products (3a–b) of the reactions between (1) and (2a–c).

We are currently considering the mechanistic aspects of this novel reaction, the crucial first step of which may involve nucleophilic attack on carbon or fluorine, or single electron transfer (SET) to form a radical anion, by the sulphur nucleophile. In this case nucleophilic attack on a carbon atom of (1) appears unlikely[12], and from the literature it appears that either of the other two pathways is more probable. A number of recent studies have shown that halofluorocarbons[13], normally considered inert, readily undergo substitution through attack on chlorine and bromine by nucleophiles (for example, sulphur, oxygen and nitrogen-based systems). The halophilic attack has in different cases been assigned to operate by both anionic and SET mechanisms. The work here may extend these findings to fluorine in such systems as (1). The high electron affinity of saturated cyclic fluorocarbons suggests generation of a radical anion could take place by a different mechanism[14]. It is interesting to note that the decomposition of *n*-perfluoroalkane in Na/NH$_3$(l) by an unknown pathway[15] may be related despite the absence of reactivity of this class of molecule under present conditions.

In the new reaction nucleophilic attack on a tertiary fluorine (see below) would lead to the bridgehead carbanion $C_{10}F_{17}^-$ (4) directly, recently isolated as its tris(dimethylamino)sulphonium salt[16], which is expected to eliminate fluoride to give the symmetric perfluorobicyclic olefin $C_{10}F_{16}$ (5) with no vinylic fluorine; equally SET would lead to a radical anion of (1), $C_{10}F_{18}^{-\bullet}$, capable of eliminating fluoride to give a tertiary carbon radical which could then gain an electron to form (4) and hence (5) or alternatively could combine with arenethiolate (2) to generate the thioether derivative[17] after losing an electron, nucleophilic attack on the sulphur of which again leads to (5) via (4)[18].

Consistent with the possible intermediacy of (5), which might be expected to lead rapidly to the observed products, we have subjected perfluorocyclohexene, a model system, to analogous reaction conditions using (2a) in DMEU. This reacted rapidly at ambient temperature to give a 96% yield of hexakis(phenylthio)benzene[19], $C_6(SPh)_6$, melting point at 185–186 °C, which was isolated as its 1,1,1-trichloroethane clathrate (host-guest ratio 1:2). Indeed, facile multiple substitution initiated by nucleophilic attack on the double bond in (5) has been demonstrated[20] and a recent paper[21] on the multisubstitution with concomitant introduction of further unsaturation of an acyclic perfluoro-olefin, under similar conditions of excess mercaptide, are in keeping with the above proposal. The importance of a tertiary centre in the molecule is indicated by the failure of perfluorocyclohexane and *n*-perfluorohexane to react under equivalent conditions, whereas perfluoro(methylcyclohexane) and perfluoroperhydrophenanthrene do.

In view of these findings it is important to eliminate the possibility of significant levels of impurities starting the process, especially olefinic residues or olefinic precursors, such as hydrogen-containing material. The starting material was rigorously checked by infrared and Raman spectroscopy, and by ^{1}H, ^{13}C and ^{19}F NMR, and all of these methods gave no evidence for the presence of hydrogen or unsaturation, while independent gas-liquid chromatographic analysis determined the content of (1) as 95.6% (the remainder being saturated fluorocarbons arising from fluorinolysis and rearrangement occurring during synthesis). In addition the reaction was repeated on a medical-grade sample of (1) which is necessarily supplied free of these highly toxic impurities.

Perfluorodecalins are used in conjugation with other perfluoro-emulsifying agents as artificial blood substitutes[22], where they have been found non-toxic. But the above findings may suggest perfluorochemicals cannot now be automatically considered indefinitely inert under all mild conditions, including biological systems, where electron donors and nucleophiles are active. Further work in this laboratory has now also shown that the 'inert' fluorocarbon (1) can be reacted with selenium and oxygen-based nucleophiles, extending the scope of this previously unsuspected reactivity; it is expected that future efforts will lead to functionalized fluoropolymers, potential applications of which have already been identified[23].

We thank the SERC for support (to C.D.R.), ISC Chemicals Ltd. (Bristol) for sample analysis, Dr P. L. Coe (University of Birmingham) for providing some samples, Dr A. Cooper (University of Glasgow) for the Raman spectra, and SERC Mass Spectrometry service (Swansea). The fluorocarbons discussed here are the subject of British Patent Application No. 8720773 (4 September 1987).

Received 2 November 1987; accepted 5 January 1988.

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Dr. Alfred Bader
Chairman

February 7, 1989

Prof. Charles J. W. Brooks
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Charles:

I am delighted to have your kind letter of January 24th and the reprint of that interesting paper on ferrocene diesters.

Isabel and I definitely plan to come to Glasgow again this year, particularly to visit with you, Dr. MacNicol and Prof. Overton. I don't recall that there has ever been a university where I had never been before where I was welcomed so kindly and learned so much chemistry. We were just sorry that we had so little time, and we would certainly like to plan to spend at least two days with you on our next trip. In all probability that will be late next November.

Best personal regards to you and your associates.

Sincerely,

Alfred Bader

AB:mmh

cc: Dr. Stephen Branca
Mr. Philip Hadley

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

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Professor C.J.W. BROOKS

Chemistry Department
The University
Glasgow G12 8QQ
TEL: 041-339 8855
EXT. ~~333~~ 4373/6583

Dr. Alfred R. Bader,
Chairman,
Aldrich Chemical Company, inc.,
P.O. Box 355,
Milwaukee,
Wisconsin 53201, U.S.A.

24th January, 1989

cc SB

Dear Alfred,

Many thanks for your kind letters of January 3 and 11. It was good to hear that you had gained a favourable impression of the Department. I was particularly pleased that you had the opportunity of assessing the excellent work of David MacNicol. His novel host molecules for the inclusion of toxic compounds certainly merit the support that you are kindly able to provide. I look forward to further developments in the family of "crystal bottles" and "crystal safes", among the general class of Aldrich Retinacular Bottles.

I much appreciate your interest in the reagents that I mentioned. In the case of ferroceneboronic acid, the main by-product of most preparations seems to be the 1,1'-diboronic acid. As far as I can discover, the latter has been very little studied, either for organic or analytical applications. My view that it merits some study was strengthened by the work (cf. attached photocopy) on analogous dicarboxylic acid derivatives as potentially useful liquid-crystal constituents. I believe the diboronic acid could be even more versatile.

The computer printout that you arranged for me came very soon after your visit. It confirmed my impression that there are very few available chiral gamma-dialdehydes or gamma-diketones. I shall renew my efforts to make some compounds of this type. Meanwhile, the list has proved interesting for other compounds, and I am grateful for your speedy help.

The news about the Fieser models is extremely welcome. I will keep in touch with Bob Smith, to ensure that we can place an early order as soon as the models are available.

We look forward to the pleasure of a visit by Isabel and yourself later in the year.

With best personal regards,

Yours sincerely,

Charles

Encl.

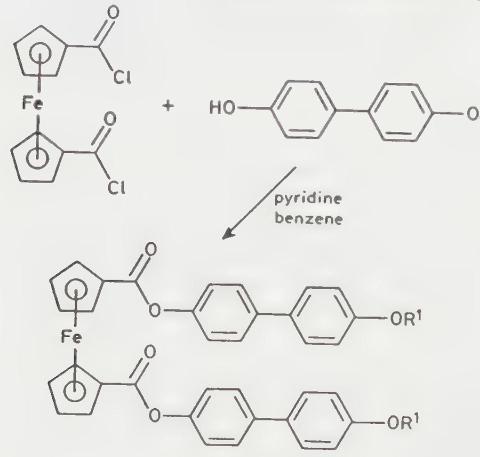
Novel Ferrocene Diesters with Liquid-Crystal Properties

J. Bhatt, B. M. Fung,* K. M. Nicholas,* and C.-D. Poon
Department of Chemistry, University of Oklahoma, Norman, Oklahoma, 73019, U.S.A.

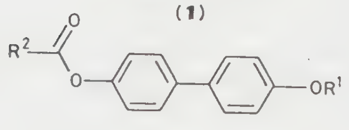
A series of bis-(4-alkoxy-4′-biphenyl)ferrocene 1,1′-diesters (**1**) has been synthesized; three compounds in this homologous series exhibit liquid-crystal phases.

Compounds which display liquid-crystal properties typically possess a rod- or disc-like core and long, flexible alkyl chains. Liquid-crystal compounds containing transition metals have recently attracted special attention because the transition-metal centre offers the appealing prospects of imparting unique optical, magnetic and electrical properties as well as enforcing various well-defined molecular geometries.[1,2]

We report the preparation and melting properties of a series of 1,1′-ferrocene diesters (**1**) which are characterized by the presence of two mesogenic groups joined by the kinked ferrocene core.[3,4] The synthesis of orange crystalline (**1a**—**h**) was accomplished by coupling readily available 1,1′-ferrocene diacid chloride[5] with various 4-alkoxy-4′-biphenols.[6] All these new compounds exhibited appropriate ^1H and ^{13}C n.m.r., i.r., and h.r.m.s. data.



(**1**)



(**2**)

a; R′ = C_5H_{11}, R^2 = Ph
b; R′ = C_6H_{13}, R^2 = Ph
c; R′ = C_6H_{13}, R^2 = $(C_5H_5)Fe(C_5H_4)$

The Table summarizes the phase transition temperatures and enthalpy data [determined by differential scanning calorimetry (D.S.C.)] for the various ferrocene esters. The compounds (**1b**, **c** and **h**) with $R^1 = C_5H_{11}$, C_6H_{13} and $C_{11}H_{23}$, respectively, were found to form monotropic mesophases over an 8—10 °C range. Under examination by a polarizing microscope the other compounds in the series were found to crystallize directly from the liquid phase upon cooling. Although the three liquid-crystal compounds display strikingly similar transition temperatures, preliminary polarizing-microscopic investigations suggest that (**1b**) and (**1c**) form a smectic C phase whereas (**1h**) exhibits a smectic A phase.[7] It is interesting to note that this change in phase behaviour follows the gap in which the C_7—C_{10} compounds do not form liquid crystals, apparently because the isotropic melts do not supercool to an adequate extent (see Table 1).

In order to probe the influence of the ferrocenyl unit on the observed liquid-crystal behaviour we prepared similarly the benzoate derivatives (**2a, 2b**) and the monoferrocenyl ester (**2c**). The benzoates were also found to form a mesophase [smectic C for (**2a**)] over a remarkably similar temperature range to that of the ferrocenyl diesters (**1b, c** and **h**). However, the presence of only one mesogenic group on ferrocene as in (**2c**) failed to induce liquid-crystal behaviour. It appears, therefore, that the ferrocene unit attenuates the liquid-crystal forming tendency of the alkoxybiphenyl group. Attachment of two such groups to the ferrocene core, however, is sufficient to restore liquid-crystal properties. At present, we do not know whether these molecules adopt a 'U' (shown) or the seemingly more likely 'S' geometry. Efforts are underway to address this point, to elucidate further the structural requirements for metallocene-based liquid crystals, and to extend their liquid-crystal range.

We are grateful for partial support provided by the University of Oklahoma Associates Fund.

Received, 12th February 1988; Com. 8/00527C

Table 1. Melting properties of ferrocene diesters.

Compound	R^1	M.p., °C	Transition temperatures, °C	ΔH (melting), kJ/mol
(**1a**)	C_4H_9	209	$I \xrightarrow{188} C$	69
(**1b**)	C_5H_{11}	167	$I \xrightarrow{140} S_c \xrightarrow{129} C$	51
(**1c**)	C_6H_{13}	181	$I \xrightarrow{139} S_c \xrightarrow{132} C$	56
(**1d**)	C_7H_{15}	180	$I \xrightarrow{146} C$	60
(**1e**)	C_8H_{17}	183	$I \xrightarrow{145} C$	52
(**1f**)	C_9H_{19}	169	$I \xrightarrow{141} C$	41
(**1g**)	$C_{10}H_{21}$	161	$I \xrightarrow{146} C$	64
(**1h**)	$C_{11}H_{23}$	163	$I \xrightarrow{141} S_a \xrightarrow{132} C$	56
(**2a**)	C_5H_{11}	142	$C \xrightarrow{129} S_c \xrightarrow{139} I^a$	27
(**2b**)	C_6H_{13}	127	$C \xrightarrow{127} S^b \xrightarrow{142} I^a$	35
(**2c**)	C_6H_{13}	153	$I \xrightarrow{80} C$	44

[a] Thermotropic transition. [b] Phase to be identified.

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Dr. Alfred Bader
Chairman

January 11, 1989

Prof. C. J. W. Brooks
Department of Chemistry
University of Glasgow
Glasgow G12 8QQ, Scotland

Dear Charles:

Thank you for your most helpful and kind letter of December 15th.

As you perhaps know, Isabel and I try to visit a great many chemistry departments around the world, but somehow had never managed to visit your university before. This was obviously a great mistake, because we so enjoyed our visit and learned a great deal.

Not just because of shades of the past and our being able to reminisce about our days at Harvard together. Nor because of the happy coincidence that Prof. Overton and I were students in the same high school in Vienna in the 1930's. The fact is simply that a great deal of very exciting chemistry is being done at your university. I could mention a number of developments, such as the really exciting work of Dr. MacNicol, but it wouldn't be really fair to mention just one or two. Suffice it to say, we already look to coming back to your university later this year, and we will then try to make certain that we have some time together.

Thank you for the details of the preparation of ferroceneboronic acid and of bis(4-methoxyphenyl)selenoxide, which will have our close attention.

I am happy to be able to tell you that since our visit with you, I have been able to finalize on a contract for the production of the Fieser models which should again become available in the middle of this year.

Best personal regards to you and your associates.

Sincerely,

Alfred Bader
AB:mmh
cc: Dr. Stephen Branca

P.O. Box 355, Milwaukee, Wisconsin 53201 USA, Telephone (414) 273-3850, Cable Aldrichem TWX 910-262-3052, Telex 26-843, FAX 414-273-4979

Professor C.J.W. BROOKS

Chemistry Department
The University
Glasgow G12 8QQ
TEL: 041-339 8855
EXT. 4373/6583

[illegible]

Dr. A.R. Bader,
President,
Aldrich Chemical Co., Inc.,
940 West Saint Paul Avenue,
Milwaukee,
Wisconsin 53233, U.S.A.

15th December, 1988

Type A

Dear Dr. Bader,

It was a pleasure to meet you again after so many years, and to meet your wife for the first time. Your visit provided a most welcome stimulus, and a reminder of the continuing vitality of organic chemistry. These were valuable antidotes to the ill-effects produced by the major building alterations that have been disrupting our work for two years.

As agreed, I am attaching references and brief notes on the preparation of ferroceneboronic acid and of bis(4-methoxyphenyl)selenoxide. I have not undertaken preparations of either compound. Fortunately I was able to obtain some ferroceneboronic acid from Koch-Light - in fact it was their final stock, I believe.

I will collect together the miscellaneous compounds that are available for disposal, so that they will be ready for inspection when you visit us in 1989. Your interest in these is much appreciated.

It was very good to learn that the Fieser models will soon be obtainable once again. We have not found any satisfactory substitutes, though the Darling models are of complementary value, in that their greater flexibility allows quite strained molecules to be represented.

Many thanks for your kind gifts of prints from your Collection. I am also grateful for the Classified Catalog Supplement and the catalogue of the Gilman Collection. I have already found one interesting compound in the latter - the boronic acid, S82,179-9 - which I shall order.

We very much look forward to seeing you and Mrs. Bader again next year. It would be particularly pleasant if you could spare a little more time, so that we might arrange an informal evening meal and meeting with colleagues.

With best regards,

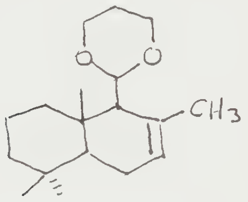
Yours sincerely,

Charles

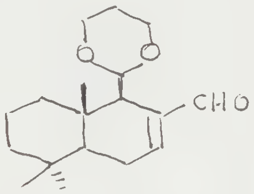
Encls.

bis(4-Methoxyphenyl)selenoxide

This is of interest to me because of its reported efficacy in the oxidation of the allylic hydrogens in the drimenal acetal (I) to the polygodial acetal (II). This is the best method for partial synthesis of polygodial, and this mild, selective reagent should be valuable for related aldehydes. [See attached reprint.]



I



II

Preparation of the reagent is described by F. Ogura *et al.*, *Bull. Chem. Soc. Japan*, 1982, *55*, 641. See also F. Ogura *et al.*, *Chem. Lett.*, 1983, 1833 for applications.

Partial synthesis of (−)-polygodial

Manuel J Cortés, Iván Razmilic, Jorge R Sierra and José López
Facultad de Quimica, Pontificia Universidad Católica de Chile, Casilla 6177, Santiago, Chile

Polygodial (I) is a sesquiterpene of plant origin[1] which recently has also been isolated from four porostome nudibranchs.[2] This product, as well as some related dialdehydes, exhibits a number of interesting biological properties, including a marked antifeedant activity against insects[3] and fish[4] and it tastes very hot[5] to humans.

In view of their varied activities, sustained interest has been shown in syntheses of such compound.[6-7] However, all the syntheses lead to racemic polygodial and racemic congeners modified on the ring B.

Now, the first partial synthesis is reported of natural polygodial starting with (−)-drimenol (II), which is readily available as a main component of the bark of *Drimus winteri.*[8]

Oxidation of (−)-drimenol with pyridinium chlorochromate in dichloromethane gave the aldehyde (III) in 75.4 per cent yield as an oil, ν_{max} (neat) 2710, 1718 and 1670 cm^{-1}; n.m.r. spectrum ($CDCl_3$)(p.p.m.) δ9.81 (1H,*d*, *J* 5Hz,CHO), 5.75 (1H,m, H(7)), 1.64 (3H,*sbr*, vinylic CH_3), 1.05 (3H,*s*, 10-CH_3), 0.98 (3H,*s*, 4-CH_3), 0.95 (3H,*s*, 4-CH_3). The aldehyde group of compound (III) was protected with trimethylene glycol to yield a ketoacetal (IV) (88.5 per cent yield), m p 66-7°C; $[\alpha]_D^{26}$ +10.30° ($CHCl_3$ *c* 3.4); $\nu_{max.}$ (KBr)1670 and 1140 cm^{-1}; n.m.r. spectrum ($CDCl_3$) (p.p.m.) δ5.58 (1H,*m*, H(7)), 4.74 (1H,*s*, H(II), *J* 0, dihedral angle 90°), 3.40-4.36 (4H,*m*, overlapping signals of acetal CH_2), 1.90 (3H, *sbr*, vinylic CH_3), 0.90 (3H,*s*, 10-CH_3), 0.88 (6H,*s*, 2x4-CH_3); *m/z* 278 (M^+), 109,87.

Oxidation of the ketoacetal (IV) with catalytic amounts of selenium dioxide and *bis* (4-methoxyphenyl) selenoxide as co-oxidant, according to Ogura's method,[9] gave the aldehyde (V) in 45 per cent yield with m p 155-7°C; $[\alpha]_D^{26}$ + 66.80° ($CHCl_3$ *c* 0.95); ν_{max} (KBr) 1680, 1620 and 1140 cm^{-1}; n.m.r. spectrum ($CDCl_3$) (p.p.m.) δ9.94 (1H,*s*,CHO), 6.92 (1H,*m*, H(7)), 4.92 (1H,*s*, H(11)), 4.30-3.58 (4H,*m*, overlapping signals of acetal CH_2), 0.96 (3H,*s*, 10-CH_3), 0.92 (6H,*s*, 2x4CH_3). Finally, quantitative deprotection of compound (V) with *p*-toluensulphonic acid in acetone gave polygodial (I), m p 55-7°C. The infrared, n.m.r. and mass spectra and the optical rotation were identical with those of an authentic sample of natural polygodial.

The overall yield of (−)-polygodial in this partial synthesis from (−)-drimenol was 30 per cent.

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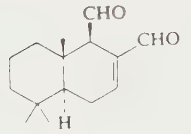
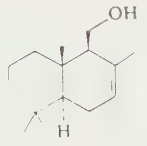


(I)



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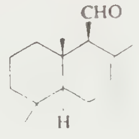

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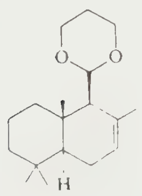

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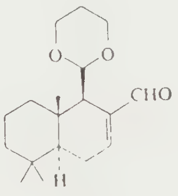

(V)

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1533 -4

Stannous chloride reduction of nitroalkenes to oximes in acetone

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The authors have been investigating the potential utility of nitroalkenes as precursors to a variety of useful synthetic intermediates such as nitroalkanes, *N*-substituted hydroxylamines,[2] amines,[3] ketones,[4] chromenes,[5] α-substituted oximes[6] and ketones.[7] Prompted by a recent report[8] on the use of stannous chloride as a reducing agent in non-acidic and non-aqueous media for conversion of nitroarenes into amines, the authors applied the methodology to reduction of nitroalkenes. Now, it is reported that stannous chloride readily reduces the αβ-unsaturated nitroalkenes to the corresponding oximes in acetone at room temperature (see Table). This appears to be the only instance of oxime formation to appear in otherwise extensive literature on the use of stannous chloride as a reducing agent.[9]

Ferroceneboronic acid and 1,1'-diboronic acid

Both of these are useful as analytical reagents, and potentially also as protecting groups. [See attached reprints.]

There are many preparations in the literature, most of which give moderate and variable yields, with the diboronic acid generally as a minor product. For example, Pauson's group[1] used the reaction of $BCl_3/Al/AlCl_3/MeI$ with ferrocene and obtained 22-54% of the mono- and 2-15% of the diboronic acid. Other references are cited below.[2-5] In a preliminary note, all the corresponding dihalides were described, and a yield of 92% was reported for ferrocenyldiiodoborane.[6]

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CHROM. 18 728

Note

Analytical separation and characterisation of 1,2- and 1,3-diols as their cyclic ferroceneboronate derivatives

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Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland (U.K.)
(Received April 14th, 1986)

Cyclic boronates are well established as effective derivatives for the selective analysis of proximally bifunctional substrates by gas–liquid chromatography (GLC) and its combination with mass spectrometry (MS). Minor drawbacks of these derivatives include their susceptibility to solvolysis, and the conformational mobility of the boronate rings: these factors can detract from the quality of gas chromatographic peaks. We considered that the use of ferroceneboronates might be advantageous. For example, electron donation to boron by the ferrocenyl group should tend to improve resistance to solvolysis. In addition, the resultant partial C–B double-bond character, together with the bulk and rigidity of the ferrocenyl ($C_{10}H_9Fe$)* moiety, would be expected to restrict the conformational range of the cyclic boronate grouping. A further aspect of interest in ferroceneboronates is their potential value for multiple-element detection in GLC combined with plasma emission spectrometry —a technique already successfully applied to the analysis of butaneboronates of catechols[1]. In this respect, ferroceneboronic acid[2] is an example of a selective "multi-element taggant"[3] for bifunctional substrates.

We have found that ferroceneboronic acid readily yields cyclic derivatives with suitably constituted 1,2-diols, 1,3-diols, and related substrates. In general, the derivatives afford sharp and symmetrical peaks in GLC, while their mass spectra (under electron impact ionisation) show abundant molecular ions, which are usually the base peaks and represent a high proportion of the total ion current. These features are exemplified in the small group of compounds (listed in Table I) that are included in this preliminary note. In general, the derivatives were prepared by adding a solution of ferroceneboronic acid (1.1 molar proportions) in dry pyridine to the substrate (100 μg) (also in dry pyridine) and heating at 70°C for 30 min. After removal of solvent in a stream of nitrogen, the residue was dissolved in ethyl acetate (100 μl) for analysis by GLC and GLC–MS. Under these conditions, there appeared to be substantially complete formation of derivatives, most of which remained stable in ethyl acetate solution for several days, as judged by GLC.

Fig. 1 shows the GLC separation of ferroceneboronates of one of the chiral forms of butane-2,3-diol and its *meso*-stereomer. The quality of the peaks is similar to that of trimethylsilyl ethers (and of cyclic di-*tert*.-butylsilylene derivatives[4]), and

---

* Denoted as Fc where appropriate.

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TABLE I

KOVÁTS RETENTION INDICES (*I*) AND MASS SPECTROMETRIC DATA (22 eV) FOR FERROCENEBORONATE DERIVATIVES OF 1,2- AND 1,3-DIOLS AND OF AN AMINO-ALCOHOL

Parent compound	$I_{OV\text{-}1}$	*Temperature (°C)*	$M^{+\cdot}$ *(base peak)**	$[Fc\text{–}B{=}O]^{+\cdot}$ *m/z 212, % abundance*
(*R,R*)-Butane-2,3-diol	1790	150	284	30
meso-Butane-2,3-diol	1840	150	284	43
Cyclohexane-*cis*-1,2-diol	2165	190	310	33
Cyclohexane-*trans*-1,2-diol	2170	190	310	82
Mephenesin**	2680	225	376	17
Chlorphenesin***	2815	225	396	20
2,2,4-Trimethylpentane-1,3-diol	2150	190	340	22
2-(2-Pyridyl)propane-1,3-diol	2610	225	347	60
(−)-2-Aminobutan-1-ol	1975	175	283	39

* Mass spectra normalised above *m/z* 40.
** 3-(2-Methylphenoxy)propane-1,2-diol.
*** 3-(4-Chlorophenoxy)propane-1,2-diol.

the separation of isomers very satisfactory. Their mass spectra are virtually identical: that of the (*R,R*)-isomer (Fig. 2) shows the characteristic isotopic pattern of the molecular ion (reflecting mainly the contributions of ^{10}B, ^{54}Fe and ^{57}Fe as relatively abundant minor isotopes). The other main ions represent the reagent moieties $[FcB(OH)_2]^{+\cdot}$ (*m/z* 230) and $[FcBO]^{+\cdot}$ (*m/z* 212). Strong metastable ions were observed in respect of the losses of C_4H_8O from the molecular ions (*m/z* 284 → *m/z* 212).

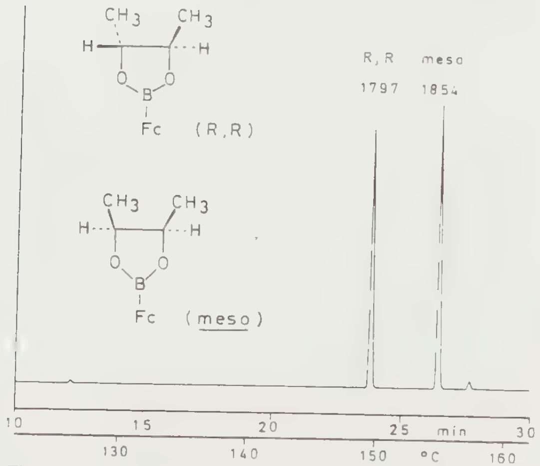

Fig. 1. GLC separation of ferroceneboronates of (*R,R*)- and *meso*-butane-2,3-diol. Column, SE-54 fused silica, 25 m × 0.32 mm I.D.; column temperature, programmed from 80°C (2 min) to 110°C (1 min) at 30°C/min, and then at 2°C/min to 200°C; helium flow-rate, 3 ml/min; flame ionization detection. Samples (*ca.* 1 μg) were introduced via a Grob-type injector operated in split mode (50:1). Fc = Ferrocenyl.

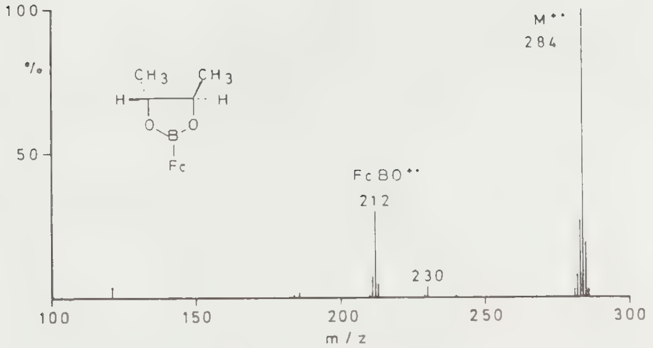

Fig. 2. Mass spectrum (22 eV) of (*R,R*)-butane-2,3-diol ferroceneboronate. The LKB 9000 gas chromatograph–mass spectrometer was operated with a DB-1 fused-silica column, 60 m × 0.32 mm I.D.; column temperature, 150°C; helium carrier gas and "make-up" gas flow-rates, 7 and 25 ml/min, respectively; accelerating voltage, 3.5 kV; source and separator temperatures, 270°C; trap current, 60 μA; filament current, 4 A. The sample (*ca.* 1 μg) was introduced via a falling-needle injector. Fc = Ferrocenyl.

The mass increment of 194 attending formation of cyclic ferroceneboronates makes these derivatives particularly suitable for the selective analysis of diols of low molecular mass. However, larger molecules can be effectively studied, as illustrated by the GLC trace in Fig. 3 for ferroceneboronates of mephenesin (a topical antifungal agent) and chlorphenesin (a muscle relaxant). The corresponding mass spectra are again dominated by molecular ions, but show a few fragment ions containing substrate moieties. In the case of mephenesin ferroceneboronate (Fig. 4), major ions derived from the reagent include *m/z* 230, 213 ($[FcBOH]^+$), 212, 186 ($FcH^{+\cdot}$) and 121 ($[C_5H_5Fe]^+$). The most prominent of the ions retaining a part of the substrate molecule occurs at *m/z* 239 ($[C_{12}H_{12}BFeO]^+$, *i.e.* $[FcBOC_2H_3]^+$).

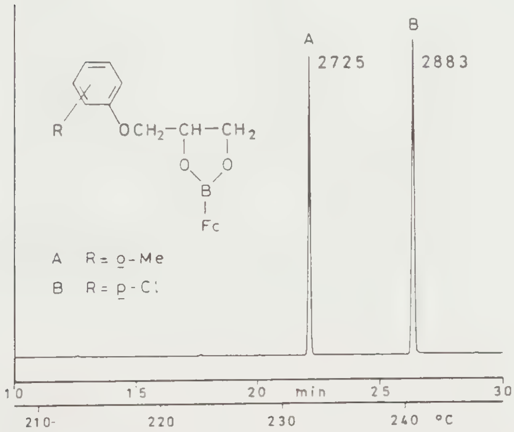

Fig. 3. GLC separation of the ferroceneboronates of mephenesin (A) and chlorphenesin (B). Column as in Fig. 1; column temperature, programmed from 80°C (2 min) to 200°C (2 min) at 30°C/min, and then at 2°C/min to 260°C; other conditions as in Fig. 1. Fc = Ferrocenyl.

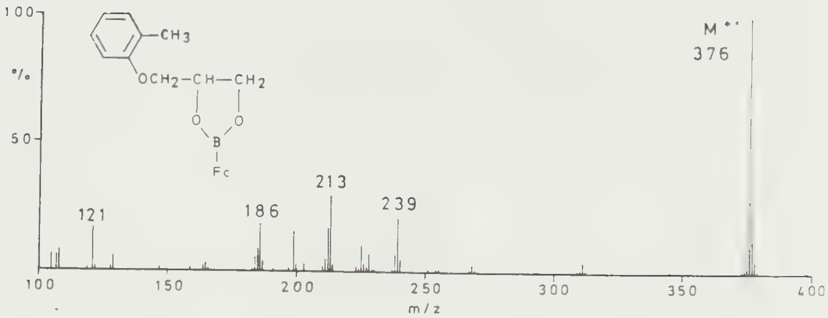

Fig. 4. Mass spectrum (22 eV) of the ferroceneboronate of mephenesin. Conditions as in Fig. 2, except for column temperature (225°C). Fc = Ferrocenyl.

Satisfactory ferroceneboronates have been obtained from a wide range of other substrates. Salient data for cyclohexane-*cis*- and -*trans*-1,2-diols (for which the derivatives were just separable by GLC), for two 1,3-diols, and for 2-aminobutan-1-ol, are given in Table I. Our results indicate that ferroceneboronic acid resembles other boronic acids in its ready reaction with most of the bifunctional substrate types for which the latter reagents are well established. The cyclic ferroceneboronates of diols also possess some special features: for example, (i) the GLC peaks are, in most instances, strikingly sharp and symmetrical; (ii) the electron impact mass spectra show strongly preponderant molecular ions (with characteristic isotopic patterns) and relatively few other substrate-derived ions; (iii) the high abundances of molecular ions, and of reagent-derived ions (*e.g.* *m*/*z* 230, 212, 186 and 121) provide potentially sensitive means of detecting, respectively, the individual boronates or the boronates as a group, by selected ion monitoring. Further aspects of the applications of ferroceneboronic acid to the analysis and characterisation of bifunctional substrates are described in a fuller paper[5].

ACKNOWLEDGEMENT

We thank Michael Hollywood for technical assistance.

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CHROMSYMP. 1151

CYCLIC FERROCENEBORONATES AS DERIVATIVES FOR THE GAS CHROMATOGRAPHIC SEPARATION AND CHARACTERISATION OF DIOLS AND RELATED COMPOUNDS

CHARLES J. W. BROOKS* and W. JOHN COLE
Chemistry Department, University of Glasgow, Glasgow G12 8QQ (U.K.)

SUMMARY

Cyclic ferroceneboronate derivatives have been obtained readily from a range of 1,2- and 1,3-diols, and from one α- and two β-hydroxyacids, three β-hydroxyamines and one γ-hydroxyamine. The pinacol ester was stable to thin-layer chromatography. The ferroceneboronates of diols afforded particularly good gas chromatographic peaks, and are well suited to the analyses of diols of low molecular weight. The mass spectra (electron impact ionisation) are dominated by molecular ions, which are usually the base peaks and carry a high proportion of the total ion current. Characteristic isotope patterns reflect the presence of ^{10}B, ^{54}Fe and ^{57}Fe as minor natural isotopes of appreciable abundances.

INTRODUCTION

The value of boronic acids, as selective reagents for compounds with suitably proximal pairs of functional groups, was originally established in the carbohydrate field[1]. The versatility of these reagents has been exploited in many ways, *e.g.* in separations based on affinity chromatography. Applications to the analysis and characterisation of diols (and other substrates) by gas chromatography–mass spectrometry (GC–MS) have been developed extensively, since their inception in our laboratory[2]. Among the limitations of cyclic boronates as derivatives are their susceptibility to solvolysis, and their conformational mobility. These factors contribute, for example, to "tailing" and/or broadening of gas chromatographic peaks. We considered that cyclic ferroceneboronates might have improved properties. Thus electron donation by the ferrocenyl group should tend to make nucleophilic attack on the boron atom less facile than in benzeneboronates or alkaneboronates. (At the same time, it was recognised that under acidic conditions ferroceneboronates would probably be less stable than the analogous benzeneboronates, by analogy with the reagents themselves[4,5].) In addition, the partial double bond character of the carbon–boron bond, together with the bulkiness and rigidity of the ferrocenyl moiety, would be expected to restrict the range of energetically favourable conformations of the cyclic boronate group.

A further feature of ferroceneboronates is their potential value for element-

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specific detection. In mass spectrometry, the characteristic isotope distributions of iron and boron give rise to distinctive clusters of peaks. Furthermore, in atomic emission spectrometry, the detection of iron in addition to boron and carbon permits increased specificity. The technique of microwave-induced plasma emission spectrometry has already been succesfully applied to detect both carbon and boron in the gas chromatographic analysis of catechol butaneboronates[6]. The presence of an iron atom in the ferroceneboronates gives scope for further specificity of detection, complementing the selectivity inherent in cyclic boronate formation. Reagents containing two or more elements with distinctive atomic emission properties have been applied to the gas chromatographic analysis of alcohols and amines in conjunction with a microwave-sustained helium emission detector[7].

Ferroceneboronic acid[5,8–10] is thus worthy of investigation as an inexpensive reagent for bifunctional substrates, and one which provides selectivity in both the formation and the detection of derivatives. The mass spectral fragmentations (under electron impact) of ferroceneboronic acid, triferrocenylboroxine and mono-methyl ferroceneboronate have been studied by Post *et al.*[11]. Prior to our preliminary note[12], cyclic ferroceneboronates apparently had not been applied in gas chromatographic separations. The present report concerns GC–MS of the derivatives obtained from a further twenty-one representative substrates.

EXPERIMENTAL

Solvents and reagents

Benzeneboronic acid was obtained from BDH (Poole, U.K.) and *n*-butaneboronic acid from Aldrich (Gillingham, Dorset, U.K.). Ferroceneboronic acid was purchased from Koch-Light (A. & J. Beveridge, Edinburgh, U.K.) and N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) from Pierce and Warriner (Chester, U.K.). Ethyl acetate (Nanograde) was obtained from Mallinckrodt (St. Louis, MO, U.S.A.). Pyridine (AnalaR grade, BDH) was dried over potassium hydroxide pellets and redistilled prior to use.

Diols and related reference compounds

The numbering sequence for the reference compounds is that depicted in Fig. 1, all chiral compounds being racemic except for compound 18 which was the (+)-enantiomer. Compounds 1, 10, 18 and 19 were purchased from Koch-Light. Compound 2 was obtained from Fisons Scientific Apparatus (Loughborough, U.K.) and compounds 3, 4, 11 and 17 from Aldrich. Compounds 5 and 8 were available from earlier work[13,14], while compounds 6, 12 and 13 were synthetic samples (donated by the late Prof. J. D. Loudon). Compound 7 was purchased from Alfred Bader Chemicals (Aldrich) and compound 9 from Frinton Lab. (South Vineland, NJ, U.S.A.). Compound 14 (SQ 22,928) was a gift from E.R. Squibb & Sons (Princeton, NJ, U.S.A.); compounds 15 and 16 were synthetic samples (C.J.W.B.). Compound 20 was purchased from Sigma (St. Louis, MO, U.S.A.) and compound 21 from CalBiochem (San Diego, CA, U.S.A.).

Gas–liquid chromatography

Packed column gas–liquid chromatography (GLC) was carried out with a Per-

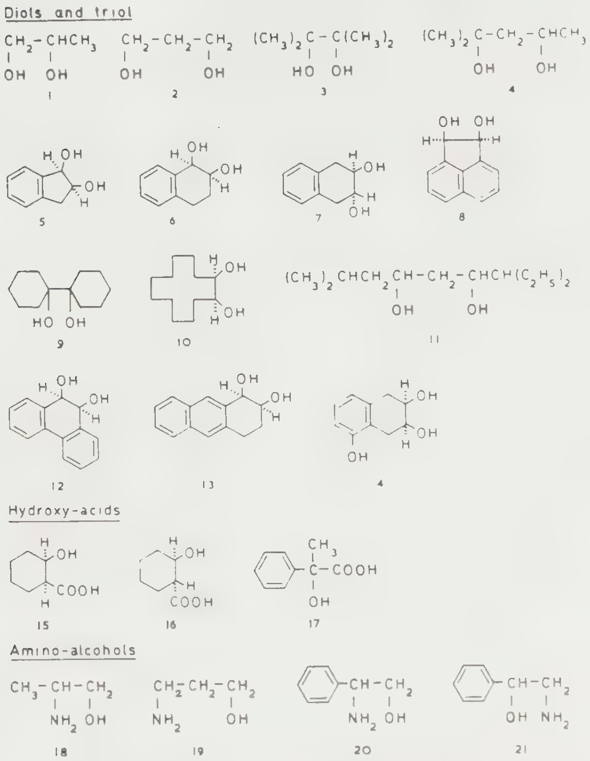


Fig. 1. Structures of diols, a triol, hydroxyacids and aminoalcohols. 1 = Propane-1,2-diol; 2 = propane-1,3-diol; 3 = 2,3-dimethylbutane-2,3-diol (pinacol); 4 = 2-methylpentane-2,4-diol; 5 = indane-*cis*-1,2-diol; 6 = 1,2,3,4-tetrahydronaphthalene-*cis*-1,2-diol; 7 = 1,2,3,4-tetrahydronaphthalene-*trans*-2,3-diol; 8 = acenaphthene-*cis*-1,2-diol; 9 = bicyclohexyl-1,1′-diol; 10 = cyclododecane-*cis*-1,2-diol; 11 = 7-ethyl-2-methylnonane-4,6-diol; 12 = 9,10-dihydrophenanthrene-*cis*-9,10-diol; 13 = 1,2,3,4-tetrahydroanthracene-*cis*-1,2-diol; 14 = 5-hydroxy-1,2,3,4-tetrahydronaphthalene-*cis*-2,3-diol; 15 = *cis*-2-hydroxycyclohexanecarboxylic acid; 16 = *trans*-2-hydroxycyclohexanecarboxylic acid; 17 = α-hydroxy-α-phenylpropionic acid (atrolactic acid); 18 = L-2-aminopropan-1-ol; 19 = 3-aminopropan-1-ol; 20 = 2-amino-2-phenylethanol (phenylglycinol); 21 = β-hydroxy-β-phenylethylamine.

kin-Elmer (Beaconsfield, U.K.) F-11 gas chromatograph equipped with a silanized glass column (1.8 m × 4 mm I.D.) packed with 1% OV-1 coated on Gas-Chrom Q, 100–120 mesh (Phase Separations, Queensferry, U.K.). The column was heated at a variety of temperatures depicted in Table I, and the nitrogen carrier gas flow-rate was 40 ml/min. Open-tubular GLC was performed with a Hewlett-Packard (Winnersh, U.K.) 5880A gas chromatograph equipped with SE-54 (GC^2, Northwich, Chester, U.K.) and CP Sil 5 CB (Chrompack U.K., London, U.K.) fused-silica capillary columns (25 m × 0.32 mm I.D.) and Grob-type injectors operated in split mode (50:1). The columns were temperature programmed under a variety of conditions (see Figs. 2, 3, 5, 7 and 8 for details); the helium carrier gas flow-rates were 3 ml/min for both columns. Hydrogen flame-ionisation detectors were employed in each instrument.

TABLE I

KOVÁTS RETENTION INDICES (*I*) AND MASS SPECTROMETRIC DATA (20 eV) FOR FERROCENEBORONATE DERIVATIVES OF DIOLS, A TRIOL, HYDROXY-ACIDS AND AMINO-ALCOHOLS

Compound	*I* *(OV-1)*	*Temperature (°C)*	$M^{+\cdot}$ *(base peak)**	*m/z for other principal ions (intensities relative to base peak in parentheses)***								
1	1770	150	270	272(5) 211(4) 128(4)	271(18) 187(9) 122(4)	269(38) 186(45) 121(20)	268(10) 185(4)	267(4) 184(6)	230(3) 160(4)	226(3) 147(4)	213(12) 134(4)	212(46) 129(5)
2	1860	150	270	272(4) 187(10)	271(20) 186(61)	269(38) 185(4)	268(12) 184(7)	267(4) 147(4)	213(6) 128(6)	212(36) 121(23)	211(11)	210(4)
3	1880	150	312	314(4) 212(27)	313(22) 211(8)	311(26) 186(4)	310(10) 121(3)	254(6)	231(5)	230(34)	229(9)	213(5)
4	1885	150	312	314(4) 186(4)	313(22) 121(3)	311(28)	310(9)	230(16)	229(4)	213(8)	212(37)	211(10)
5	2450	205	344	346(4) 187(6)	345(25) 186(28)	343(35) 184(5)	342(12) 132(6)	341(4) 121(15)	213(11) 116(9)	212(71) 115(7)	211(22) 104(9)	210(6)
6	2600	220	358	360(6) 210(5)	359(29) 121(4)	357(30)	356(11)	355(4)	230(4)	213(9)	212(50)	211(14)
7	2690	220	358	360(5) 121(5)	359(27)	357(28)	356(8)	355(4)	213(6)	212(40)	211(10)	186(11)
8	2865	245	380	382(5) 187(4)	381(25) 186(15)	379(25) 168(5)	378(12) 152(6)	377(5) 140(4)	213(8) 121(10)	212(50)	211(16)	210(5)
9	2645	225	392	394(6) 211(4)	393(29) 186(8)	391(35) 162(4)	390(12) 121(3)	389(4)	231(5)	230(38)	229(11)	212(14)
10	2855	230	394	396(6) 211(6)	395(29) 187(7)	393(33) 186(11)	392(15) 184(6)	391(7) 128(4)	230(10) 122(4)	229(4) 121(16)	213(4)	212(21)

11	2450	215	396	398(7)	397(30)	395(37)	394(14)	393(5)	230(6)	212(8)	186(14)	121(5)
12	3125	245	406(72)	408(7)	407(25)	405(25)	404(11)	403(5)	213(9)	212(100)	211(26)	210(10)
				209(5)	195(4)	194(15)	187(5)	186(29)	184(4)	178(14)	166(4)	165(6)
				121(13)								
13	3225	255	408	410(9)	409(32)	407(38)	406(15)	405(7)	213(10)	212(62)	211(20)	210(4)
				196(7)	186(15)	180(5)	179(4)	178(4)	154(6)	121(9)		
14***	2840	220	446	448(11)	447(38)	445(37)	444(15)	443(7)	381(6)	212(9)	186(8)	121(3)
15	2480	220	338	340(5)	339(24)	337(30)	336(10)	335(4)	230(4)	213(7)	212(46)	211(12)
				210(4)	186(17)	121(6)						
16	2480	220	338	340(6)	339(25)	337(33)	336(11)	335(5)	294(2)	230(8)	229(4)	213(13)
				212(77)	211(23)	210(7)	187(4)	186(42)	184(4)	121(15)		
17	2450	220	360(30)	361(8)	359(12)	358(4)	213(15)	212(100)	211(30)	210(9)	187(6)	186(39)
				184(4)	121(16)	104(11)						
18	1840	150	269	270(19)	268(29)	267(11)	226(8)	213(4)	212(23)	211(7)	188(7)	186(43)
				184(5)	121(16)							
19	1920	150	269	270(19)	268(30)	267(9)	213(4)	212(26)	211(8)	186(12)	121(6)	
20	2480	205	331	333(6)	332(25)	330(29)	329(11)	328(4)	226(8)	225(4)	212(13)	211(4)
				186(16)	134(4)	121(7)	104(4)					
21	2490	205	331	333(5)	332(24)	330(33)	329(12)	328(5)	213(9)	212(59)	211(17)	210(5)
				187(7)	186(43)	184(5)	121(16)	119(5)	118(4)	106(7)	105(8)	104(3)

* Except for compounds 12 and 17. Mass spectra normalised above m/z 40.

** Only ions in excess of 3% abundance have been tabulated (above m/z 100) unless of specific importance.

*** 5-TMS ether.

GC–MS

GC–MS was carried out with an LKB 9000 instrument fitted with a DB-1 fused-silica capillary column, 60 m × 0.32 mm I.D. (J. & W. Scientific, Rancho Cordova, CA, U.S.A.) and a falling needle injector[15]. The helium carrier and make-up gas flow-rates were 7 ml/min (measured at ambient temperature) and 25 ml/min, respectively. Mass spectra (22 eV) were recorded under electron-impact conditions; accelerating voltage, 3.5 kV; filament current, 4 A; trap current, 60 μA; source and separator temperatures, 270°C.

Preparation of derivatives

Boronate esters. Ferrocene-, benzene-, or *n*-butaneboronic acid (1.1 mol proportion) in dry pyridine was added to the substrate (100 μg) in dry pyridine (20 μl) and the mixture heated at 70°C for 30 min. After removal of solvent under nitrogen, the residue was redissolved in ethyl acetate (100 μl) and analysed by GLC and GC–MS.

Trimethylsilylation. Compound 14 was converted to its ferroceneboronate derivative as above. The dried product was treated with BSTFA (20 μl), heated at 70°C for 5 min and immediately evaporated to dryness to yield the 2,3-ferroceneboronate 5-trimethylsilyl ether. The derivative was redissolved in ethyl acetate (100 μl) for GLC and GC–MS analyses.

Isolation of pinacol ferroceneboronate

Pinacol (118 mg) and ferroceneboronic acid (210 mg), dissolved in dry pyridine (10 ml), were heated at 80°C for 3 h. After removal of solvent under nitrogen, the product was crystallised from absolute ethanol as yellow prisms, yield 134 mg. A portion (30 mg) was recrystallised from absolute ethanol and dried *in vacuo* at 40°C; m.p., 118–121°C; U.V. (cyclohexane): λ_{max} 442 nm, ε_{max} 135; 330 nm, ε_{max} *ca.* 110. Elemental analysis: calculated for $C_{16}H_{21}BFeO_2$, C = 61.52, H = 6.78%; found C = 61.34, H = 6.53%.

Nuclear magnetic resonance

Nuclear magnetic resonance (NMR) ^{1}H spectra of C^2HCl_3 solutions were recorded with a Perkin-Elmer R32 90 MHz instrument using tetramethylsilane as reference standard. Pinacol ferroceneboronate exhibited sharp singlets at δ 1.32 (12H; 4-CH_3), δ 4.15 (5H; C_5H_5) and a broad signal at δ 4.40 (4H; C_5H_4). For comparison, pinacol shows a sharp singlet at δ 1.21 (12H; 4-CH_3) and a broad signal at δ 1.92 (2H; 2-OH).

RESULTS AND DISCUSSION

Preparation and properties of cyclic ferroceneboronates

Most of the derivatives of the compounds studied (Fig. 1) were prepared on an analytical scale from 100-μg amounts of samples, by heating at 70°C for 30 min in dry pyridine with a 1:1 molar proportion of ferroceneboronic acid. The reactions with diols proceeded in high yield, while hydroxyacids and hydroxyamines appeared to give less complete derivative formation. In addition, the diol ferroceneboronates remained stable in ethyl acetate solution for at least several days, whereas the analogous derivatives from hydroxyamines, in particular, were much less stable on storage. The ferroceneboronate of the di-tertiary diol, pinacol, was readily obtained, and was stable to thin-layer chromatography (TLC): a crystalline sample was prepared and characterised by its m.p., analysis and spectroscopic data. The yellow colour imparted by the reagent moiety allowed visual detection during TLC.

Attempts to obtain derivatives from a catechol (methyl 2,3-dihydroxybenzoate) and from two substituted salicylic acids (3,6-dimethyl and 4,6-dimethyl) produced no indication, by GLC, of peaks attributable to the expected products.

It is obvious that the scope for ferroceneboronates in gas chromatography is limited by the relatively high mass increment (194) contributed by the reagent. Even so, diols of molecular mass exceeding 200 (Table I, Nos. 12, 13) afforded very satisfactory derivatives. For diols of low molecular mass, ferroceneboronates are particularly useful, because their selective formation yields peaks in GLC that are eluted much later than likely impurities in the substrate. The potential utility of ferroceneboronates in quantitative analysis is supported by the linearity of flame ionisation detector response we have observed for the derivatives of pinacol and acenaphthene-*cis*-1,2-diol over the range 10–100 ng.

Gas chromatographic data and salient features of the mass spectra of the 21 compounds studied are summarised in Table I. Structures of substrates are in Fig. 1. The mass spectra were characterised in almost all instances by molecular ions that formed the base peaks (above m/z 40). The principal fragment ions were due to reagent moieties, notably $FcBO^{+\cdot}$ (m/z 212), $[FcB(OH)_2]^{+\cdot}$ (m/z 230) and $FcH^{+\cdot}$ (m/z 186), where the symbol Fc denotes ferrocenyl. Fragment ions derived from substrates were of minor occurrence. Further details of the data in Table I are discussed below.

GC–MS of diol ferroceneboronates

The sharpness of gas chromatographic peaks obtained for diol ferroceneboronates was observed to be as good as, or better than, this quality in the analogous benzene- and *n*-butane-boronates. The gas chromatogram (Fig. 2) of the three derivatives of indane-*cis*-1,2-diol (No. 5) illustrates this feature: the retention index increment from benzene- to ferroceneboronate in this case is 533.

Cyclic boronates have proved effective in the gas chromatographic separation of many isomeric and stereoisomeric diols. The ferroceneboronates of propane-1,2-diol and propane-1,3-diol (Nos. 1 and 2) were very well separated ($\Delta I = 90$), although there were many common features in the mass spectra. However, the retention times of the ferroceneboronates of the structural isomers, pinacol and 2-methylpentane-2,4-diol (Nos. 3 and 4), proved to be closely similar on the column used, and the peak separation ($\Delta I = 5$) was only just adequate to distinguish the isomers (Fig. 3). A further difference was discerned in the mass spectra (Fig. 4): even though these were dominated by the molecular ions and the reagent-derived ions mentioned above, the pinacol derivative yielded an ion at m/z 254 (M − 58, attributable to the loss of acetone in a four-centre rearrangement, as in Scheme 1) which was in extremely low abundance in the spectrum of the isomeric derivative.

A more typical separation, of positional isomers, is depicted in Fig. 5 for the ferroceneboronates of two tetrahydronaphthalenediols (Nos. 6 and 7). A very small degree of "tailing" of the peak from the 2,3-*trans* diol derivative could be seen, but

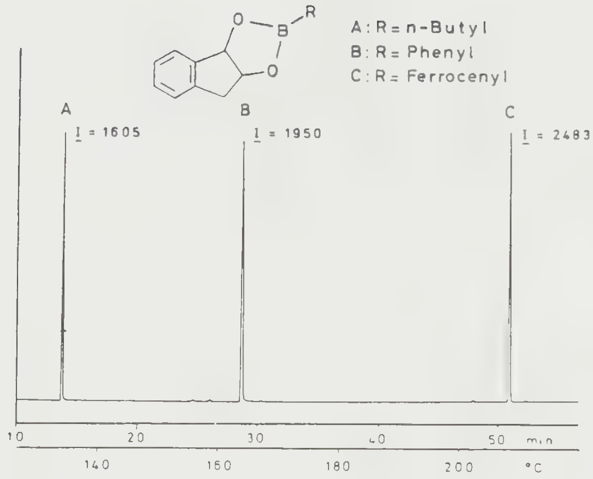


Fig. 2. Gas chromatographic separation of the *n*-butaneboronate (A), benzeneboronate (B) and ferroceneboronate (C) of indane-*cis*-1,2-diol. Column, SE-54 fused-silica capillary (25 m × 0.32 mm I.D.); column temperature, programmed from 80°C (2 min) to 115°C (1 min) at 30°C/min, and then at 2°C/min to 230°C; helium flow-rate, 3 ml/min.

the peak separation (ΔI = 90) was very large. The almost complete identity of the mass spectra (Table I) is in strong contrast with the markedly different fragmentations of the corresponding methaneboronates[16].

The remaining individual diols of various types (Nos. 8–13) each gave very satisfactory ferroceneboronate peaks in GLC. Limited interest attaches to the mass

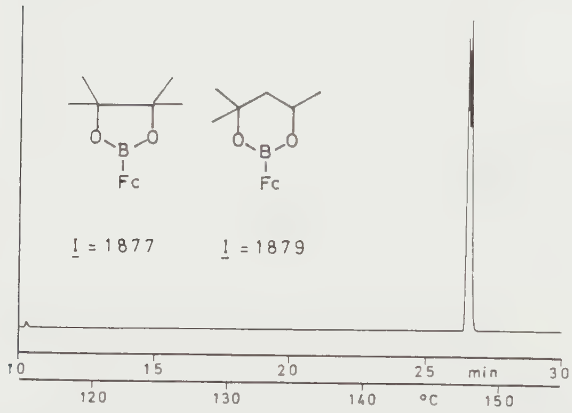


Fig. 3. Gas chromatographic separation of the ferroceneboronates of pinacol and 2-methylpentane-2,4-diol. Column, CP Sil5 CB fused-silica capillary (25 m × 0.32 mm I.D.); column temperature, programmed from 80°C (1 min) to 100°C (1 min) at 30 °C/min, and then at 2°C/min to 200 °C; helium flow-rate, 3 ml/min. Fc = Ferrocenyl.

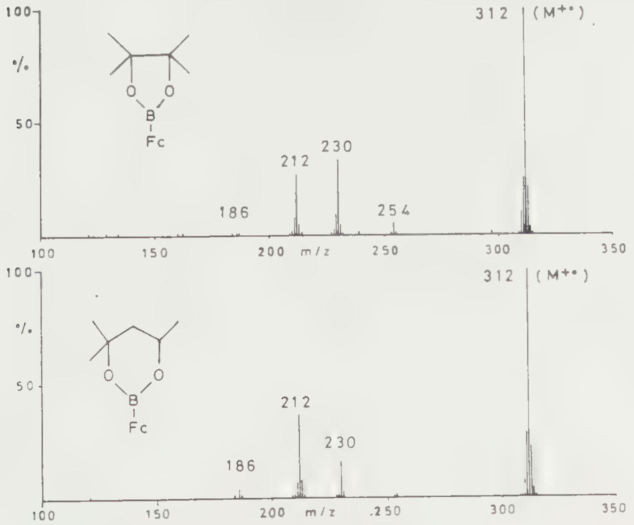
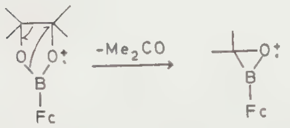


Fig. 4. Mass spectra (22 eV) of the ferroceneboronates of pinacol (top) and 2-methylpentane-2,4-diol (bottom). The LKB 9000 gas chromatograph-mass spectrometer was operated with a DB-1 fused-silica column, 60 m × 0.32 mm I.D.; column temperature, 150°C; helium carrier gas and "make-up"gas flow-rates, 7 ml/min and 25 ml/min, respectively; accelerating voltage, 3.5 kV; source and separator temperatures, 270°C; trap current, 60 μA; filament current 4 A. Samples (*ca.* 1 μg) were introduced via a falling-needle injector.



Scheme 1. A postulated mode of fragmentation of pinacol ferroceneboronate under electron impact.

spectra, in view of the preponderance of molecular and "reagent" ions. Hydrocarbon ions corresponding to [M$-$FcB(OH)$_2$] occurred in the spectra of the alicyclic diols Nos. 9 and 13, whereas the diols Nos. 8 and 12 yielded respectively the acenaphthylene and phenanthrene ions by loss of FcBO$_2$ from the molecular ion. No significant hydrocarbon ions were observed from the ferroceneboronates of the cyclododecanediol (No. 10) or the acyclic 1,3-diol (No. 11).

One of the features of cyclic boronate esters in their general stability towards mild acylating or silylating reagents. The tetralin triol (No. 14), when treated successively with ferroceneboronic acid and BSTFA, readily yielded the boronate trimethylsilyl ether: this showed good gas chromatographic behaviour, while its mass spectrum included one unusual ion at *m/z* 381, ([M$-$65]$^+$) ascribed to the loss of one cyclopentadienyl radical, as indicated by the observation of a metastable ion at *m/z* 325.5.

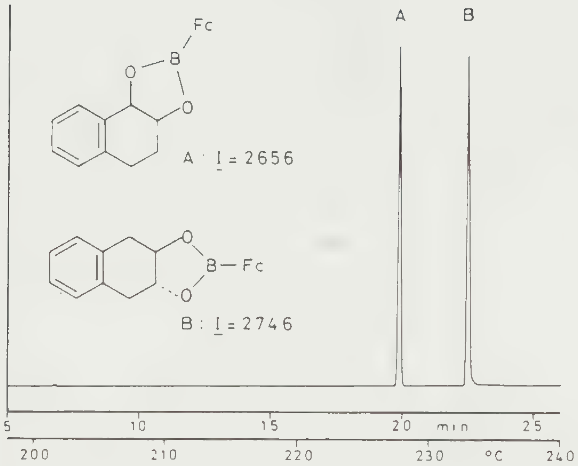


Fig. 5. Gas chromatographic separation of the ferroceneboronates of 1,2,3,4-tetrahydronaphthalene-*cis*-1,2-diol and 1,2,3,4-tetrahydronaphthalene-*trans*-2,3-diol. Column, SE-54 fused-silica capillary (25 m × 0.32 mm I.D.); column temperature, programmed from 80°C (1 min) to 200°C (1 min) at 30°C/min, and then at 2°C/min to 260°C; helium flow-rate, 3 ml/min.

GC–MS of hydroxyacid ferroceneboronates

The isomeric β-hydroxyacids, *cis*- and *trans*-2-hydroxycyclohexanecarboxylic acid, were known to be distinguishable by GLC of their butaneboronates[17], and were particularly well separated as their di-*tert*.-butylsilylene derivatives[18]. However, the ferroceneboronates gave identical retention values and closely similar mass spectra (Table I, Nos. 15 and 16). The mass spectrum of the *trans*-isomer (Fig. 6) is illustrative. Apart from the major "reagent" ions and the molecular ion, the prominent ion at *m/z* 121 is notable: an ion at this position occurs in many of the spectra, and in the case of the pinacol derivative has been shown to be $C_5H_5Fe^+$ (see further below). Fig. 6 also indicates a minor fragment ion (*m/z* 294) representing $[M-CO_2]^{+\cdot}$, as shown by a weak metastable ion at *m/z* 255.7. Corresponding ions were much more prominent in the mass spectra of di-*tert*.-butylsilylene derivatives[18].

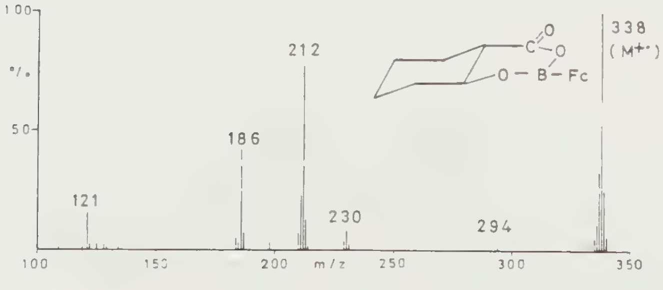


Fig. 6. Mass spectrum (22 eV) of the ferroceneboronate of *trans*-2-hydroxycyclohexanecarboxylic acid. Conditions as in Fig. 4, except that column temperature was 220°C.

The α-hydroxyacid atrolactic acid (No. 17) yielded a ferroceneboronate, the mass spectrum of which was unremarkable except for an ion at m/z 104, presumably a styrene fragment.

GC–MS of amino-alcohol ferroceneboronates

Two isomeric pairs of aminoalcohols were examined. The derivatives of 2- and 3-aminopropanol (Nos. 18 and 19) were widely separated by GLC ($\Delta I = 80$ on OV-1). A comparable separation ($\Delta I = 86$) was found on a capillary column (SE-54) as shown in Fig. 7. Moreover, the six-membered derivative had the longer retention time, in conformity with the order for the corresponding diol derivatives (Nos. 1 and 2): the degree of separation was also comparable. The mass spectra of the isomeric aminopropanol ferroceneboronates were very similar, except for the higher abundance of m/z 226 ($[\text{FcBO{=}CH}_2]^{+\cdot}$) for No. 18.

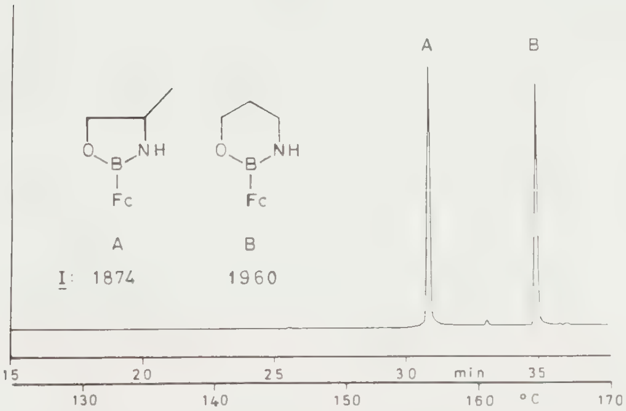


Fig. 7. Gas chromatographic separation of the ferroceneboronates of (L)-2-aminopropan-1-ol and 3-aminopropan-1-ol. Column, SE-54 fused-silica capillary (25 m × 0.32 mm I.D.); column temperature, programmed from 80°C (1 min) to 100°C (1 min) at 30°C/min, and then at 2°C/min to 220°C; helium flow-rate, 3 ml/min.

The isomeric phenylethanolamines (Nos. 20 and 21) were satisfactorily separated by GLC of their ferroceneboronates, as shown in Fig. 8. It was noted that No. 21 was the more difficult substrate to derivatise, requiring the use of freshly dried and distilled pyridine for satisfactory results. Nevertheless, ferroceneboronates could be obtained for these "biogenic" amines. The mass spectra (Fig. 9) were largely similar, but only the phenylglycinol derivative (No. 20) (upper spectrum) showed a prominent ion at m/z 226 ($[\text{FcBO{=}CH}_2]^{+\cdot}$) attributable to the loss of the PhCH=NH moiety (Scheme 2): a metastable ion was present at m/z 154.5 (calculated, 154.3).

Further observations on mass spectra of ferroceneboronates

As emphasised above, major ions in all spectra were the molecular ions and ions derived from ferroceneboronic acid. All the spectra contained a strong ion at m/z 212 ($\text{FcBO}^{+\cdot}$) together with a metastable ion indicating its direct formation from

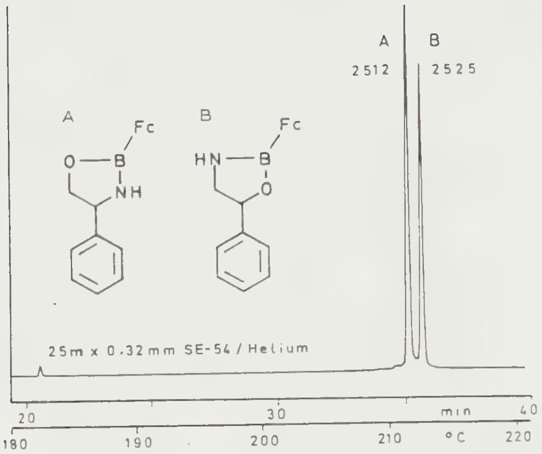


Fig. 8. Gas chromatographic separation of the ferroceneboronates of 2-amino-2-phenylethanol (A) and β-hydroxy-β-phenylethylamine (B). Column, SE-54 fused-silica capillary (25 m × 0.32 mm I.D.); column temperature, programmed from 80°C (1 min) to 150°C (1 min) at 30°C/min, and then at 2°C/min to 230°C; helium flow-rate, 3 ml/min.

the molecular ion. For the derivatives of compounds Nos. 3, 4, 9, 10, 11 and 14, metastable ions also reflected the direct formation of the ion of *m/z* 230 ($[FcB(OH)_2]^{+\cdot}$) from $M^{+\cdot}$. The electron-impact "dehydration"[11] represented by *m/z* 230 → 212 gave rise to metastable ions in the spectra from compounds Nos. 3, 4, 6, 9, 10, 11 and 14.

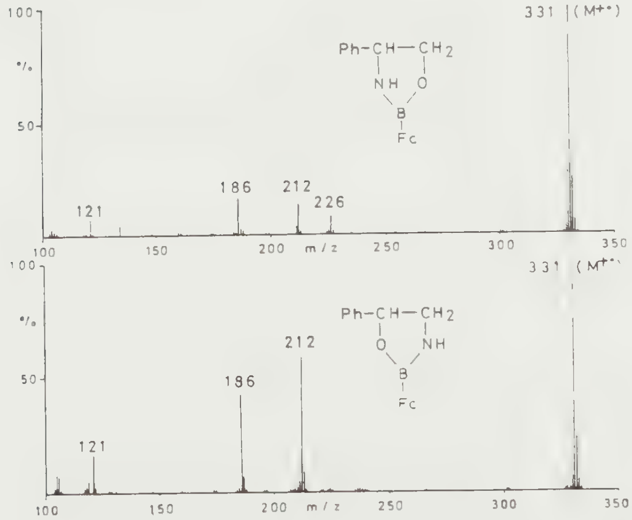
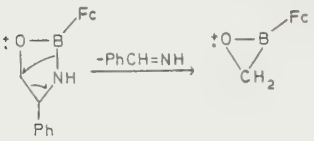


Fig. 9. Mass spectra (22 eV) of the ferroceneboronates of 2-amino-2-phenylethanol (top) and β-hydroxy-β-phenylethylamine (bottom). Conditions as in Fig. 4, except that column temperature was 205°C.



Scheme 2. A postulated mode of fragmentation of phenylglycinol ferroceneboronate under electron impact.

The elemental compositions of some major ions – all but one retaining the ferrocenyl group – that were common to the mass spectra of the ferroceneboronates were determined for the analytically pure derivative of pinacol by accurate mass measurements (Table II). The principal ions had the expected compositions[11], and the ion of *m/z* 121 corresponded to $[C_5H_5Fe]^+$. Table II also shows data verifying the composition of the ion of *m/z* 226, observed in the spectrum of phenylglycinol ferroceneboronate (see above) as $FcBOCH_2$.

TABLE II

ELEMENTAL COMPOSITION OF IONS IN THE MASS SPECTRA OF PINACOL FERROCENEBORONATE AND PHENYLGLYCINOL FERROCENEBORONATE

*Measured mass, m/z**	*Ion composition*	*Ion type*
Pinacol ferroceneborate		
312.0982	$C_{16}H_{21}{}^{11}B^{56}FeO_2$	$M^{+\cdot}$
311.1018	$C_{16}H_{21}{}^{10}B^{56}FeO_2$	$M^{+\cdot}$ (^{10}B)
310.1024	$C_{10}H_{21}{}^{11}B^{54}FeO_2$	$M^{+\cdot}$ (^{54}Fe)
230.0198	$C_{10}H_{11}{}^{11}B^{56}FeO_2$	$Fc^{11}B(OH)_2{}^{+\cdot}$
229.0249	$C_{10}H_{11}{}^{10}B^{56}FeO_2$	$Fc^{10}B(OH)_2{}^{+\cdot}$
212.0080	$C_{10}H_9{}^{11}B^{56}FeO$	$Fc^{11}B{=}O^{+\cdot}$
211.0139	$C_{10}H_9{}^{10}B^{56}FeO$	$Fc^{10}B{=}O^{+\cdot}$
186.0127	$C_{10}H_{10}{}^{56}Fe$	$FcH^{+\cdot}$
120.9743	$C_5H_5{}^{56}Fe$	Fe^{3+}  2− ?
Phenylglycinol ferroceneborate		
226.0258	$C_{11}H_{11}{}^{11}B^{56}FeO$	$Fc^{11}BOCH_2{}^{+\cdot}$
225.0288	$C_{11}H_{11}{}^{10}B^{56}FeO$	$Fc^{10}BOCH_2{}^{+\cdot}$

* Recorded on an MS 902 mass spectrometer by Mr. A. Ritchie. Deviations of measured from calculated mass values ranged from −0.0016 to +0.0012.

CONCLUSIONS

Cyclic ferroceneboronates are readily obtained from suitably constituted 1,2- and 1,3-diols: the yields are generally high and the excellent quality of the gas chromatographic peaks makes the derivatives suitable for qualitative (and potentially for quantitative) analyses, including separations of isomers. Analogous derivatives are obtainable from non-phenolic hydroxyacids and from 1,2- and 1,3-aminoalkanols, but the reactions are often less satisfactory than those with diols, and the ferroceneboronates are less stable on storage.

The stability of the ferrocenyl group largely inhibits fragmentation modes within the substrate moiety. The molecular ions are in many instances the base peaks, and the only other major ions are those of composition FeBO [frequently accompanied by $FcB(OH)_2$], and, ferrocene, with isotopic congeners. This pattern in the mass spectra of ferroceneboronates, comprising two or more common ions together with one ion distinctive for the molecular weight of the substrate, would be useful for selected ion monitoring of diols. The high increments in mass, and in gas chromatographic retention time, attending ferroceneboronate formation are advantageous for the analysis of diols of low molecular weight. Short-chain alkanediols are of clinical and forensic interest by reason of their occurrence in blood at elevated concentrations in some conditions, notably in cases of alcoholism and of generally high intake of ethanol[19,20]. Analysis and characterisation of free diols by GC–MS has been reported[21]. Among the selective reagents already applied to the derivatisation of serum diols for analysis by GLC are benzeneboronic acid[22] and butaneboronic acid[23] for ethylene glycol, and *p*-bromobenzeneboronic acid[24] for a number of diols, including the diastereomeric butane-2,3-diols. The last of these reagents is particularly useful, in affording selectivity and sensitivity of detection by electron-capture GLC, together with convenient retention characteristics of the cyclic esters. Ferroceneboronic acid is likely to be of complementary value. For example, the cyclic ferroceneboronate of ethylene glycol has retention index 1740 on the OV-1 phase[25], and would yield a peak that is eluted in a region well clear of solvent and of many other likely contaminants in serum extracts: In GC–MS of this derivative, the characteristic isotopic cluster of molecular ions (principal ion, mass m/z 256, as base peak) provides a basis for sensitive and specific determination.

The ferrocenyl moiety appears to have little effect on the susceptibility of the cyclic boronate group to solvolysis. However, the sharpness of the gas chromatographic peaks may be ascribed to the conformational control imposed by the bulk and rigidity of the ferrocenyl group.

It is concluded that ferroceneboronic acid is a useful addition to the armoury of boronic acid reagents, with particularly promising applications to diols of molecular masses below about 300 daltons. The use of the reagent for the selective detection of terpenoid diols, in extracts of plants and of plant tissue cultures[26], is being explored.

ACKNOWLEDGEMENTS

We thank Mr. M. Hollywood for valuable experimental assistance. We are also indebted to Miss L. Williamson, for preliminary studies of two ferroceneboronates; to Mr. A. Ritchie (Departmental Mass Spectrometry Unit) for accurate mass measurements; and to Mrs. F. Lawrie and Mr. R. Sharp for U.V. and NMR data, respectively. The LKB 9000 instrument was provided under SRC grants B/SR/2398 and B/SR/8471.

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Dr. Alfred Bader
Chairman

January 3, 1989

Prof. Karl Overton
Department of Chemistry
Glasgow University
Glasgow, Scotland

Dear Prof. Overton:

You must have realized how happily surprised we were to be able to meet a student from the Speleum!

The next time we come to Glasgow, either in the summer or next December, we must set a little time aside to be able to chat about the years at the same school, before 1938.

In the meantime, please do remember that if you have any old research samples, even if they are only in very small quantities, we would be delighted to purchase them for our Library of Rare Chemicals.

Best personal regards.

Sincerely,

Alfred Bader

AB:mmh

cc: Mr. Philip Hadley

SIGMA-ALDRICH

P.O. Box 355 Milwaukee Wisconsin 53201 USA Telephone (414) 273-3850 Cable Aldrichem TWX 910-262-3052 Telex 26-843

Dr. Alfred Bader
Chairman

January 3, 1989

Prof. C. J. W. Brooks
Department of Chemistry
Glasgow University
Glasgow, Scotland

Dear Prof. Brooks:

You will have realized how delighted I was to be able to chat with you again after these many years. When next we come to Glasgow, hopefully next year, I trust that we will have some time to compare notes about what each of us has done since 1949.

Thank you for suggesting that we offer that dimethoxyselenium compound as a new oxidizing agent, and I look forward to your sending me the reference.

Similarly, we look forward to receiving the details of the preparation of ferrocenediboronic acid.

I trust that you have received the computer printout of our gamma diketones, ferrocenes and dialdehydes which my secretary has sent to you.

Please also remember that we would be most interested in purchasing whatever research samples you no longer require, for our Library of Rare Chemicals.

Best personal regards.

Sincerely,

Alfred Bader
AB:mmh
cc: Dr. Stephen Branca
Mr. Philip Hadley

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Dr. Alfred Bader
Chairman

January 3, 1989

Prof. G. Kirby
Department of Chemistry
Glasgow University
Glasgow, Scotland

Dear Prof. Kirby:

Thank you for your kind reception of Isabel, Philip Hadley and me on December 5th and 6th.

We were delighted to be able to tell you that the Fieser models will again be available next summer.

Thank you for suggesting that we offer deuterated DIBAL, and we will consider it carefully.

We were shaken by your telling us that your large collection of research samples was just thrown out. Please do remind your associates that we would be most interested in purchasing whatever research samples are no longer required, for our Library of Rare Chemicals.

Best personal regards.

Sincerely,

Alfred Bader
AB:mmh
cc: Dr. Clinton Lane
    Dr. Joseph Porwoll
    Mr. Philip Hadley

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UNIVERSITY OF
STRATHCLYDE

Please quote ref.
WCB/AE

13 August 1993

Dr Alfred Bader
Alfred Bader Fine Arts
Aspen Hotel Suite 622
924 East Juneau Avenue
MILWAUKEE
Wisconsin
USA 53202

Dear Dr Bader

Thank you for your letter and for your advice, which I shall follow forthwith.

I am delighted to receive the copy of Hofmann's signature, which we will certainly try to use.

Yours sincerely

(Prof W C Bowman)

DEPARTMENT OF PHYSIOLOGY & PHARMACOLOGY
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Glasgow G1 1XW
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ALFRED BADER FINE ARTS

DR. ALFRED BADER · ESTABLISHED 1961

August 4, 1993

Prof. W. C. Bowman, Head
Department of Physiology & Pharmacology
University of Strathclyde
Royal College
204 George Street
Glasgow G1 1XW, Scotland

Dear Prof. Bowman:

I am sorry that a long trip to Europe has delayed my responding to your inquire of June 25th.

Unfortunately, I do not know where you could obtain a color photograph of A. W. Hofmann. The best source for information might be Mrs. McCabe, the Library of the Royal Institution in Albemarle Street in London and Dr. Arnold Thackray, Director, Beckman Center for the History of Chemistry, 3401 Walnut Street, Philadelphia, PA 19104 6228, U.S.A.

By chance, I have just acquired an original announcement of a course given by Prof. Hofmann bearing his signature, and I enclose a Xerox copy thereof. Possibly, you might like to use the signature, and if the Xerox copy is not good enough, then I could loan you the original document.

All good wishes.

Sincerely,

Enclosure

By Appointment Only
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Dr M Duwiejua

Dr N Pyne

Bet 7/9

UNIVERSITY OF
STRATHCLYDE

Please quote ref. **WCB/SM**

25 June 1993

Dr Alfred Bader
Suite 622
924 East Junean Avenue
Milwaukee
Wisconsin 53202
USA

Dear Dr Bader

A.W. Hofmann

You probably know the story about my colleague, John Stenlake, who designed Wellcome's neuromuscular blocking drug called atracurium. Atracurium was designed to break down by Hofmann elimination, and this self-destruct mechanism means that so long as the patient is wet and warm, he or she will get rid of the drug even if kidney and liver function are totally lost. The University gets over £2 million per annum from royalties on atracurium.

John Stenlake is now retiring, and I have been given the task of both organising a "Festschrift" for him, and of producing a booklet giving his story and reproducing the various lectures of the symposium. I hope that the front cover of the book will have, in the background, the various bits and pieces of a curare-collector's regalia (feathers, gourds, blowpipe etc), and also pictures of the important individuals in the "curare field". They will be Claude Bernard, Hofmann (hopefully), Harold Griffith (who first used curare in a surgical operation), and John Stenlake. The whole will be in colour. I already have coloured pictures of three of these individuals, but I cannot find one of Hofmann. I wonder if you can help? Can you direct me to a source from which I could obtain a coloured photograph of an oil-painting of A.W. Hofmann? I would be most grateful.

Yours sincerely

WC Bowman

(Professor W C Bowman
Head of Department)

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