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Supplementary Material for

Crystal Structure of Burgess Inner Salts and their Hydrolyzed Ammonium Sulfaminates

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Computational Details

Final minimum energy geometries from G3MP2 calculations were:

Triethylamine C6H15N

0 1

N	0.000000	0.000000	0.008903
C	0.331579	1.352712	-0.447319
C	-0.294241	2.423878	0.433118
C	-1.337272	-0.389201	-0.447319
C	-1.952019	-1.466759	0.433118
C	1.005694	-0.963512	-0.447319
C	2.246260	-0.957119	0.433118
H	1.418407	1.465779	-0.408027
H	0.041491	1.498564	-1.504671
H	0.038956	2.292207	1.464631
H	-1.385857	2.378681	0.421266
H	0.000000	3.420951	0.091228
H	-1.978605	0.495487	-0.408027
H	-1.318540	-0.713349	-1.504671
H	-2.004587	-1.112367	1.464631
H	-1.367070	-2.389528	0.421266
H	-2.962631	-1.710476	0.091228
H	1.277049	-0.785214	-1.504671
H	0.560198	-1.961266	-0.408027
H	1.965631	-1.179840	1.464631
H	2.752927	0.010847	0.421266
H	2.962631	-1.710476	0.091228

Trimethylamine C3H9N

0 1

N	0.000000	0.000000	0.395836
C	0.000000	1.378031	-0.065174
C	-1.193410	-0.689015	-0.065174
C	1.193410	-0.689015	-0.065174
H	0.886156	1.891057	0.317663
H	0.000000	1.461164	-1.167896
H	-2.080781	-0.178094	0.317663
H	-1.265405	-0.730582	-1.167896
H	1.194625	-1.712962	0.317663
H	2.080781	-0.178094	0.317663
H	1.265405	-0.730582	-1.167896
H	-1.194625	-1.712962	0.317663
H	-0.886156	1.891057	0.317663

N-carbomethoxy sulfonylimine C2H3NO4S

0 1

S	1.528894	-0.046670	-0.057624
O	1.573439	1.304724	-0.613284
O	2.740041	-0.822243	0.177302
O	-1.116518	1.092644	0.778337
O	-1.938112	-0.728678	-0.319333
N	0.213793	-0.781499	0.240987
C	-0.982413	0.002664	0.261115
C	-3.243653	-0.108307	-0.280333
H	-3.220645	0.847513	-0.803864
H	-3.553226	0.046326	0.753355
H	-3.899397	-0.814327	-0.783288

Burgess Reagent trimethylamine analog C5H12N2O4

0 1

S	-0.569070	-1.007139	0.144369
O	-0.566073	-1.009668	1.608702
O	-1.452453	-1.904632	-0.593966
O	1.566521	1.117062	0.491000
O	2.946166	-0.436700	-0.412069
N	-1.656133	0.709950	-0.128551
N	0.768213	-0.803015	-0.616890
C	-3.075884	0.302746	-0.208175
C	-1.206902	1.321866	-1.396076
C	-1.450472	1.626316	1.016869
C	1.730521	0.047707	-0.094220
C	4.035047	0.415049	-0.013857
H	-3.334509	-0.245427	0.698061
H	-3.694967	1.201156	-0.291140
H	-1.252626	0.568861	-2.183732
H	-1.861367	2.164005	-1.641046
H	-0.387598	1.839496	1.102594
H	-1.799080	1.131771	1.921919
H	4.039021	0.549450	1.068326
H	3.956333	1.389574	-0.497891
H	4.930975	-0.107768	-0.341822
H	-2.022640	2.542153	0.838201
H	-0.181521	1.665429	-1.270640
H	-3.218603	-0.343619	-1.071244

Burgess Reagent C8H18N2O4S

0 1

S	0.053960	-1.182725	0.176582
O	0.041962	-1.705804	-1.191212
O	0.809492	-1.877784	1.218004
O	-2.167337	0.212977	-1.289976
O	-3.380183	-0.028657	0.612732
N	1.358709	0.386039	-0.104802
N	-1.231296	-0.524971	0.761407
C	2.332095	-0.038448	-1.159190
C	3.203977	-1.231274	-0.803475
C	2.052071	0.676481	1.184407
C	1.155582	1.251663	2.266960
C	0.524198	1.519652	-0.600952
C	1.284844	2.803698	-0.890123
C	-2.236061	-0.097654	-0.107556
C	-4.508474	0.435804	-0.146666
H	1.735303	-0.260470	-2.045671
H	2.966900	0.826953	-1.373282
H	2.615287	-2.112050	-0.554451
H	3.887554	-1.016058	0.019374
H	3.811871	-1.464363	-1.682354
H	2.464241	-0.271453	1.526456
H	2.884492	1.354687	0.961190
H	0.290476	0.612769	2.447965
H	0.809388	2.260304	2.033103
H	1.739390	1.309205	3.189839
H	-0.241159	1.694220	0.154588
H	0.007539	1.151772	-1.489625
H	1.996532	2.698468	-1.710622
H	1.815123	3.180033	-0.012430
H	0.557783	3.564733	-1.186677
H	-4.712206	-0.235928	-0.981559
H	-4.327119	1.440348	-0.532441
H	-5.337517	0.437557	0.558246

Crystallographic Data Collection and Refinement

Table of crystal data 1, 2, 3b, 4, and 5.

	Burgess Reagent (OMe) **1**	Burgess Reagent (OEt) **2**
Mol. Form.	$C_8H_{18}N_2O_4S$	$C_9H_{20}N_2O_4S$
Formula weight	238.30	252.33
Temperature (K)	120	296
Wavelength (Å)	0.71073	0.71073
Crystal system	*monoclinic*	*monoclinic*
Space group	*P*21/n	*P*21
a (Å)	6.8066(5)	6.6023(9)
b (Å)	12.8754(9)	12.8477(17)
c (Å)	13.1397(9)	7.8470(10)
α(°)	90	90
β (°)	93.8074(8)	106.2153(16)
γ (°)	90	90
Volume (Å^3)	1148.99(14)	639.14(15)
Z	4	2
Density (g/cm^3)	1.378	1.311
Absorp. coefficient (mm^{-1})	0.280	0.256
F(000)	512	272
Crystal size (mm)	0.48 x 0.43 x 0.21	0.51 x 0.50 x 0.33
Index ranges	-6<=h<=9, -17<=k<=13, -15<=l<=17	-8<=h<=5, -17<=k<=16, -10<=l<=9
Reflections collected	6633	3796
Independent reflections	2741 [R(int) 0.0147]	2838 [R(int) 0.0143]
Data/restraints/ parameters	2741/0/140	2838/1/149
GOF	1.053	1.044
Final R indices [I>2σ(I)]	R1 = 0.0274, wR2 = 0.0742	R1 = 0.0295, wR2 = 0.0691
R indices (all data)	R1 = 0.0314, wR2 = 0.0768	R1 = 0.0326, wR2 = 0.0710
Absolute structure parameter		0.05(3)

	Ethyl(chlorosulfonyl) carbamate **3b**	Triethylammonium methylcarboxysulfaminate **4**	Triethylammonium ethylcarboxysulfaminate **5**
Mololecular Formula	$C_3H_6ClNO_4S$	$C_8H_{20}N_2O_5S$	$C_9H_{22}N_2O_5S$
Formula weight	187.60	256.32	270.34
Temperature (K)	120	120	120
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	*monoclinic*	*monoclinic*	*triclinic*
Space group	*P*21/c	*P*21/n	*P*-1
a (Å)	8.6823(14)	8.3942(5)	8.4796(8)
b (Å)	9.5159(16)	15.2853(8)	8.7778(8)
c (Å)	9.3527(15)	9.9254(5)	10.2367(10)
α(°)	90	90	106.1971(12)
β (°)	99.645(2)	90.1786(7)	93.6684(12)
γ (°)	90	90	108.6712(11)
Volume (Å^3)	761.8(2)	1273.50(12)	683.28(11)
Z	4	4	2
Density (g/cm^3)	1.636	1.337	1.314
Absorp. coefficient (mm^{-1})	0.734	0.263	0.249
F(000)	384	552	292
Crystal size (mm^3)	0.50 x 0.32 x 0.05	0.43 x 0.41 x 0.40	0.53 x 0.33 x 0.23

Index ranges	-11<=h<=5, -12<=k<=12, -12<=l<=12	-10<=h<=11, -19<=k<=17, -10<=l<=13	-11<=h<=9, -9<=k<=11, -11<=l<=13
Reflections collected	4334	7352	3993
Independent reflections	1813 [R(int) 0.0238]	3034 [R(int) 0.0142]	3073 [R(int) 0.0074]
Data/restraints/ parameters	1813/0/96	3034/0/157	3073/0/166
GOF	1.043	1.044	1.074
Final R indices [I>2σ(I)]	R1 = 0.0305, wR2 = 0.0659	R1 = 0.0272, wR2 = 0.0716	R1 = 0.0269, wR2 = 0.0669
R indices (all data)	R1 = 0.0416, wR2 = 0.0707	R1 = 0.0295, wR2 = 0.0729	R1 = 0.0295, wR2 = 0.0686

Selected Images from George M. Atkins' Ph.D. thesis (Georgia Institute of Technology, 1968). The complete original docment is available online at: https://smartech.gatech.edu/handle/1853/27323

REACTIONS OF N-SULFONYLAMINES

A THESIS

Presented to

The Faculty of the Graduate Division

by

George Milton Atkins, Jr.

In Partial Fulfillment

of the Requirements for the Degree

Doctor of Philosophy

in the School of Chemistry

Georgia Institute of Technology

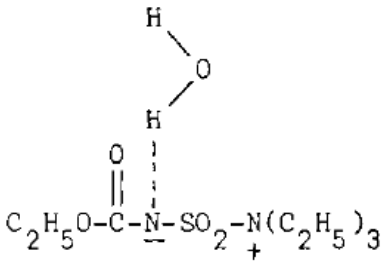
May, 1968

Monohydrate of XXXI (XXXII)

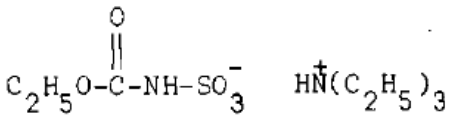
When XXXI was dissolved in benzene containing 1 per cent water and allowed to stand for several hours at ambient temperatures, a crystalline precipitate formed which upon filtration gave the monohydrate of XXXI (XXXII) as transparent plates: mp 89-90°C; ir ($CHCl_3$) 3410, 2980, 1720 (C = O), 1465, 1435, 1270 and 1035 cm^{-1}; nmr ($CDCl_3$) τ 1.62 (broad, s, 2H), 5.86 (q, 2H, $\underline{J}$ = 7 Hz), 6.70 (q, 6H, $\underline{J}$ = 7 Hz), 8.63 (t, 9H, $\underline{J}$ = 7 Hz) and 8 77 (t, 3H, $\underline{J}$ = 7 Hz).

It was possible to convert XXXII to XXXI by gentle heating under vacuum. In one example, XXXI was dissolved in water and allowed to stand for several hours at 25°C. Evaporation of the water under reduced pressure at 45-50°C gave XXXI displaying the same melting point and infrared spectrum as before hydration.

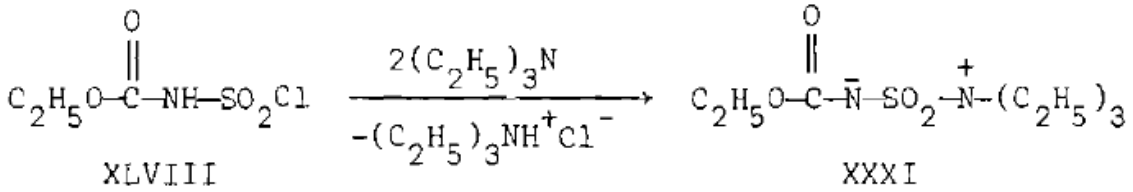
.... In addition, XXXII dehydrated under conditions milder than would be expected for the dehydration of LIII.



XXXII



LIII



XLVIII XXXI